Handbook of natural fibres

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Volume 1: Types, properties and factors affecting breeding and cultivation

Edited by
Ryszard M. Kozłowski

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1

Introduction to natural textile fibres

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Abstract: Natural fibres with their long history of serving mankind are very important in a wide range of applications, and they compete and co-exist in the twenty-first century with man-made fibres, especially as far as quality, sustainability and economy of production are concerned. This 'Introduction' describes the classification of natural fibres, levels of production and advantages derived from their excellent permeability, biodegradability and healthy properties. Natural fibres conduct heat, can be properly dyed, resist mildew, have natural antibacterial properties, block UV radiation and can be easily made flame retardant. Genetic modification of natural fibrous raw materials improves their productivity and performance. The confirmed importance of natural fibres resulted in the United Nations and the Food and Agriculture Organization of the United Nations declaring 2009 as the International Year of Natural Fibres.

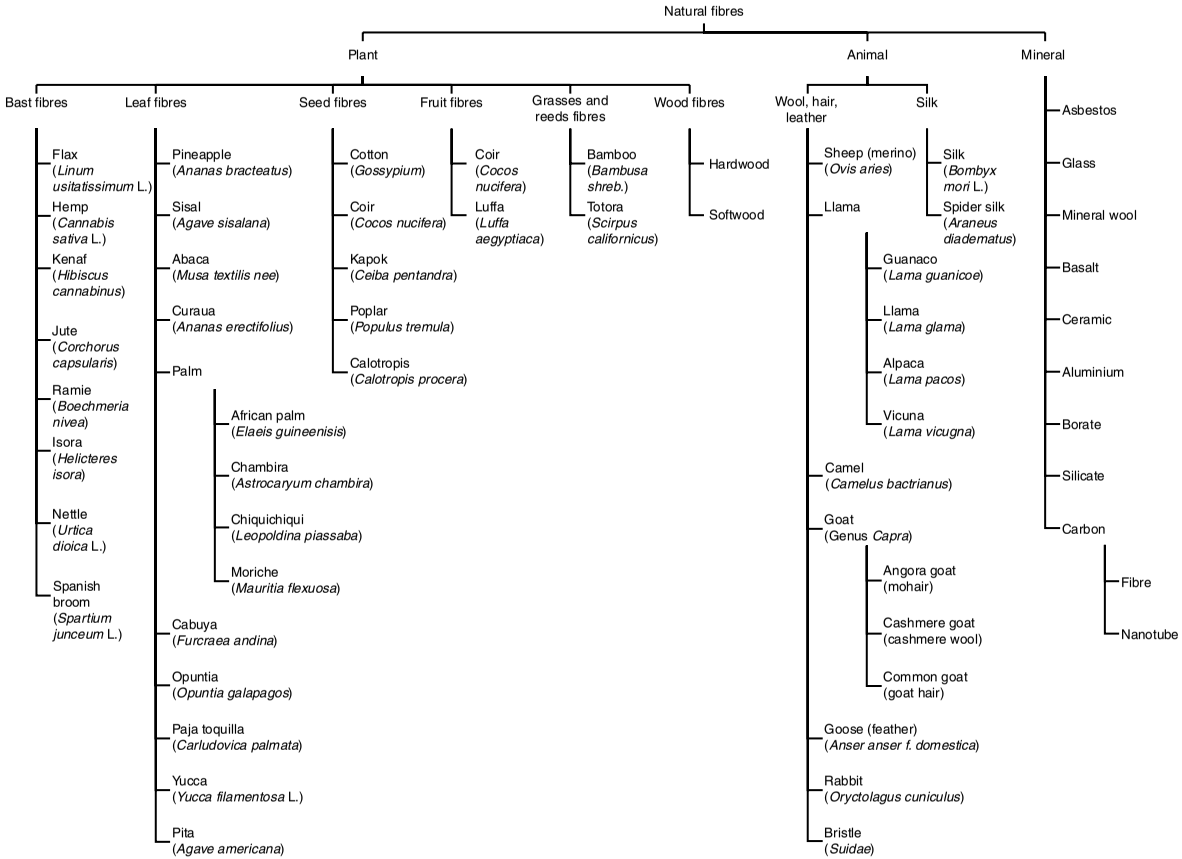
Key words: natural fibres: silk, cotton, wool, bast, hard, treatment, physiological effect on human body, genetically modified (GM), history of natural fibres.

1.1 Introduction

The United Nations and the Food and Agriculture Organization of the United Nations (FAO) declared 2009 as the International Year of Natural Fibres. Their objectives were: to raise the profile of natural fibres, to stimulate demand for them by promoting the efficiency and sustainability of the natural fibre industries and to encourage appropriate policy responses from governments to the problems faced by these industries. In so doing they hoped to establish an effective and enduring international partnership among the various natural fibre industries (FAO documents, e.g., regarding International Year of Natural Fibres 2009, or Joint Meetings of the Intergovernmental Groups (IGGs) on Hard Fibres and on Jute, Kenaf and Allied Fibres).

The classification of natural fibres is presented in Fig. 1.1.

Nature, in its abundance, offers us numerous fibrous materials, which grow in multiple geographical altitudes. Various parts of these lignocellulosic plants such as the woody core, bast, leaf, cane, straw, grass and seed are sources of valuable lignocellulosic fibres suitable for use not only in textiles,



1.1 Classification of natural fibres. (Based on Kozlowski R., Kicińska-Jakubowska A. and Muzyczek M. (2009), 'Natural fibres for interior textiles', in T. Rowe (ed.), *Interior Textiles Design and Development*, Woodhead Publishing Ltd, UK.)

but also in building materials, human and animal food, agro-fine chemicals, environment friendly cosmetics and as sources of biopolymers and energy.

Natural fibres have played an important role in human society since approximately 7000 BC.[1,2] They are completely biodegradable and their production does not generally damage the ecosystem. They can grow in different climates and recycle carbon dioxide (CO_2). Several species, for example bast fibrous plants, can be used to clean soil polluted by heavy metals, due to their ability to extract cadmium (Cd), lead (Pb), copper (Cu) and zinc (Zn).[3–5]

The growing world population will mean increased demand for and consumption of fibres. The current population explosion is alarming enough to have necessitated a search for alternative textile resources, mainly for healthy, sustainable and comfort-providing natural fibres.[3,4,6,7]

The production of these fibres is expected to reach levels of 35–40 million tons per year by the middle of the twenty-first century. The response to this demand is likely to mean an increase in cotton production of up to 26–30 million tons per year. It will also mean growth in the production of other natural materials, including modified bast fibres, as well as in man-made fibres.

The textile industry is currently broadening its search for alternative green fibres with the aim of providing healthy, comfortable clothing, which, at the end of its lifetime, will be fully recyclable and biodegradable. Most natural fibres can be taken from lignocellulosic fibrous plants such as flax, jute, hemp, kenaf, sisal, ramie, abaca, curaua, coir, cabuya, pineapple and bamboo. Many others can be extracted, processed, modified and used both in textiles (woven, knitting, nonwoven, technical) and for technical purposes, for example, as reinforcements to create more environmentally friendly composites in areas such as pulp and paper, agro-fine chemicals and as sources of energy.[8]

Special treatments like enzymatic, liquid ammonia, plasma and corona ultrasound provide promising new features and properties for fibres and fabrics.

Natural fibres are generally characterized by their most important properties such as air permeability, hygroscopicity, their ability to release moisture, the fact that they do not release any substances harmful to health or cause allergic reactions (resulting from a higher level of histamine in human blood), and by their biodegrability and lower flammability in comparison to man-made fibres.[6]

The potential for genetically modified (GM) fibrous plants has increased in recent years, due to the better yield efficiency of major natural products such as fibre cellulose and carbohydrates. The possibility now also exists for obtaining polyhydroxyalkanoate (PHA), for example, polyhydroxybutyrate – a natural polyester *in statu nascendi*, which isn't typically synthesized in a plant.[7]

GM fibrous plants can be resistant to herbicides and environmental stress including salinity and drought. In the case of bast fibrous plant production, the most important future direction is toward the genetic modification of lignin and pectin content.

GM plants could be used for improving the biomass of bast plants as well as for obtaining a higher oil content in plants. The potential for novel fibrous plants containing nanofibres and cellulose modified with phosphate groups to achieve a higher thermal resistance has been the focus of many researchers' and scientists' attention. Bio-silk fibres based on polyamino acids, and very promising fibres based on fibroin and chitins have also aroused increasing interest.[7]

Natural fibres, with their long history in the service of humankind, are finally gaining recognition as welcome and user-friendly materials, both in clothing and for other end uses.[1,2,9] After a period of dynamic development for man-made fibres and one of drastically lowered production for natural fibres in the last decade, a more stable coexistence between them may now be observed.[4] The conditions for this potential future coexistence will be dictated by the level of ecological alarm over atmospheric and water pollution. In the opinion of textile authorities, the present ecological legislation pales in comparison to that which will be needed in the future to deal with the ecological problems that the world is facing. The treatments of effluents and disposal of waste and packing materials will require enormous capital outlays. The competitive edge in the future will be determined to a great extent by a company's ability to solve its ecological problems. This may prove to be one of the greatest challenges for the textiles industries this century.

Currently, the coexistence and competition between man-made and natural fibres is stabilized – especially in the areas of quality, sustainability and economy of their production. Natural and renewable fibres conduct heat, dye well, resist mildew, have natural antibacterial properties, block ultraviolet (UV) light and are easily treated for flame retardancy. This makes them ideal for the production of comfortable healthy clothing that provides UV protection for the body, decreases oxidative stress and muscle tension and increases the level of alpha-globulin, thus improving the wellbeing of the user.[6,7]

Natural fibres also provide the industry with valuable, renewable resources such as flax and curaua, which, blended with carbon fibres, provide an excellent reinforcing material for composites with a high mechanical strength. Jute, hemp, kenaf and abaca are used for rope, canvas and pulp and paper, and also to reinforce moulded thermoplastics in the automobile industry. The short core fibres are used in insulation products, fibre boards and erosion control mats, while the fibrous core can be blended with lime to make strong and lightweight concrete. Shives can also be used as raw materials in the production of particle boards and bedding for edible fungi production. Kenaf, jute and hemp shives are excellent bedding materials for horse stables and chicken farms. By-products from bast fibrous plants, especially seed capsules, are also a source of valuable fatty acids, amino acids, phytoestrogens, cyclolinopeptides, waxes, lignin, pectin, mucilage and other agro-fine chemicals. They are one of the richest plant sources of polyunsaturated fatty

acids (omega 3 and omega 6 'family'), which are effective for the prevention of cardiovascular and heart diseases (CHD).

The above mentioned facts now influence the position of natural fibres and the stable level of their production thanks to the growing areas of their application, not only in textiles (woven, knitting, nonwoven, technical textiles), but also in more eco-friendly composites such as agro-fine chemicals used in nutrients, pharmaceuticals and cosmetics.

1.2 Historical background of natural fibres

Textile fibres have been used to make cloth for the last 4000 or 5000 years. Until 1885, when the first man-made (artificial) fibre was produced commercially, fibres were obtained from plants and animals. The fibres most commonly used were: flax, hemp, silk, wool and cotton.

Rayon, also known as artificial silk, was the first man-made fibre and was produced in filament length until the early 1930s, when an enterprising textile worker discovered that the broken and wasted rayon filaments could be used as a staple fibre.

Many man-made fibres were developed in the first half of the twentieth century and, from that time onwards, tremendous advances have been made in the man-made fibre industry.

Cotton cloth was used in ancient China, Egypt and Peru. Cloth fabrics found in Egypt indicate that cotton may have been used there in 12 000 BC, before flax was discovered.[10]

Cotton spinning and weaving as an industry began in India and fabrics of good quality cotton cloth were being produced as early as 1500 BC. The Pima Indians were growing cotton when the Spaniards came to the New World. One of the items that Columbus took back to Queen Isabella was a hank of cotton yarn, and until the eighteenth century Europe was dominated by flax.

It has been argued that flax is the oldest fibre used by mankind. Flax samples, delivered by the British National Museum of Antiques for testing at the Institute of Natural Fibres in Poznan (Poland), were taken from Çatal Hüyük (Turkey) and Kerma (Nubia, Sudan) and proved to be flax from 6500 years BC and 2000 years BC, respectively.[1] It is widely known that linen mummy cloth more than 5000 years old has been found in Egyptian tombs. It is fascinating that this fibre is so fine that it has lasted until the present day.[9]

Wool-sheep were probably the first animals domesticated by people. Cross-breeding sheep to increase the amount of hairy undercoat began in approximately AD 100. By AD 1400 the Spanish had developed the merino sheep, whose fleece is the precursor of contemporary wool raw materials.[11,12]

Silk is a natural protein fibre. Silk culture, according to oldest Chinese literature, began when a Chinese empress observed the life of silkworms and studied their features. Based on this observation Chinese experts began to breed them and from the filaments gained the empress produced expensive silk cloth. The Chinese silk industry spread over the years and brought China an extensive income – to the extent that revealing the secret of silk production became punishable by death. For 300 years, therefore, the secret was known only to Chinese experts. During that time China was the only producer of silk in the world and sold it through Persian and Egyptian agents at the price of gold.[13]

The elements of the history of natural fibres, their properties and classification have been incorporated into the *Handbook of natural fibres* chapters.

1.3 Handbook of natural fibres

The *Handbook of Natural Fibres* describes the present state-of-art in the area of natural fibres. The authors have been chosen from a group of high-ranking, renowned specialists in the area of natural fibres. It consists of two volumes: Volume 1 is entitled: *Types, properties and factors affecting breeding and cultivation* with 18 chapters, written by 35 authors from 13 countries. Volume 2 entitled *Processing and applications* includes 16 chapters, prepared by 26 authors from 10 countries.

Volume 1 consists of the introduction, prepared by the Editor and two parts: Part I: Fundamentals: types of fibre, properties, identification and testing and Part II: improving natural fibre production through breeding and cultivation. Part I provides fundamental knowledge about types of major plant and animal fibres, their properties, identification and testing. The fibres described are: cotton, selected bast fibres (jute, ramie, flax and hemp), silk, wool, mohair, cashmere and other hair fibres as well as bioengineered natural fibres. Chapter 11 on identification of natural fibres provides knowledge about contemporary methods in that regard, allowing for unambiguous identification of diversified natural fibres. Chapter 12 on testing of natural fibres describes traditional as well as contemporary testing methods.

Part II of Volume 1 deals with the following subjects: developments in fibrous flax breeding and cultivation; cotton breeding; breeding and cultivation of fibre flax in sustainable agriculture; prevention of fungal growth, especially mildew, in natural fibres; genetic engineering and biotechnology in natural fibre plants; wild silk fibres for poverty alleviation near protected areas.

Volume 2: *Processing and applications* consists of two parts: Part I: Processing techniques for natural fibres and Part II: Applications of natural

fibres and case studies. Part I describes current knowledge regarding methods of improving specific properties of natural fibres as well as selected treatments and processing, the following areas are covered: silk production and future trends; improving the flame retardancy of natural fibres; improving the properties of natural fibres by chemical treatments; ultraviolet-blocking properties of natural fibres; enzymatic treatment of natural fibres; electrokinetic properties of natural fibres. Part II of Volume 2 discusses major textile and non-textile utilizations of natural fibres in such industries as automotive, aerospace, building, geotextiles and paper and packaging. Antimicrobial natural fibres, biomimetics and textile materials, methods of enhancing consumer demand for natural textile fibres and environmentally friendly textiles from jute and coir are also described.

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2

Cotton fibres

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Abstract: Cotton, the seed hair of plants of the genus *Gossypium*, and the purest form of cellulose available in nature, is the dominant natural fibre. Cotton has a multilayered structure which consists of a primary wall, a secondary wall and lumen. Under the microscope it looks like a twisted ribbon or like a collapsed and twisted tube. The strength of cotton has been attributed to its highly fibrillar and crystalline structure and its strength is increased by 25% when it is wet. Cotton is a good conductor of heat, is susceptible to damage by mildew, turns yellow and becomes weak when exposed to prolonged sunlight. It is easily flammable and has rather poor resistance to wear. The highest quality cotton varieties have the longest fibres, thin, with good resilience and elasticity which makes them easy to spin, suitable for the production of high-quality goods – especially garments. Recent advances have included production of genetically modified cotton (GM) and also organic cotton.

Key words: cotton, organic cotton, genetically modified cotton, genus *Gossypium*, structure, strength, short, medium, long staple, quality measuring, mildew resistance.

2.1 Introduction

Cotton has many desirable fibre properties making it a major fibre for textile applications. It combines strength with good absorbency, for example, making it a comfortable and durable apparel fabric. Mankind first learnt to utilize cotton more than 5000 years BC in India and the Middle East. Its use spread to Europe via the Greeks after the invasion of India by Alexander the Great. Modern cotton manufacture began in England in the eighteenth century and rapidly spread to the United States, resulting in a huge increase in production and international trade. It was not until the emergence of man-made fibres in the twentieth century that cotton was displaced as the most important textile fibre. It is still the most widely used natural textile fibre with over 25.2 million tons produced annually. Consumption of cotton is still growing at a rate of 2% per annum. The English word cotton comes

from the Arabic word katan, the name originally given to flax. Cotton is known by various names throughout the world, including:

- Arabic countries – katan, gatu, kotan, kutn, gutn, or kuteen,
- China – hoa main,[1]
- France – coton,
- Germany – baumwolle,
- Greece – vamvax,
- India – pucu,
- Iran – pembeh or poombch,
- Italy – cotone,
- Japan – vatta ik or vatta noki,
- Poland – bawełna,
- Russia – khlopok.

2.2 The cotton plant

Cotton, the seed hair of plants of the genus *Gossypium*, is the purest form of cellulose available in nature. After flowering, an elongated capsule or boll is formed on the cotton plant in which the cotton fibres grow. Once the fibres have completed their growth cycle, the capsule bursts and fibres emerge. A cotton capsule contains about 30 seeds. Each seed contains around 2000–7000 seed hairs (fibres). Depending on the cotton type and growing conditions, the colour of the fibre is usually creamy white or yellowish. Cotton fibre is mostly composed of cellulose. Under 10% of the weight of the raw fibre consists of waxes, protein, pectate and minerals. Table 2.1 shows the chemical composition of cotton fibre. The chemical structure of cotton is discussed in more detail in Hsieh.[2]

Table 2.1 Chemical composition of cotton fibre[20]

	Per cent (dry basis)		
Constituents	Typical	Low	High
Cellulose	94.0	88.0	96.0
Protein	1.3	1.1	1.9
Pectic substances	0.9	0.7	1.2
Ash	1.2	0.7	1.6
Wax	0.6	0.4	1.0
Malic, citric and other organic acids	0.8		1.0
Total sugars	0.3		
Pigment	Trace		
Other	0.9		

The length of different kinds of cotton fibre varies from 22 to 50 mm, and the diameter from 18 to 25 μm. The higher quality fibres are known as long-staple fibres or extra-long staple.[3]

There are four main commercial species of cotton from the genus *Gossypium*:

- *G. arboretum* (Middle and Far East),
- *G. herbaceum* (Middle and Far East),
- *G. hirsutum* (America),
- *G. barbadense* (America and Egypt).

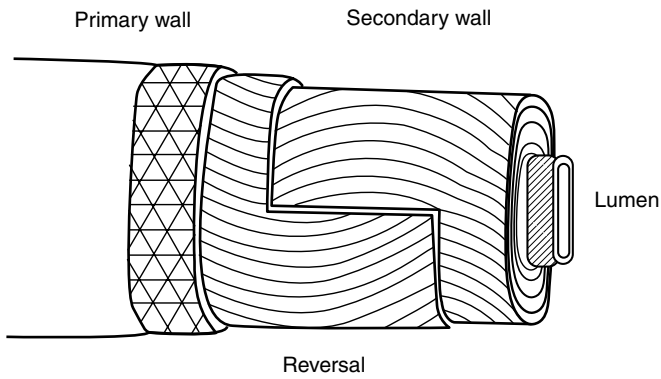
Of these varieties:

- 80% of the world's cotton fibre production is from *G. hirsutum*,
- 10% is long and extra long fibre varieties of *G. barbadense*,
- the remaining 10% is the two short fibre Asiatic types.[1]

The cotton plant grows best in subtropical countries in warm, humid climates. The best conditions for cotton growing are between 47°N and 35°S. The plant requires 6–7 months of warm weather. More than 80 countries grow cotton. Cotton is cultivated in North and South America, the Middle East, Africa, India, China and Australia. In Europe it is grown in Mediterranean countries such as Greece and Bulgaria. Recently cotton production has shifted to more environmentally friendly techniques such as organically produced cotton. The market for organic cotton was estimated to be more than US$7 billion in 2010. The top organic cotton growing countries are: Turkey, India, China, Syria, Peru, the United States, Uganda, Tanzania, Israel and Pakistan.[4] There has also been a greater emphasis on naturally coloured cotton which does not need dyeing. This is produced by both traditional breeding and genetic engineering. Organic production of cotton is discussed in Wakelyn and Chaudhry[5] whilst genetic engineering of cotton is discussed in Orford *et al*.[6] Naturally coloured cotton has pigmented fibres with the colours as a part of the lumen. Exposure to sunlight tends to deepen the colour (flavonoids are the major contributors to the colour). Generally naturally coloured cotton is finer, shorter and weaker. A wide range of colours can be produced from green and brown cotton.[7]

2.3 Cotton fibre structure

Cotton fibres have a multilayered structure that has been studied for nearly a century. The structure of the primary cell wall of the cotton fibre, and particularly the outer surface layer (the cuticle), has a major influence on fibre properties, processing and use.[8] Cotton fibre has a fibrillar structure which consists of a primary wall, a secondary wall and a lumen (see Fig. 2.1).[9,10] The typical



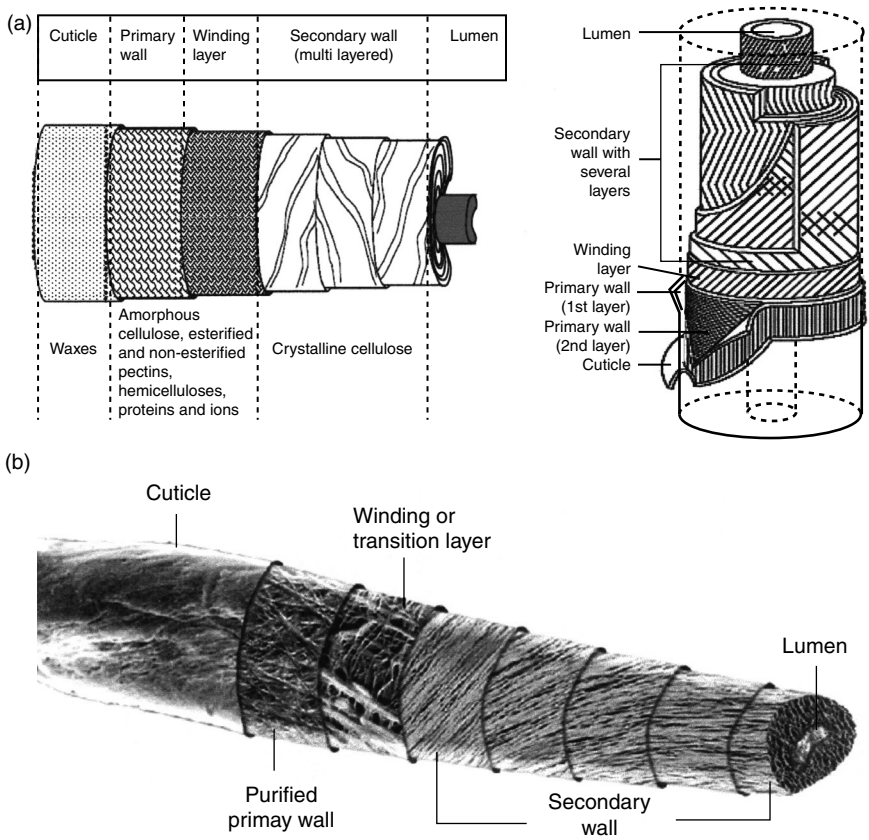
2.1 Structure of cotton.[9,10]

components of dry mature cotton fibres are shown in Figs 2.2 and 2.3. Most of the non-cellulosic materials are present in the outer layers of cotton fibre.[3,11]

Under a microscope a cotton fibre looks like a twisted ribbon or a collapsed and twisted tube (Fig. 2.4).[10–12] These twists are called convolutions: there are about 60 convolutions per centimetre. The convolutions give cotton an uneven fibre surface, which increases inter-fibre friction and enables fine cotton yarns of adequate strength to be spun. The cross-section of a cotton fibre is often described as being kidney-shaped. Figure 2.5 shows scanning electron microscopy (SEM) images of different layers in the figure. The outermost layer, the cuticle (Fig. 2.5b), is a thin film of mostly fats and waxes. Figure 2.5b shows the waxy layer surface with some smooth grooves. The waxy layer forms a thin sheet over the primary wall that forms grooves on the cotton surface. The primary wall (Fig. 2.5c) comprises non-cellulosic materials and amorphous cellulose in which the fibrils are arranged in a criss-cross pattern. Owing to the non-structured orientation of cellulose and non-cellulosic materials, the primary wall surface is unorganized and open. This gives flexibility to the primary wall, which is required during cell growth. The basic ingredients responsible for the complicated interconnections in the primary wall are cellulose, hemicelluloses, pectins, proteins and ions. The secondary wall, in which only crystalline cellulose is present, is highly ordered and has a compact structure with the cellulose fibrils lying parallel to one another (Fig. 2.5d).[3]

2.4 Physical properties of cotton

As has been seen, cotton consists essentially of pure cellulose, with fibres having a flat, twisted, ribbon-like appearance. This structure determines its physical properties. The structure and physical properties of cotton have been reviewed in detail by Hearle.[13] The strength of cotton has been attributed to its highly fibrillar and crystalline structure. Cotton increases its

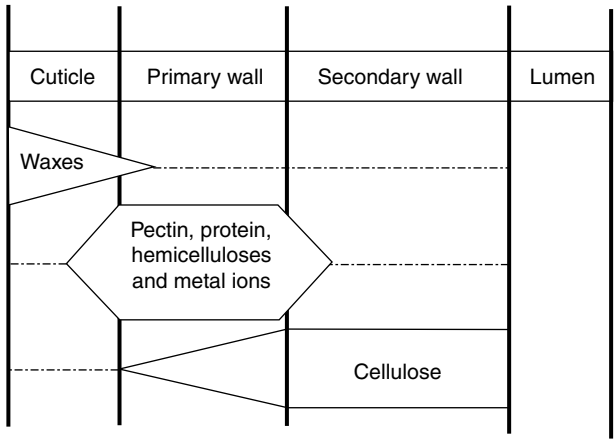


2.2 A schematic representation of mature cotton fibre showing its various layers.[3,11] (a) Cross section of cotton fibre. Typical components in dry, mature cotton fibres and compositions of each layer. (b) Morphological model of cotton fibre.

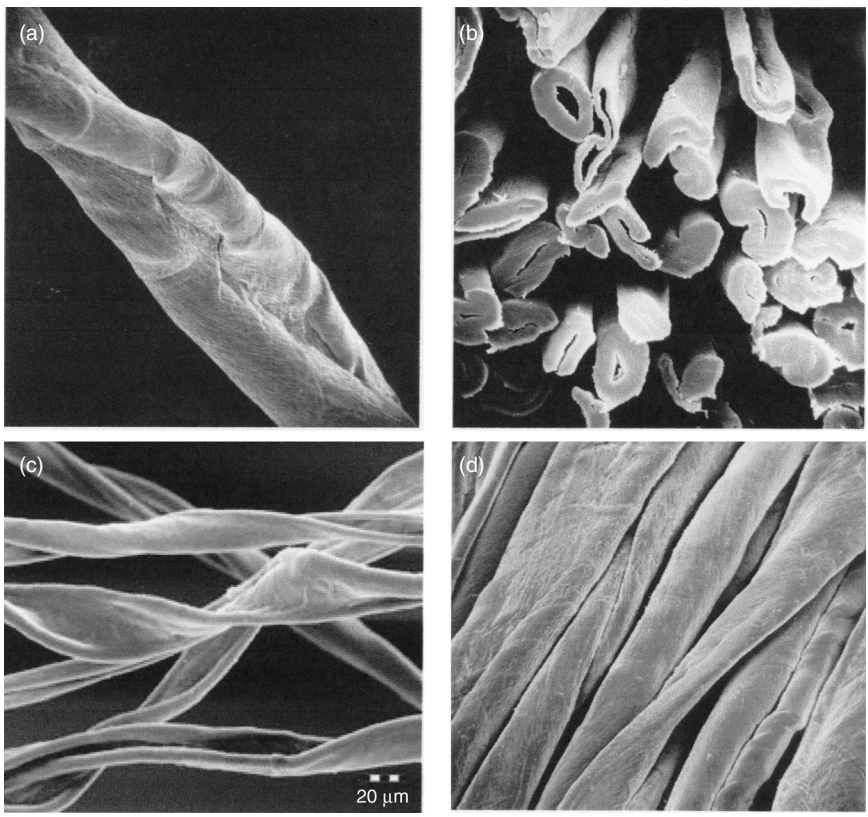
strength by 25% when wet. However, cotton is not extensible, which means untreated cotton fabrics crease badly. It is a good conductor of heat, making cotton clothes cool to wear. It absorbs water but dries slowly (standard moisture regain = 8.5%), soils easily because of the rough surface of cotton yarns and shrinks on washing, particularly when strongly alkaline washing solutions are used. It is susceptible to damage by mildew (it should not be stored damp), and turns yellow and weakens when exposed to prolonged sunlight. Cotton is resistant to milder alkalis but is damaged by acids, is very flammable and has rather poor resistance to wear.[14]

The basic physical properties of cotton are:[10]

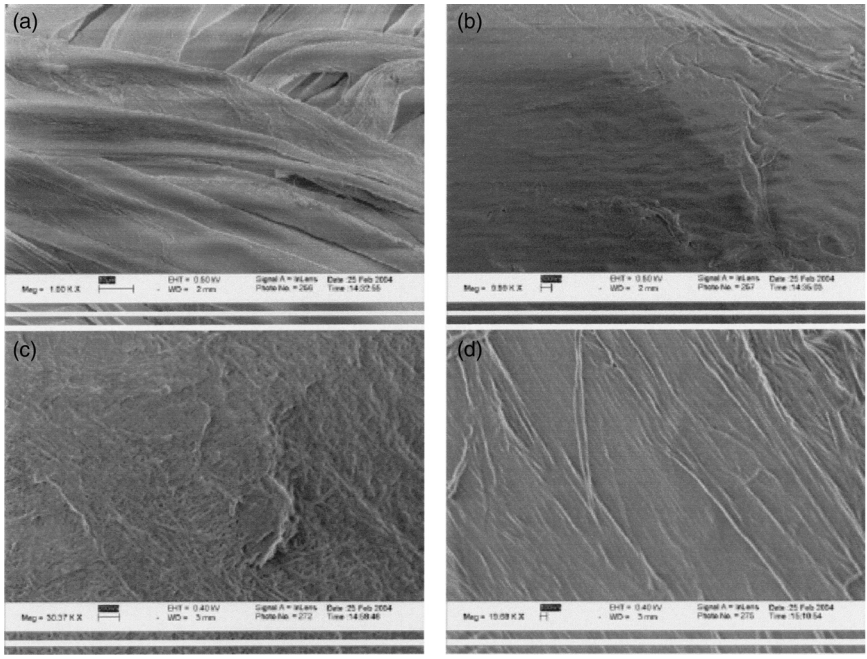
- fibre length (mm) – 12–60,
- fibre diameter (μm) – 12.22,



2.3 A schematic representation of the cellulosic and non-cellulosic materials in the cotton fibre.[3]



2.4 The appearance of cotton fibre under the microscope.[10–12] (a) Cotton fibre looking like a twisted ribbon; (b) the cotton fibre's cross section is referred to as being kidney-shaped; (c) cotton fibre looking like a collapsed and twisted tube and (d) bundle of cotton fibres.



2.5 SEM images of the different layers. (a) Fibres from desized cotton fabric; (b) amorphous wax surface of the desized cotton fibre; (c) network of primary wall of cotton fibre and (d) secondary wall of cotton fibre

- tenacity, dry (g/den) – 2.1–6.3,
- tenacity, wet (g/den) – 2.5–7.6,
- elongation (% at break) – 3.10,
- specific gravity (g/cm^3) – 1.50,
- moisture regain, 21°C, 65% – relative humidity (RH) 8.5.

The physical properties of cotton are summarized in Tables 2.2 and 2.3.[7]

The basic parameters of cotton fibre, on which the technological suitability and the price of the particular cotton lot depend, are:

- staple length,
- colour grade,
- fibre strength,
- fibre thickness,
- micronaire value: an indication of fibre-specific surface, determined by fibre fineness and maturity,
- leaf grade.

Table 2.2 Physical properties of naturally coloured cotton[7]

Cotton	Spun length (2.5 mm)	UR classification (%)	Fineness (microns)	Strength (g/tex)	Elongation (%)	Ash content (%)
Brown	20.10	48.5	3.9	19.50	5.2	1.15
White	19.50	47.8	3.2	18.98	5.6	0.88

UR, uniformity ratio.

Table 2.3 Fibre properties of naturally coloured cotton[7]

Fibre properties of coloured cotton	DDCC-1 – cultivar of coloured cotton	Hybrid
2.5 Span length (mm)	24.5	26.6
Uniformity ratio (UR) (%)	46.0	50.6
Micronaire value (to evaluate fibre fineness and its suitability for spinning)	3.7–4.5	3.8
Tenacity = max. tensile force/linear density [gram force (gf)/tex], 1 tex is equivalent to the mass in grams/1 km fibre	18.7–21.3	23.4
Counts – linear density or yarn count (tex: weight in grams of 1000 m yarn)	30's	40's

Cotton fibre is classified into three basic commercial groups, based on fibre length:

1. Top quality fibres with a staple length of between 30 and 65 mm. Staple cottons – well-known types such as Egyptian and Sea Island are included in this group.
2. Fibres with a staple length of between 20 and 30 mm. These medium length fibres are the most common form of cotton and include the American upland variety.
3. Fibres with a staple length of less than 20 mm from coarse, lower grade cotton, which includes many Asiatic and Indian fibres.

The highest quality cotton varieties have the longest fibres which are simultaneously thin and very resistant to tearing (breaking). Such fibres are characterized by good resilience and elasticity which makes them easy to spin. They produce thin, strong cotton yarns suitable for the production of high-quality goods (especially garments). Long staple cotton represent 3–5% of the world's production and are used for delicate fabrics with specific weight of <100 g/m^2 in the production of high-quality shirts and blouses, the best quality bed linen and underwear, etc. Relevant parameters are:

- staple length: between 30 and 65 mm,
- micronaire: 2.8–4.5,
- strength: 33–45 g/tex pound force per square inch (PSI).

Long staple cotton typically comes from Egypt and the USA.

Medium staple cotton (about 85% of the world production and processing) is designated for the production of medium thick fabrics with specific weight 100–250 g/m^2 appropriate for the production of bed linen, table cloths, good quality denim (jeans) cloth as well as underwear. Medium cotton parameters include:

- staple length: between 20 and 30 mm,
- micronaire: 3.5 – 4.8,
- strength: 25–33 g/tex (PSI).

Medium staple cotton is produced in Central Asia (Uzbekistan, Tajikistan, Kazakhstan, Turkmenistan), West Africa (Chad, Mali, Ivory Coast, Burkina Faso), Europe (Greece, Spain), the Middle East (Turkey, Syria), the USA, Brazil and Pakistan.

The cheapest cotton varieties with short, rigid fibres are used for the production of thick yarns of lower quality. Short staple cotton is designated especially for the production thick fabrics with specific weight >250 g/m^2, for example, for denim, drill, flannel for work clothes, upholstery, carpets, etc. Typical parameters are:

- staple length: less than 20 mm,
- micronaire: 4.5 – 6,
- strength: 14–18 g/tex (PSI).

Lower quality varieties come from Central Asia (Uzbekistan, Tajikistan, Kazakhstan, Turkmenistan), the USA and India.[1]

Various treatments can improve the physical properties of cotton. Anhydride liquid ammonia produces effects similar to mercerization such as swelling of cellulosic fibres, but does not degrade the fibres as the caustic soda traditionally used in mercerization does. Mechanical properties like tensile properties and abrasion resistance are slightly improved, and the resulting cloth has better hand as well as better washing performance (e.g., less shrinkage). The process is performed at temperature lower than – 34°C by evaporation of liquid ammonia. Cellulose I, which has the semi-crystalline structure of native cellulose, is transformed to cellulose III which has a more crystalline structure.[15]

2.5 Measuring cotton quality

Cotton fibre quality is reviewed in Gordon[16] whilst testing is discussed by Hunter.[17] Government bodies such as the US Department of Agriculture (USDA) set standards for instrument type, calibration and accuracy. Maximum allowable tolerances for accuracy and precision are listed in Table 2.4. Bodies such as USDA also set standards for testing conditions.

Table 2.4 Maximum allowable tolerances for accuracy and precision of cotton testing instruments[18,19]

Fibre property	Accuracy	Precision
Length (inch)	±0.018	±0.012
Uniformity (%)	±1.200	±0.800
Strength (g/tex)	±1.500	±1.000
Micronaire (units)	±0.150	±0.100
Colour (Rd) (units)	±1.000	±0.700
Colour (+b) (units)	±0.500	±0.300
Trash (% area)	±0.100	±0.040

According to USDA, the testing laboratory and the cotton conditioning area must have an atmosphere maintained as follows: temperature 21 ± 0.6°C, RH 65% ± 2%. For testing purposes, a cotton sample must be taken from opposite sides of a cotton bale. Each portion should be approximately 300 mm long and ca. 150 mm wide and should have approximately the same mass. The total sample submitted for testing should weigh approximately 225 g, and be identified with an identification tag (coupon) rolled between the sides giving the bale number. Cotton samples must be brought to a moisture content that is at equilibrium with the approved atmospheric conditions before testing: moisture content between 6.75% and 8.25% (dry mass basis) prior to instrument testing. Cotton samples can be conditioned passively or actively, not covered by sacks, wrappers or any bother coverings, and conditioning takes usually 48 h.[18]

The International Cotton Advisory Committee (ICAC), which represents cotton producers around the world, set up a task force in 2003 on the Commercial Standardization of Instrument Testing of Cotton (CSIRC). This has seven key objectives:

1. Define specifications for cotton trading.
2. Define international test rules.
3. Implement test rules.
4. Certify test centres.
5. Define calibration standards.
6. Specify commercial control limits for trading.
7. Establish uniform arbitration procedures.

The CSIRC process is designed to ensure greater uniformity in testing procedures and results.

High volume instrument (HVI) systems use techniques such as near infrared spectroscopy. They can accurately measure length, strength, micronaire, length uniformity index, colour Rd, colour +b and trash content. HVI fibre testing systems provide accurate information on how fibre properties will

affect yarn and ultimately fabric quality. The focus is on predicting how the fibre will perform in subsequent processing and in the final product. As an example the Rothschild MC-CT RO 2005 Mini Card can measure fibre cohesion and drawing force in staple fibres. The H2SD High Speed Cotton Stickiness Detector (CIRAD, France) can assess the presence of sticky cotton of sample in less than 30 s. Zweigle's F 460-stick-Slip Friction Tester measures gliding properties (drafting behaviour). SDL's Quickspin system allows a rapid determination of raw material characteristics and thus yarn quality. The Zellweger Intelligin system optimizes the ginning process by continuous on-line measurement of fibre moisture, trash level and colour grade. HVI systems are also used to monitor yarn quality. The Uster HVC Spectrum system includes automated sampling, maturity index measurement and moisture sensor.

The Uster Yarn Tester 4 SH measures diameter, shape, yarn structure, dust and trash content, evenness, imperfections and hairiness. The OASYS (Optical Assessment System through Yarn Simulation) system optically scans yarns independently of mass distribution and transmits the yarn profile to OASYS software, which simulates woven or knitted fabrics according to the users' specifications.[19]

2.6 Future trends

Key research and development trends in cotton include:

- improving knowledge of the cotton plant and fibre properties;
- developing cotton cultivation and processing to reduce its environmental impact;
- improving and standardizing method for fibre and yarn quality evaluation;
- promoting the appeal of cotton as a natural fibre and finding new applications of cotton seeds and their by-products.

A major trend in this sector is in reduction of the use of fertilizers and other chemicals in agricultural production, both to control costs and reduce the environmental impact of cotton processing. Countries such as the USA have turned increasingly to organic production and genetically engineered cotton to reduce costs and improve yields. Smart sensor systems are also used to ensure more efficient application of fertilizers and irrigation. Similar efforts are going into later stages of cotton processing, such as bleaching and dyeing, which have traditionally had a significant environmental impact. As an example, non-impact dyes can reduce water use and require lower temperatures to fix the dyes. Enzyme technologies are also being used to replace chemical processing.[1]

2.7 Acknowledgement

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3

Bast fibres: jute

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Abstract: Among all bast fibres, jute is one of the most significant and versatile fibres of commercial and technical importance. Jute, also a cellulosic fibre, ranks next to cotton in terms of production. Jute is an annual herbaceous plant mainly cultivated in the equatorial, the tropical and the sub-tropical zones. Jute plays a vital socio-economic role in producing countries. About 12 million farming families, mainly in South East Asia, are dependent on this crop. Out of over 30 important species belonging to the genus *Corchorus*, only two – *C. capsularis* commonly known as 'White jute' and *C. olitorius* known as 'Tossa jute' – are utilized for fibre production on a commercial scale. This chapter discusses almost the whole gamut of jute starting from its origin, history, agro-climatic condition and production area, economic importance, botanical description, different stages of cultivation, fibre extraction, fibre quality and grading, fibre morphology, structure, chemical composition, properties, products, traditional/unconventional uses, etc. In addition, the chapter briefly describes the environmental advantages and the socio-economic impacts of jute along with its future potential.

Key words: jute, genus *Corchorus*, *C. capsularis*, *C. olitorius*, cellulosic fibre, morphology, structure, environmental advantages, socio-economic impact.

3.1 Introduction to jute

Among the natural fibres, jute ranks next to cotton in terms of production. Jute is a cellulosic fibre under the category of bast fibres and its cultivation is almost as old as human civilization. Jute, an annual herbaceous plant, is mainly cultivated in South and South East Asia. Jute was first used as an industrial raw material for making packaging materials, replacing flax and hemp grown in Europe.

3.1.1 Origin

Corchorus species are found in warm regions throughout the world, in all continents and in numerous tropical and sub-tropical regions. *Corchorus* is a pan-tropical genus comprising an uncertain number of species, with estimates ranging from 40 to 100. However, the centre of diversity and origin of the genus

appears to be Africa, where the largest number of *Corchorus* species (around 30) has been found with the highest concentration reported from East and South Africa. Of the cultivated species *C. capsularis* is omnipresent in Indo-Myanmar and South China, from where it migrated to India and Bangladesh. The primary centre of origin of *C. olitorius* is probably Africa, while India or the Indo-Myanmar region is a secondary centre. Both species are cultivated and naturalized in many parts of the tropics, including South East Asia.

3.1.2 History

When the jute plants were recognized as a source of fibre and utilized for making ropes and sacking, mainly in the Indian subcontinent, is not known definitely. References to sacking bags made of jute have been traced to the literary works of the region as far back as 1575. Sackcloth made of jute has been referred to as an article of trade in several Bengali poetical works of the sixteenth and seventeenth centuries. Rumphius in 1743, one of the earliest workers on Bengal plants, gave an illustrated account of jute plants along with a figure of *C. capsularis* mentioning therein that it was under cultivation in Bengal (India), the Arakans and South China. He even mentioned that the fine white thread made out of this fibre was stronger than that from cotton. Jute is believed to have been traditionally in use in many other parts of Asia and Africa since ancient times to provide cordage and weaving fibres from the stem and vegetables of the leaves.

3.1.3 Adaptation/agro-climatic conditions

Favourable conditions for jute cultivation are found in the floodplains of the great rivers of the tropics and sub-tropics – the Ganges, the Irrawaddy, the Amazon and the Yangtze – where irrigation, often characterized by extensive flooding, and alluvial soils combine with long day lengths. The crop traditionally thrives very well under rain and hot humid and sub-tropical conditions in the Bengal Basin in India and Bangladesh where more than 80% of the world crop is grown.

Jute is mainly grown between 16°N and 27°N, during the hot wet summer season in a hot and humid climate with temperature in the range of 24°C–37°C. Growth is retarded at temperatures below 17°C and above 42°C. The annual rainfall should be 1000–2000 mm of pre-monsoon showers at sowing time.

3.1.4 Areas of production

Jute is mainly cultivated in the equatorial, the tropical and the sub-tropical zones. It is extensively cultivated in India and Bangladesh. Other major

jute growing countries are Myanmar, Nepal, China, Vietnam, Thailand and Brazil.

3.1.5 Economic importance

Jute plays a vital socio-economic role in producing countries. It is produced by hundreds of thousands of small-farm families in South East Asia. About 12 million farm families are dependent on this crop. It provides a vital cash flow to the people who live at subsistence level. Besides the fibre, farmers get a substantial quantity of jute sticks, which also have a variety of applications such as fuel, fencing and particle boards.

The importance of jute in the national economy of the producing countries, especially Bangladesh, can hardly be overemphasized. The jute sector in Bangladesh provides about 4% of gross domestic product (GDP) and has a 4.07% share of the country's national export earnings.

Moreover, jute occupies the land for fibre production for only 4–5 months and is commonly rotated with food crops including rice and potatoes. Multiple cropping gives increased agricultural production, tends to maintain and improve soil fertility and reduces the incidence of weeds and plant diseases. The inclusion of jute in the cropping system enhances soil organic matter through leaf shedding during the growing season and improves soil nutrient availability by breaking the sub-soil hard pan caused by continuous cropping of rice.

3.2 Types of jute

Although there are several types of jute plant which yield fibre, only two species are cultivable types and the others are mostly wild types, bushy and dwarf.

3.2.1 Taxonomy

Jute is an annual plant belonging to the genus *Corchorus* of the family Tiliaceae, and also belongs to the sub-order Malvineae of the order Malvales. The number of *Corchorus* species is probably around 50–60, however, over 170 *Corchorus* names are given in the Index Kewensis. The genus is extremely variable, but all species are apparently highly fibrous.

3.2.2 Main species

Out of over 30 important species belonging to the genus *Corchorus*, only two – *C. capsularis* and *C. olitorius* – are utilized for fibre production on

a commercial scale. *C. capsularis* is commonly known as 'White jute' and *C. olitorius* is known as 'Tossa jute'.

3.2.3 Botanical description

Jute is an annual herbaceous dicotyledonous plant that grows to a height of 1.5–4.5 m. The stems are about 1–2 cm in diameter with few branches. The colour of the stem, petiole, leaf and pod varies in different forms. Jute fibre is obtained from the bast or phloem layer of the stem. The two species differ in the quality of fibre they yield. Fibres of *C. olitorius* are frequently softer, stronger and more lustrous than those of *C. capsularis*.

3.2.4 Cultivation

Jute is grown in a wide range of soil types, mainly alluviums, laterite and calcareous with soil texture varying from sandy loam to clay loam. Basically, the soil should be well-drained, and its pH should preferably be in the range of 5.5–6.5. White jute is relatively more tolerant to waterlogging especially at later stages of crop growth. Conversely, *Tossa* jute does not tolerate waterlogging and is usually grown on higher lands. In general, both species are more sensitive to waterlogging during the early stage of crop growth.

Jute is propagated by seed. Both in India and Bangladesh sowing is done mostly by broadcasting at seed rates of 7–13 kg/ha for *C. capsularis* and 5–9 kg/ha for *C. olitorius*, though line sowing or planting in lines has many advantages such as 50% lower seed rates, fewer rounds of thinning and weeding, opportunities to reduce costs by utilizing mechanical implements for sowing, thinning and weeding, more convenient harvesting and generally higher fibre yields. Despite these advantages, row cropping is not widely popular and not widely practised by South Asian jute growers (Fig. 3.1).

Organic manure is mainly used to provide nutrients to the jute crop. Usually 6–8 t/ha of farmyard manure are applied to the field during land preparation. Reports indicate that inorganic fertilizers are used by a very small percentage of farmers for jute cultivation, the main reason being that most jute farmers are poor and occupy smallholdings and thus cannot afford to incur the cost of fertilizers. Even when fertilizers are used they are applied in small doses.

Because of the thermosensitive nature of the jute plant it requires an appropriate time for sowing, matching with the temperature and day-length required for optimum growth and development. Soil temperature needs to be 15°C or above for favourable seedling growth.

Raking, thinning and weeding are generally adopted in all jute fields, which are best practised under optimum soil moisture conditions.

3.1 A view of a jute field.

Jute is mostly grown in rotation with other crops. It should be emphasized that jute leaves left on the field improve the soil fertility, and increase yields of the follow-on crop. Many farmers adopt crop rotation practices with a view to minimizing crop losses due to diseases, insects and pests.

3.2.5 Harvesting

The harvesting time of jute is calculated by taking into account the crop age, height and flowering stage, which varies according to the varieties grown and sowing time, but usually 110–120 days are required for jute to mature for harvesting. Jute is generally harvested when the plants are at early pod stage. The fibre remains weak if it is harvested before flowering. On the other hand, the fibre becomes coarse and lacks lustre if harvesting is delayed.

The plants are usually cut close to the ground by hand. A curved, sickle type knife is used to cut the plants. The plants are then sorted out according to height and diameter. The assorted plants are tied into bundles. Each bundle weighs 8–10 kg. The bundles are kept standing in the fields for 3–4 days for defoliation. The bundles of jute plants are carried to ponds, canals, ditches or any other water bodies for retting. Harvesting the plants at the correct time is most important and requires long experience.

3.2.6 Retting

After harvesting and defoliation of plants in the fields for 3–4 days, the jute stems are retted in water and the fibre is extracted. The traditional method is to ret the jute stems for about 15–18 days and extract the fibre manually after retting.

During retting bacteria break down the soft tissues around the fibre bundles and the fibres. Retting is complete when the bark separates out easily from the core. The end-point of retting is a critical stage that largely determines the quality of the fibre. It is difficult to extract the fibre if the plants are taken from the water too early. On the other hand, the fibre is weakened if retting is continued for too long. Before extraction, the farmers make frequent checks to determine the end-point of retting. Correct retting is essentially the first step in the production of good quality fibre.

Conventionally, all the operations of retting followed by extraction of the fibres are done manually to date.

An alternative to this traditonal retting method is to ret the outer ribbons without the woody stick. This is called ribbon retting and widely practised in China. Different retting techniques, ribboners/fibre decorticating machines/decorticators have been developed by different R&D organizations in Bangladesh and India. The ribbons, the outer skins of the stem, are extracted mechanically or manually by these machines.

There are also other methods of retting, chemical, enzymatic/microbiological, etc., advocated by different scientists/researchers.

3.2.7 Extraction

After retting, plants are taken out of water and the fibre is traditionally extracted by hand.

3.2.8 Washing

The washing process consists of holding the extracted fibre bundle by the butt end and jerking it through the water. Clean water is used for washing. The entire dirt, gum, extraneous plant materials and retting residues are removed thoroughly.

3.2.9 Drying

The washed fibre is spread over a bamboo perch or bar for thorough sun drying for 4–7 days before storage. Drying on bare ground is discouraged, because it affects quality of fibre by contamination with dirt, sand, dust particles, etc.

3.2.10 Fibre quality and grading

The main parameters of fibre quality include colour, lustre, strength, texture, length and presence or absence of root cuttings, the hardy reeds basal portion of fibre, etc.

The outcome of desired attributes or undesired defects is dependent on various factors such as cultivation practices, diseases and pests, and retting. Agronomic manipulation in terms of balanced fertilizer, maintenance of desired plant population and yield, adoption of a new cropping system approach, integrated pest management and improvement of retting facility are suggested for quality improvement of jute.

3.2.11 Grading systems in different countries

In Bangladesh, based on the major quality attributes, fibres are graded by two methods for commercial purposes. One is called the *Kutcha* grading method, where fibre is categorized into five grades: Top, Middle, B-Bottom, C-Bottom and X-Bottom in descending order of quality mainly for the local markets. The other method is called *Pucca* grading, where the basal/root parts of the fibres are cut away as cuttings and are categorized into six grades, namely BW-special, BW-A, BW-B, …, BW-E for White jute and BT-special, BT-A, BT-B, …, BT-E for Tossa jute in descending order of quality, mainly for the international market.

In India depending on the six fibre quality attributes – namely strength, fineness, defects, root contents, colour and density – jute is graded into eight White (*C. capsularis*) grades, that is, $W_1, W_2, \ldots, W_8$ and eight Tossa (*C. olitorius*) grades, that is, $TD_1, TD_2, \ldots, TD_8$ in descending order of quality.

Similarly, different grading systems are used in different countries, for example, a grading system with 4 grades in China, with 3 grades in Indonesia, with 2 grades in Nepal, and with 3 grades in Thailand.

3.3 Fibre morphology

Jute fibre, unlike cotton, is a multicellular fibre. In the jute plant the fibre is formed as a cylindrical sheath made up of single fibres (ultimate cell) joined together in such a way as to form a three-dimensional network from top to bottom of the stem.

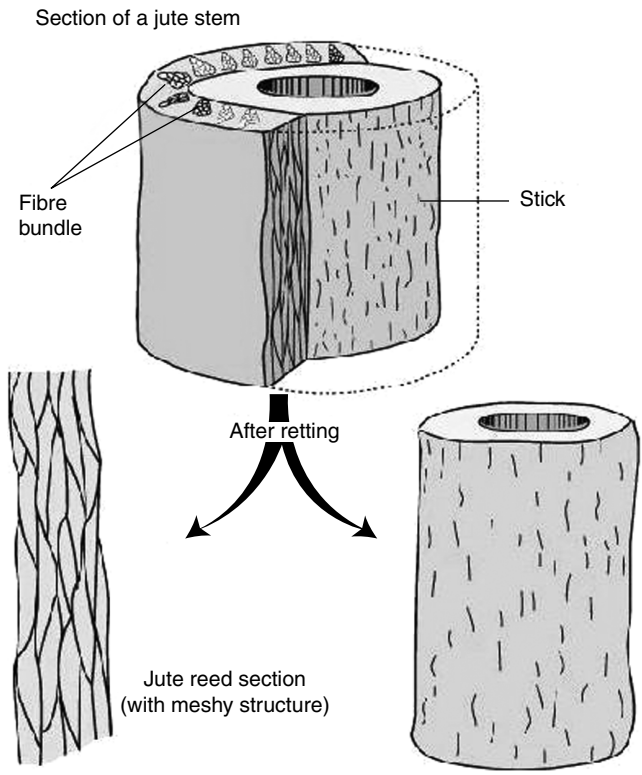
The commercial fibres, in the form of fibre bundles of 1.5–3 m long, called *reed*, are held together as a unit by the meshy or network structure of the fibre elements and represents only a very small proportion (4–6%) of the whole plant.

3.3.1 Macrostructure

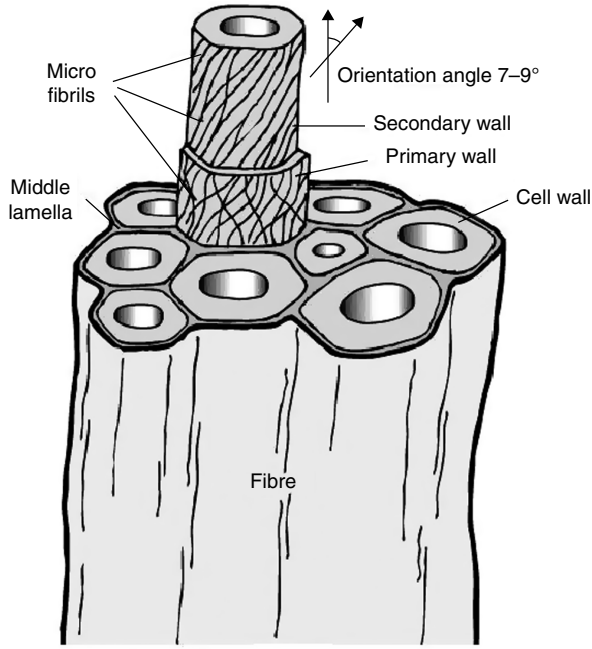
Each fibre element of these meshes of a raw jute reed is basically a group of *ultimate cells*, cemented together laterally and longitudinally by means of inter-cellular materials being chiefly non-cellulosic in composition. A single fibre of jute thus comprises a bundle of ultimates. Thus, jute fibre is multicellular.

The ultimate cells are spindle-shaped and of variable size in length and width, being on average 2.5 mm long and 0.02 mm width at the middle. The cells are some 200 times longer than their breadth.

The cross-sections of the ultimate cells are found to be polygonal with rounded corners. The layer of natural gum present between the ultimate cells is known as the *middle lamella.* Each ultimate cell has thick cell wall and *lumen*, the central canal, with a more or less oval cross-section (Fig. 3.2).



3.2 Disintegration of a jute stem into a jute reed and a stick after retting.



3.3 Jute fibre morphology: microstructure.

3.3.2 Microstructure and appearance

The cell wall of each ultimate cell is composed of an outer thin *primary wall* and an inner thick *secondary wall*, differing from each other in the molecular architecture. Both these walls of a jute ultimate cell are composed of ultra-fine *microfibrils* (Fig. 3.3).

While in the primary wall the fibrils lie in a criss-cross manner, the fibrils are arranged almost parallel as right-hand spirals in the secondary wall. The fibrils in the jute cell wall are arranged in a right-handed spiral with angle of orientation of 7–9° in reference to the cell axis.

Within the ultimate cells of a jute fibre, the ultrafine fibrils, being purely cellulosic, are the highly ordered regions, while the inter-fibrillar regions are less ordered regions which can make room for the presence of short chain hemicellulose molecules to a larger extent and the bulky lignin molecules to a smaller extent as the bonding material of the middle lamella, providing strong lateral adhesion between the ultimates.

3.4 Chemical composition

The major aspects related to the chemistry of jute and its composition have been described below.

3.4.1 Description

Jute fibre is basically a compound of lignocellulose. It is a complex of organic molecules, which on combustion leaves a little ash consisting of calcium, magnesium, aluminium, iron, etc., that are present either in the free state or bonded with functional groups of cellulosic chain.

The number of ultimate cells in one such bundle constituting a single fibre ranges from a minimum of 8–9 to a maximum of 20–25. This wide variation in the number of cells in a bundle is believed to be a major cause of variation in the physical and mechanical properties of the fibre and its quality.

The spinnable units in jute fibre strands are, like those in most other bast fibre crops, filaments composed of a string of cells bonded together by pectin and hemicelluloses.

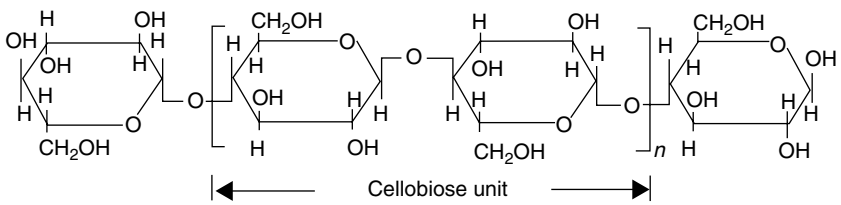
3.4.2 Chemical structure

Chemically, jute fibre is mainly composed of polysaccharides and lignin. The fibre also contains smaller amounts of chemical compounds such as fats and waxes, pectin, nitrogenous, colouring and inorganic matters. The polysaccharides are also called carbohydrates (or holocellulose), and are divided into two groups: alphacellulose and hemicellulose.

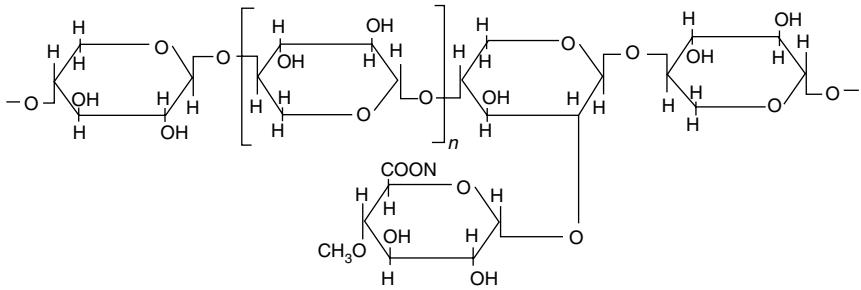
Structure of alphacellulose

Alphacellulose is the major constituent of jute. It forms the skeletal structure of jute fibre and belongs to the family of the compounds of carbohydrates. It contains 44.4% carbon, 6.2% hydrogen and 49.4% oxygen. Its molecular formula is expressed as $(C_6H_{10}O_6)_n$. The chemical structure of alphacellulose is shown in Fig. 3.4.

Alphacellulose is a natural polymer of the poly-condensation kind consisting of D-anhydroglucopyranose units linked together in the chain molecule by 1, 4 β-glycosidic bonds. The alphacellulose is a long chain polymer. The degree of polymerization (DP) of an alphacellulose chain is about 10 000 if we consider the 'glucose unit' as the monomer or about 5000 if the 'cellobiose unit' is considered to be the monomer.



3.4 Chemical structure of alphacellulose.



3.5 Chemical structure of hemicellulose.

Structure of hemicelluose

The hemicellulose in jute is a complex mixture of polysaccharides and polyuronides. It is formed by a number of comparatively low molecular weight polysaccharides of various sugar units, namely xylan in the pentosan, galactan in the hexosan, araban, rhamnosan, mannose, etc. Polysaccharide hemicellulose contains a large number of hydroxyl groups and also carboxyl groups. The hemicelluloses are short chain linear polymers. The molecular weight of hemicellulose is 26 000 (determined osmometrically). The chemical structure of hemicellulose is shown in Fig. 3.5.

Structure of lignin

In jute, lignin with a DP of 60, the third major constituent, is a long-chain substance of high molecular weight which, like the hemicelluloses, varies in composition from one type of vegetable material to another. Structurally, the molecule of lignin is a polymer of phenylpropane units consisting of phenolic hydroxyl group, methoxyl group, ether linkages and conjugated double bond in the alpha-position to the benzene ring. Lignin is a complex non-crystalline polymeric constituent which functions as the structural support material in the plant body.

Structure of pectin

A small quantity of pectin is present in the jute fibre. The pectin holds the fibre bundles together. Pectin is a large molecule built up of repeating units of hexuronic acid. It undergoes decomposition during retting to water-soluble products by bacteria.

Mineral and colouring matters

The jute fibres are found to be associated with small amounts of different inorganic metals such as calcium, potassium, magnesium aluminium, iron

Table 3.1 Chemical composition of jute (in % of bone dry weight of the fibre)

	Jute	
Constituents	*C. capsularis*	*C. olitorius*
Alphacellulose	60.0–63.0	58.0–59.0
Hemicellulose	21.0–24.0	22.0–25.0
Lignin	12.0–13.0	13.0–14.0
Fats and waxes	0.4–1.0	0.4–0.9
Pectin	0.2–1.5	0.2–0.5
Proteins/nitrogenous matter, etc.	0.80–1.9	0.8–1.6
Ash	0.7–1.2	0.5–1.2

and silica in the form of their salts. The fibre also contains some pigments and colouring matter. The colouring matter consists of xanthophyll, carotene and tannin. The tannin combines with the iron of the retting water to form iron tannate which imparts a dark grey colour on the fibre surface after retting. They are also present within the cortical cells and in the fibre bundle. The minerals and the colouring matters are removable from the fibre by chemical means such as treatments with dilute mineral acids, bleaching agents, etc.

3.4.3 Chemical composition of jute

The retted jute fibres consist of alphacellulose, hemicellulose and lignin along with some minor constituents. The chemical composition of jute fibres is given in Table 3.1.

3.5 Properties of jute

Commercial jute ranges from pale cream to golden yellow and from light brown to dirty grey in colour. It possesses a natural silky shine. Jute is a relatively coarse, stiff, inelastic and somewhat rigid fibre that has slightly higher moisture regains (12–13%) than cotton (7–8%). Good frictional property, tenacity, very high modulus and low breaking elongation make jute an ideal packaging material.

The use of jute is limited to coarse fabrics, because the length/diameter ratio of jute filaments is only 100–120, which is much below the minimum of 1000 required for fine spinning quality. Jute fibre is hygroscopic and wetted filaments may swell up to 23% in diameter.

Other than being of agro-origin (and hence renewable) and biodegradable (and hence environmentally friendly), the major advantageous features of jute are its high strength and initial modulus, moderate moisture regain,

good dyeability using different dyes, good heat and sound insulation properties and low cost. However, the major disadvantages of jute are its coarseness, stiffness, low wet strength, moderate wash shrinkage, harsh feel, hairiness and high fibre shedding, photo-yellowing, and poor crease recovery.

3.5.1 Physical properties

Like other bast fibres, jute is a strong fibre with very low extensibility due to its '*composite-like*' structure with highly oriented long chain molecules. The jute fibre shows very poor extensibility, the breaking elongation ranging between 1.0% and 1.8%. Tossa jute is found to be stronger than White jute. Tenacity-wise, the jute filament is comparable with steel.

The flexural and torsional rigidities of jute fibre are quite high as compared to cotton or wool due to its coarseness and inelastic structure. For this, jute textile material develops wrinkles and creases easily (Table 3.2).

3.5.2 Thermal properties

Heating at very high temperature, jute fibre chars and burns without melting. With increasing heat, the chain molecules vibrate more increasingly and ultimately disintegrate leading to violent chemical reactions observed as fibre combustion. With a high specific heat value as of 1.36×10^3 J/kg/K the jute fibre shows good thermal insulation. The ignition temperature of jute, about 193°C, is not high and may be one of the reasons making jute susceptible to catching fire.

Like all cellulosic fibres, jute also loses strength on prolonged exposure to sunlight. Jute loses strength at more than twice the rate for cotton.

3.5.3 Electrical properties

Dry jute exhibits high electrical resistance but in moist jute, the electrical resistance drops by about 10 000 times. Such variation of electrical resistance with moisture led to the development of an electronic moisture meter. The dielectric constant of jute at a frequency of 2 kHz is 1.8 in the dry state, 2.4 at 65% relative humidity (RH) and 3.6 at 100% RH.

3.5.4 Optical properties

The colour of jute fibres varies widely from pale creams/yellow to dark brown. In general, the Tossa variety tends to have a reddish tinge while the colour of White jute varies from pale yellow to yellow. Good quality jute shows excellent lustre. Jute fibres with good molecular orientation show

Table 3.2 Physical properties of jute

Physical properties (macro and micro structure)	
Ultimate cell length (L)	
Average	2.50 mm
Range	0.8–6.0 mm
Ultimate cell breadth (B)	
Average	18 μm
Range	10–25 μm
L/B ratio (average)	110
Fibre fineness	1.3–4.0 tex
Fibre length (after carding)	2–50 cm
Density	
True	1.46 g/cc
Apparent	1.10–1.34 g/cc
Bulk density	0.4–0.5 g/cc
Degree of crystallinity (X-ray)	55–60%
Angle of orientation (X-ray)	7–9°
Moisture absorption	
Moisture regain	
At 65% RH	13.8%
At 100% RH	36.0%
Transverse swelling in water	
Diameter-wise	20%
Cross-sectional area-wise	45%
Water holding capacity	500%
Mechanical properties	
Tenacity	
Single (gauge length – 1 cm)	30–50 gf/tex; 0.29–0.48 N/tex
Bundle (gauge length – 5 cm)	12–35 gf/tex; 0.18–0.34 N/tex
Elongation at break	1.0–1.8%
Initial modulus	1700–3000 gf/tex; 17–30 N/tex; $0.9–1.7 \times 10^{11}$ dynes/cm^2
Specific torsional rigidity	0.4–0.5 mN · mm^2/tex^2
Specific flexural rigidity	0.7–0.8 mN · mm^2/tex^2
Elastic recovery:	
From 3 g/den stress	75%
From 1.5% strain	75%
Specific work of rupture	2.7 mN/tex
Work factor	0.5
Thermal properties	
Specific heat	1.36 × 103 J/kg/K
Thermal conductivity	427.3 mW/m/K
Heat of combustion	17.5 J/g
Ignition temperature	193°C
Heat of wetting	18.2 Cal
Electrical properties	
Dielectric constant (at 2 kHz)	
Dry	1.8
At 65% RH	2.4
At 100% RH	3.6

(*Continued*)

Table 3.2 Continued

Insulation resistance	10^{14}–10^{17} ohm; depending strongly upon RH
Electric strength	500 kV/cm
Optical properties	
Refractive index	
Parallel to fibre axis	1.577
Perpendicular to fibre axis	1.536
Birefringence	0.041
Frictional properties	
Coefficient of inter-fibre friction	
Tossa jute	0.45
White jute	0.54

Note: RH, relative humidity.
Source: Sur (2005).

good amount of birefringence, +0.041, the difference between the refractive index along the fibre axis and that perpendicular to it, due to high orientation of chain molecules.

3.5.5 Frictional properties

The higher coefficient of friction in the case of White jute may be attributed to its better fineness and higher meshiness as compared to Tossa jute.

Jute fibre shows increasing friction with increase in moisture regain. The removal of fat and wax from the fibre surface also increases the friction. However, the batching oil helps to reduce the friction between jute fibres and metallic pins during carding and drawing stages undertaken for jute yarn spinning. Lower inter-fibre friction is desirable for reduced drafting-related irregularity but higher inter-fibre friction helps to develop good yarn strength.

3.5.6 Chemical properties

Jute fibre is an acid fibre and contains a fairly large amount of hemicellulose (21–25%) having acidic group and lignin (12–14%). The fibre is susceptible to the action of alkali. Even a mild treatment with alkali reduces the wet strength of the fibre considerably. The treatment weakens the inter-cellular cementing materials to hold together the short ultimate cells of the fibre. The acidity of the fibre is due to carboxylic group and phenolic hydroxyl group. By virtue of carboxylic acid, the fibre binds cationic ash minerals and possesses strong affinity for basic dyes such as methylene blue. The chemical properties of jute fibre chiefly arise due to the major three integral

constituents alphacellulose, hemicellulose and lignin which comprise more than 97% of the total jute constituents.

3.5.7 Effect of chemicals on jute

Effect of alkali

Jute fibres have poor resistance to alkali due to presence of hemicelluloses. Alkali treatment extracts out hemicellulose from the jute fibre structure making it weak. With treatment of strong (18%) alkali, jute fibres become much weaker but develop crimp due to irregular swelling, a process popularly known as the woollenization of jute.

Effect of acid

Jute fibres are weakened and destroyed by acids. The cellulose chains disintegrate due to hydrolysis of the glucoside oxygen atom (which joins glucose units to form a cellulose chain). Inorganic acids have a more adverse effect on jute than organic acids as they hydrolyse cellulose chains more rapidly.

Effect of bleaching agent

The most common bleaches used for jute are sodium chlorite and hypochlorite. Sodium chlorite/hypochlorite dissolves out lignin from jute making it whiter as lignin is responsible for the natural yellow to reddish colour of jute. Due to partial removal of lignin, bleaching treatment makes jute weaker as the middle lamella (major location of lignin) between the ultimate cells gets weakened. Due to this, jute fibres also become finer on partial disintegration of the multicellular structure

3.6 Typical applications

Jute and its products are being used historically in a number of ways. The conventional or traditional uses of jute along with some unconventional and newly developed innovative uses of jute have been described below under the section.

3.6.1 Conventional/traditional applications

The jute industry is about 150 years old and its products met the global share of packaging successfully though it had ups and downs. Traditionally jute has been used to manufacture packaging materials like hessian, sacking, ropes, twines, carpet backing cloth, etc.

The large historical markets for these traditional products of jute have decreased over the years as it has been replaced by synthetics.

Jute has been the most widely used packaging fibre for more than 100 years because of its strength and durability, low production costs, ease of manufacturing and availability in large and uniform quantities. Other traditional products include hessian cloth, carpet backing, yarn, twine, cordage, nonwoven felts and jute carpets.

More than 50% of the jute consumed annually is manufactured into sacks and bags for transportation and storage of agricultural and industrial commodities. Apart from this, jute is also used on a large scale for twines, carpet yarns, cloth backings for linoleum and carpets, etc., as well as for webbing, covering of cables, and a host of other purposes of conventional use.

3.6.2 Unconventional/diversified applications

The steady decline in markets for traditional jute products during the 1970s–1980s forced governments and the jute industry to take up programmes for development of new jute products over the last few decades.

New technologies have been evolved for bulk use of jute as a raw material in the production of high value-added and price competitive intermediaries or final products. A host of innovative new products have been developed with high value-addition such as home textiles, jute composites, jute geotextiles, paper pulp, technical textiles, chemical products, handicrafts, fashion accessories, etc. Thee products from new, alternative and non-traditional uses of jute are generally termed diversified jute products.

Growing consciousness about the use of renewable resources, and the need for environment friendly, energy-saving materials and processes in the industrial sector, has changed the focus and emphatically renewed world interest in jute and allied fibres. They are valued not only in the traditional fields of application but also in many other new and diverse application areas.

Jute is a versatile natural fibre finding a wide range of new applications in fabrics for furniture, upholstery, soft luggage, fancy bags, wall coverings, laminated sheets for packaging boxes and panelling, fibre reinforced composites for automobile, construction and building materials, home textiles, technical textiles, geotextiles, floor coverings, blankets and semi-apparels, nonwovens, insulation materials, asbestos replacing materials, particle boards, shopping bags, handicrafts and fashion accessories, etc.

Whole jute stems are suitable as raw material for paper pulp. The woody central core or 'jute stick' remaining after removal of the bast is used as a rural building material, for thatch, fences, fuel and for charcoal-making. It can also be processed into paper, board and cellulose derivatives.

The leaves and tops of *C. olitorius* are eaten as a vegetable, for instance in Malaysia, Indonesia and the Philippines. In Africa and the Middle East

C. olitorius is mainly grown and used for this purpose, and not for its fibre. The leaves of *C. capsularis* are applied medicinally in Peninsular Malaysia to poultice sores, and in decoction they are used to treat dysentery, phthisis and coughs, and as a tonic for children. In the Philippines the leaves of *C. capsularis* are used to treat headaches.

A great deal of research is presently going on in each of these fields; however, the largest potential markets are in composite products. These composites range from value-added specialty products to very large-volume commercial materials. These markets are potentially larger than the past markets for jute and could lead to new dynamic uses. Technical textiles, namely geotextiles, especially for soil erosion control and rural road construction are another area of potential use in huge volume.

3.7 Conclusions

A few success stories illustrate that jute, the golden fibre, could make a comeback. For example, a jute mill in Bangladesh recently reduced its losses by developing and introducing linoleum fabric, which is being used as an industrial material. Some manufacturing units of India are profitably making attractive bags and diversified jute products including shopping bags. It is perceived that demand for home textiles, particle board, jute-based composites, technical textiles, etc., is increasing. People around the world are becoming more conscious about the pollution caused by synthetics and are increasingly opting for natural fibre products.

It is evident that worldwide use of more traditional jute products and new, alternative and non-traditional items together with the diversified jute products would certainly rejuvenate the jute sector and would reduce pollution to a great extent. It is likely that development of the diversified sector would provide more employment opportunities and alleviate poverty.

3.7.1 Environmental advantages

Jute is a natural fibre and an annually renewable resource with high biomass production per unit land area. Moreover, leaf and crop waste, left out in the field, is transformed into organic materials, thereby reducing demand for supplementary chemical fertilizers for subsequent crops. In terms of global warming, a concern of great importance in the modern world, jute and jute products are proven to be absolutely harmless.

The following are among its numerous environmental advantages:

- In the 100 days of the jute-growing period, one hectare of jute plants can absorb about 15 MT of CO_2 from the atmosphere and liberate about 11 MT of O_2.

- Jute plants purify air and have carbon dioxide (CO_2) assimilation power several times higher than that of trees.
- The environmental impacts of jute production are much less harmful since in jute agriculture the use of chemical fertilizers, pesticides and weed killers/fungicides is scanty.
- The biological efficiency of jute is much higher than that of wood as it is a fast growing annual crop that takes only 4–5 months to mature.
- Wastes from jute production are biodegradable and can always be used as manure.
- Jute products require only 7% of the energy required for the production of its synthetic counterparts.
- Total energy consumption during jute agriculture and jute sack production is 10.0 GJ per MT of jute fibre.
- Jute is biodegradable and therefore does not cause any environmental pollution during disposal.

3.7.2 Socio-economic impact

It is worth mentioning that the subsistence farmers still grow jute mainly using family labour. So they can afford to continue with jute production. This crop is an important source of cash from the sale of jute fibres for the farming community. The farmers also need jute sticks for their domestic purposes.

There is a dilemma, however, for the producing countries in providing the farmers with adequate return while at the same time satisfy the requirements of the world market.

Farmers and processors also face technical and economical problems in cultivating and processing fibre crops. Information and research can solve technical problems, such as lack of expertise in cultivation of fibre crops, ignorance of new extraction methods, poor harvesting machinery, and fibre quality inconsistency. Economic problems are the price level of plant fibres, inconsistency of prices, competition with other fibres, limited and small markets, uncertain financial returns, required capital investments, and storage and transportation costs. The economic problems may be reduced when the costs of harvesting and processing become lower due to simpler methods and shorter production-trade chains or productivity is increased along with fibre quality.

3.8 Sources of further information and advice

Further information may be available from the IJSG website: www.jute.org and its publications uploaded in the site.

3.8.1 Key books

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- Sur, D. (2005), *Understanding Jute Yarn*. Kolkata, India: Anindita Sur.

3.8.2 Major trade and professional bodies

The major jute trade associations/bodies are provided below with their e-mail addresses or other contact numbers.

- Bangladesh Jute Association (BJA), Dhaka, E-mails: bjute@bangla.net, beejay@bangla.net
- Bangladesh Jute Exporters Association (BJEA), Dhaka, Tel: ++880 2 955 2910, 955 1515
- Bangladesh Jute Goods Association (BJGA), Dhaka, Fax: ++880 2 955 0664
- Bangladesh Jute Mills Association (BJMA), Dhaka E-mail: bjmajute-good@agnionline.com
- Bangladesh Jute Spinners Association (BJSA), Dhaka, E-mail: bjsa_bd@yahoo.com

- Burlap and Jute Association (Association du Jute), Ohio, USA, E-mail: sspiegel@daybag.com
- European Association for the Trade in Jute and Related Products (EUROJUTE), Germany, E-mails: info@wgc.de; corchorus@wgc.de
- Gunny Trades Association (GTA), Kolkata, Tel:++91 33 220 3233
- Hanoi Jute Company, Hanoi, Vietnam, E-mail: hanoijute@fpt.vn
- Indian Jute Mills Association (IJMA), E-mail: ijma@cal2.vsnl.net.in
- Japan Jute Products Import Council (JJPIC), Osaka, Japan, Tel: ++81 6 227 1841, Fax: ++ 91 6 202 5585
- Jute Baler's Association, Tel: ++91 33 2220 2805
- Myanma Jute Industries (MJI), Yangon, Myanmar, E-mail: mji@myanmar.com.mm
- Nepal Jute Mills Association (NJMA), Biratnagar, Nepal, E-mails: rkg@golchha.com; swastik@cworld.com.np; brt@golchha.com; sanjay@golchha.com
- The Pakistan Jute Mills Association (PJMA), Lahore, Pakistan, E-mails: secretary@pjma.com; info@pjma.com.pk; azhar@habib-jute.com; pjma2000@yahoo.com

3.8.3 Research and development organizations and groups

The important R&D organizations/interest groups for jute are listed below.

- Bangladesh Jute Mills Corporation (BJMC), Dhaka, E-mail: bjmc@bttb.net.bd
- Bangladesh Jute Research Institute (BJRI), Dhaka, E-mails: info@bjri.gov.bd, infobjri@yahoo.com
- Central Research Institute for Jute & Allied Fibres (CRIJAF), Barrackpore, Kolkata, India, E-mail: crijaf@wb.nic.in
- Indian Jute Industries' Research Association (IJIRA), Kolkata, India, E-mails: director@ijira.org; ijiraweb@ijira.org
- Institute of Bast Fiber Crops (IBFC), Hunan Province, China, E-mails: ibfczyx@163.com, jgsu@vip.163.com
- Institute of Jute Technology (IJT), Kolkata, India, E-mail: ijt@cal2.vsnl.net.in
- Jute Corporation of India Limited (JCI), Kolkata, India, E-mail: jutecorp@vsnl.net
- Jute Diversification Promotion Centre (JDPC), Dhaka, E-mail: edjdpc@yahoo.com
- Jute Manufactures Development Council (JMDC), Kolkata, India, E-mails: jmdc@jute.com; jmdcindia@vsnl.com

- National Centre for Jute Diversification (NCJD), Kolkata, India, E-mail: ncjd@vsnl.com
- National Institute for Research on Jute and Allied Fiber Technology (NIRJAFT), Kolkata, India, E-mail: nirjaft@wb.nic.in
- South India Textile Research Association (SITRA), Coimbatore, India, E-mails: sitra@vsnl.com; sitraindia@dataone.in

3.8.4 Websites

www.jute.org; www.motj.gov.bd; www.Juteenterprisebd.com; www.bjmc.gov.bd; www.bjri.gov.bd; www.bjsa.org; www.texmin.nic.in; www.jmdcindia.com; www.jute.com; www.juteworld.com; www.iuteenterprise.in; www.ijira.org; www.ijtindia.org; www.sitraindia.org; www.crijaf.org.in; www.nirjaft.res.in; www.jutecorp.com; www. worldjute.com; www.jute-industry.com/ www.pjma.com.pk; www.eurojute.com; www.iwn.inf.poznan.pl

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4

Bast fibres: ramie

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Abstract: Ramie is one of the oldest fibre crops in the world. Ramie fibre is also one of the world's strongest and longest natural fine textile fibres. China, mainly the central and southern part, leads the world in the production of ramie. Ramie is a perennial plant belonging to the genus *Boehmeria* under the Urticaceae or Nettle family of the order Urticales and class Magnoliopsida. There are about 100 species under the genus *Boehmeria*. This fibre is of secondary importance in world trade despite its unique characteristics. This is mainly due to the lack of suitable large-scale fibre extraction equipment until recent years and costly methods of degumming, spinning and weaving of the fibre. This chapter discusses different aspects of ramie in brief, including its origin, history, climatic requirements, production area, economic importance, types of ramie, taxonomy, botanical description, cultivation, harvesting, fibre extraction, fibre quality and grading, fibre morphology, chemical composition, properties, typical applications, etc.

Key words: perennial plant, genus *Boehmeria*, Urticaceae or Nettle family, order Urticales, class Magnoliopsida, degumming, spinning and weaving.

4.1 Introduction to ramie

Ramie is one of the oldest fibre crops, having been used for at least 6000 years. Ramie fibre is one of the strongest and longest natural fine textile fibres in the world. It is a bast fibre derived from the bast layer of the stem, that is, phloem of the vegetative stalks of the plants.

4.1.1 Origin

This dicot, angiosperm, semi-perennial shrubs of the Nettle family Urticaceae is a native of the Far East and probably originated in the mountain valleys of southwestern China.

4.1.2 History

Ramie is reported to have been used in mummy cloths in Egypt during 5000–3000 BC. Since prehistoric times, ramie has been used in China, India

and Indonesia. It was used for Chinese burial shrouds over 2000 years ago. Ramie was mentioned and praised as grass cloth in the Sanskrit poems of *Kalidasa* and *Ramayana*. It was used in the south of Russia about 900 BC. It is said to have been grown in China for many centuries and was one of the principal fibres used in ancient China for making cloth previous to the introduction of cotton around AD 1300. Ramie was first introduced from the East Indies to Holland in 1733, France in 1844, Germany in 1850, England in 1851 and Belgium in 1860. In 1857 ramie plants were introduced into the United States from Java and planted in the Botanical Gardens in Washington.

Ramie fibre, also known as China grass, grass linen or Chinese silk, was exported by China to the Western world at the beginning of the eighteenth century. However, it was not until 1930 that ramie textile production was established on a commercial basis in Western Europe. Commercial ramie production in Brazil first began in the 1930s with production peaking in 1971. In Japan and the Philippines concentrated efforts were made to produce ramie during the Second World War. With the establishment of sizeable ramie acreages in south Florida, commercial scale processing equipment was developed and operated successfully from 1946 to 1955. Ramie's popularity actually increased in the mid-1980s with the fashion emphasis on natural fibres.

4.1.3 Adaptation/agro-climatic conditions

Ramie is adapted to a wide range of latitudes from almost equatorial conditions to about latitude 45°N in Russia. In the temperate areas, between latitudes 25° and 38°N or S, ramie may produce two to three crops annually. In sub-tropical areas, between 20° and 25°N or S, four to five crops may be harvested, usually with supplementary irrigation. At latitudes below 10°N or S, it may be possible to harvest six or more crops annually, but irrigation must be provided for 2–3 months when there is insufficient rainfall. Ramie has been found to grow well in humid climates under moderate temperature. It requires a uniformly well distributed rainfall of 1500–3000 mm annually. The optimum temperature for good harvest is around 20–31°C and the relative humidity should be at least 25%. However, the crop is sensitive to waterlogging, frost and strong winds.

4.1.4 Areas of production

China, mainly the central and southern part, leads the world in the production of ramie. Other major producers of ramie fibre are Japan, Taiwan, Brazil, the Philippines, Korea, Indonesia and India.

4.1.5 Economic importance

Ramie, the longest and one of the strongest fine textile fibres, has been grown experimentally throughout the tropical, subtropical and temperate zones of the world.

Ramie has been proved quite remunerative when grown under favourable edapho-climatic conditions. The income generally starts from the second year and continues thereafter. Fibres up to 4–5% by weight of total biomass may be obtained from ramie. Raw fibre yield up to 1.6–2.2 ton per hectare may be harvested per year under ideal conditions.

However, it is of secondary importance in world trade despite its unique characteristics. This is mainly due to a lack of suitable large-scale fibre extraction equipment until recent years and costly methods of degumming, spinning and weaving of the fibre.

Only a small portion of the ramie produced is available in the international market to be imported mainly to Japan, Germany, France and the UK.

4.2 Types of ramie

Different types of ramie of different quality originates from *Boehmeria* species of different habitats, some being herbs, some others are trees or shrubs.

4.2.1 Taxonomy

Ramie is a perennial plant belonging to the genus *Boehmeria* under Urticaceae or Nettle family of the order Urticales and class Magnoliopsida. There are about 100 species under the genus *Boehmeria*.

4.2.2 Main species

Boehmeria nivea L. Gaud., commonly known as 'white ramie' and *B. utilis* generally referred to as *B. nivea* var. t*enacissema* and also known as 'green ramie' are the two major species of ramie fibre. This green ramie is probably a hybrid of white ramie.

The true ramie or China Grass also called 'white ramie' is the Chinese cultivated plant. The second type, the 'green ramie' or rhea is believed to have originated in the Malay Peninsula.

The two varieties differ in their habitat: whereas *B. nivea* grows best in temperate and sub-tropical regions, green ramie occurs in tropical regions. Green ramie or *B. utilis* is said to give higher fibre yield and stronger fibre

than *B. nivea*, although the fibre quality is not so good in regards to its fineness and colour.

4.2.3 Botanical description

Ramie is the only member of the family used commercially for fibre production and grows well in warm climates. It is a type of shrub. The plant is an erect, usually non-branching, tall, fast-growing herbaceous perennial reaching 1–2.5 m in height at maturity. The leaves of ramie are heart shaped, 7–15 cm long and 6–12 cm broad, and white on the underside of white ramie and green on the underside of green ramie with dense small hairs – this gives a silvery appearance; unlike nettles, the hairs do not sting.

4.2.4 Cultivation

Fertile, deep loam and well-drained sandy loam soils are suitable for ramie cultivation. Soil rich in organic matter with an optimum pH range from 6 to 7 is preferred (Fig. 4.1).

Since this is a perennial plant, plantings are mainly made from asexual plant materials. The seeds of ramie are used only for selection work, since

4.1 A view of a ramie field.

the seedlings rarely develop into plants having desired qualities. Most ramie seedlings are almost unsuitable for fibre. Ramie, thus, may be propagated by the following four methods, in order of importance: (1) rhizome cuttings, (2) division of parent rootstock, (3) laying and (4) stem cuttings.

4.2.5 Harvesting

Ramie, being a perennial plant, is normally harvested two to three times a year but under favourable growing conditions it can be harvested up to six times per year.

Ramie plants produce a large number of unbranched stems from underground rhizomes and have a crop life of 6–20 years. Thus harvesting of ramie can be done intermittently for up to 20 years.

Ramie should be harvested just before flowering or soon after flowering starts. The time of harvesting is important for getting the best fibre and also to obtain maximum fibre yield from the plants. Stems are harvested by either cutting just above the lateral roots or bending the stem.

4.2.6 Fibre extraction

The extraction of ramie fibre occurs in three stages. First the cortex or bark is removed just after harvesting while the plants are still fresh, by hand or machine. This process is called decortication. Present-day decortication is vastly mechanized. Second, the cortex is scraped to remove most of the outer bark, the parenchyma in the bast layer and some gums and pectins. Finally, the residual cortex material is washed, dried and degummed to obtain the spinnable fibre.

4.2.7 Degumming

Degumming, the next step, is the process of removal of gum from the decorticated fibre. About 20–30% of natural adhered gums, holding the fibres in dense strands, are removed so as to make the fibres suitable for spinning and weaving. Degumming may be done by different methods, namely chemical method, microbial method, etc.

4.2.8 Fibre quality and grading

Ramie fibre generally is graded according to length, colour and cleanliness. Top grades usually are washed and sometimes brushed. There is no standard set of grades for ramie fibre, but several countries have set up their own grades.

Eight ramie grades are established in China, 4 in Japan, 3 in Brazil, and 4 in the Philippines.

4.3 Fibre morphology

Ramie is a multicellular bast fibre, by and large cellulosic in nature, having very little lignin and hemicellulose (Table 4.1). The inter-cellular binding constituents present in significant amounts are natural gums and pectinous matters. The cells of ramie fibre may be as long as 40–45 cm, cylindrical in nature and characterized by thick walls and narrow, curved lumens. The surface of the cell is marked by distinct ridges.

4.4 Properties of ramie

The physical properties of ramie are given in Table 4.2, while the thermal properties are given in Table 4.3. The raw ramie fibre strand has an average length of 0.61–1 m. The longer fibres are sometimes more than 1.5–2 m in

Table 4.1 Chemical composition of ramie

Constituent	Content (%)
Cellulose	68.6–76.2
Hemicellulose	13.1–16.7
Lignin	0.6–0.7
Pectin	1.9
Fats and wax	0.3
Other extractives	6.1

Table 4.2 Physical properties of ramie

Property	Value
Length, L (mm)	60–250
Breadth, B (μm)	15–80
Width (μm)	50
L/B ratio	3500
Fineness (tex)	0.4–0.8
Microfibrillar angle (deg)	7.5
Density (mg/m^3)	1.50
Tenacity (gf/tex)	40–65
Breaking elongation (%)	3.0–4.0
Flexural rigidity (dynes.cm^2)	0.8–1.2
Diameter swelling (%)	12–15
Volume swelling %	32.0
Moisture regain (65–100% RH)	6.5–17.5
Moisture content (wt%)	8.0

RH, relative humidity.

Table 4.3 Thermal properties of ramie

Property	Values
Specific heat	1.36×10^3 J/kg/K
Thermal conductivity	427.3 mW/m/K
Heat of combustion	17.5 J/g
Ignition temperature	193°C
Heat of wetting	18.2 Cal

length. These are not single fibres, rather a bundle of shorter single fibres, as in other bast fibres, held together by gummy and pectinous matters. The elementary cells/single fibres of ramie are longer and thicker than all other bast fibres. Ramie is characterized by its exceptionally long ultimate fibre cells which average to about 150 mm in length and highest length/breadth ratio of ultimate cell (3000).

Properly degummed ramie fibre is the longest and the strongest of all vegetable fibres. It is lustrous, possesses high tensile strength, is extremely absorbent, gains strength appreciably when wet, and is highly resistant to bacteria, mildew, insect attack and rot. Ramie absorbs moisture and gives it up quickly with almost no shrinking and stretching. Thus ramie has very good comfort properties and is suitable for summer clothes. Very low elasticity, low abrasion resistance, lack of resiliency, stiffness and brittleness, necessary degumming process and high cost are some of the weak points of ramie.

Ramie resists action of chemicals better than most of the other natural fibres do. It is dye absorption efficient and fast and has considerable resistance to microbial attack.

4.5 Typical applications

Historically, ramie has been used as a precious material for very fine upper cloth including high grade *Kimono* cloth, especially in Japan. The Korean traditional costume, *Hanbok* – made of ramie – is renowned for its fineness. Fabrics of ramie are lightweight and silky, similar in appearance to linen and suitable for a wide range of garments and home textiles.

Coarse ramie fibres are generally used for making twines and threads, for which its strength and lack of stretch make it most suitable. Because of its high wet strength, quick dryability and considerable resistance to bacterial action, it is very useful for making fishing nets.

Ramie is used in many diverse applications like suiting, shirting, sheeting, dress materials, table cloths, napkins, towels, handkerchiefs, fine furniture upholstery, draperies, mosquito netting, gas mantles, industrial sewing

thread, packing materials, fishing nets, fire hose, belting, canvas, marine shaft packing, knitting yarns, hat braids, filter cloths, etc.

Since ramie has low elasticity and resilience, it is usually blended with other textile fibres like cotton, wool, etc. It increases the lustre and strength of cotton blends and reduces shrinkage in wool blends. It is also blended with silk.

Shorter fibres and waste, called noils, blended with cotton/stapled rayon are used for making low-grade fabrics like dish towels. The noils are also used in the manufacture of high-grade specialty papers such as cigarette paper, bank note/currency paper, etc.

For the 2010 Prius, Toyota will begin using a new range of plant-derived eco-friendly bioplastics made from cellulose in wood or grass, instead of petroleum. One of the two principal crops to be used is ramie.

4.6 Conclusions

The use of ramie is limited by its price and spinning properties. Apart from any economic or technical reasons, the development of any new fibre needs market development. Despite its excellent properties, wide occurrence/cultivation/adaptation of the plant and reasonable publicity, ramie fibre has not been developed or used to the expected level especially outside China and Japan.

Reliable figures relating to production of ramie in China, as the leading ramie producer of the world, are still very difficult to obtain.

The economic factors that prevent farmers from cultivating fibre crops include: price level, inconsistency of prices, competition with cheaper synthetic fibres, too few off-takers, uncertain financial returns, and the required capital investment in machinery. In addition, the production costs of plant fibres are relatively high compared to man-made fibres.

4.7 Sources of further information and advice

Important literature and organizations supplying information on ramie and other fibre crops are listed below.

4.7.1 Key books

- Dempsey, J. M. (1975), *Fiber Crops*. Gainesville, FL: University Press of Florida.
- European Commission (1994), *Industrial Fibre Crops*. Studies by Science Research Development, Agro-Industrial Research Division, EC.
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- Nakamura, A. (2000), *Fibre Science & Technology* (Translated from Japanese). New Delhi: Oxford & IBH Publishing.
- NIIR Board of Consultants & Engineers (2005), *Natural Fibres: Handbook with Cultivation and Uses*. Delhi, India: National Institute of Industrial Research.

4.7.2 Research and development organizations and groups

- Central Research Institute for Jute and Allied Fibres (CRIJAF)
- Institute of Bast Fibres (IBFC), China
- Institute of Natural Fibres (INF), Poznan, Poland
- National Institute for Research on Jute and Allied Fiber Technology (NIRJAFT)**,** India
- South Indian Textiles Research Institute (SITRA), India.

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5

Bast fibres: flax

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Abstract: Flax (*Linum usitatissimum* L.) is the oldest natural fibre used by our ancestors as early as 10 000–8000 BC (Neolithic period), when they changed their way of life from nomadic hunting and gathering to a more sedentary, agrarian style of living. Until the eighteenth century, flax was the dominant fibre in Europe, but later on cotton, cultivated in America and India, began to systematically replace linen. This chapter describes the history of flax and discusses flax plant morphology – the root and rootlets, the wooden cylinder, the holding tissue, structure and chemical compositions of flax stem and fibre. It also describes flax harvesting, decortication of green straw, degumming including water and dew retting, osmotic and enzymatic degumming as well as degumming by using electric resonance technology. The processes of scutching, hackling (combing) and 'cottonization' are described for fibres extracted from flax straw (generally degummed). Information on the development of different kinds of linen spinning is also given. The main areas of application of linen fibres and yarns are presented. Bleaching, dying and dyes and some finishing methods are discussed. The chapter ends by focusing on the main problems arising from the coexistence and competition of linen with other natural and man-made fibres and discusses future trends.

Key words: flax, linen, harvest, water and dew retting, degumming, scutching, hackling, cottonization, natural dyeing.

5.1 Introduction

Various parts of the flax plant (*Linum usitatissimum* L.) – such as seeds, leaves, straw, bast and the woody core – have potential as a source of valuable textile fibres, and are also suitable for technical applications. Flax fibres, applied traditionally for textile utilization such as woven, knitting and technical textiles (described in Chapter 10, vol. 2) have been used for many centuries.

Raw materials derived from flax can also be applied in biopolymers, in aerospace and automotive industries, as well as for insulation, building materials, for production of agro-fine-chemicals, medicines, cosmetics, and for nutrition. Another application of short coarse fibres are: pulp, cigarette paper, packaging, laminates, coatings, particleboards and nonwovens. The

application of nonwovens is very wide ranging from furniture to geo- and chemotextiles (Kozłowski, 2010).

In the last years new achievements have been made in the field of research and development on bast plants (especially flax) such as emerging new varieties, new agrotechnology, new technology for processing, more competition with other fibres and, importantly, widening the area of application of flax. Generally, from an economic viewpoint two types of flax are known in the agro-industry: fibrous flax and oleaginous flax (linseed). Oleaginous flax (called also linseed or oilflax) is cultivated on large areas in Canada, Argentina, USA and India and has been successfully cultivated in Egypt. Its seed crop is much more rewarding than the textile flax crop – it does not need complicated processing operations, in addition to being highly demanded by international linseed markets at increasingly rewarding prices.

The different practical uses of the oleaginous flax crop (linseed) include:

- The oily seeds are crushed to produce linseed oil (edible, and for pharmaceutical and technical uses) and linseed cakes (see Chapter 11, vol. 2: The application of flax and hemp seeds in food, animal feed and cosmetics production).
- Broken capsules make an energizing tonic feed for animals, especially for race horses and for sheep.
- Short broken straws can be used as a convenient raw material for the production of particleboards and composites, as a raw material for the production of particleboards glued by organic glue as well as cement or lime, and as a convenient raw material for the production of other composites.
- Seed capsules can be used, after being transformed into powder with the use of an appropriate hammer mill, as a good cellulose source in poultry feeding and according to recent research to obtain cyclolinopeptides.

5.1.1 Current world demand for flax fibres

The capacity of the world market for flax fibres can currently be evaluated at about 400 000 tons per year and that for linseeds at not less than 2 500 000 tons per year. Both fibres and linseeds are bought at very high prices, as supply is much lower than demand.

At present, there is an urgent demand for textile flax fibres of high quality, suitable for the production of fine yarns, without major spinning problems. They are, in fact, the natural potential characteristics of the pure flax fibres in general, which can be summarized in the following specifications:

- Homogeneous individually separated single filaments of same thickness, strength and suppleness.

- Thickness: diameter not more than 24 microns.
- Strength: tensile strength not less than 60 g per tex.
- Suppleness: smooth, glossy shiny velvety touch, free of rigidity.

5.1.2 History

According to a very old legend, at one time late in the afternoon, a tired traveller while passing by a small lake considered that it was a good opportunity to refresh, renew his water supply and rest for a while under a nearby shady tree. When the traveller lay down on his back under the tree, he noticed a flying bird carrying pieces of straw that it took to the top of the tree. After several trips to and from the tree, the bird started carrying some fluffy shining fibres instead of the straw; the traveller deduced that the bird was finishing its nest by covering its interior with the fluffy fibres, to form a soft base for the future laid eggs. The curious traveller wanted to know the nature of these fluffy fibres and started to follow the bird from the tree to the source of the fibres. The bird landed down near the shallow water and then plunged its head into the lake and came out with a piece of straw and started to repeatedly beat the surface of water with it, until fibres appeared from the adhering pieces of straw. After the bird flew away, the traveller went to the place that the bird had just left, took off his shoes, waded in the shallow water and found the straw lying on the bottom. He took some pieces of the wetted straw and started repeatedly beating them on the surface of the water, and like the bird the traveller obtained several glossy shining fluffy wet fibres. The straw was flax straw, which contained hidden fibres that only became visible and available after the straw remained under water for a sufficient time to expose them, after the separation from other plant tissues that surrounded them.

Thus, it is said that through the natural behaviour of a bird, mankind discovered valuable and useful knowledge about the existence of invisible fibres, hidden inside the flax plant and covered by other plant tissues. These fibres become visible and available for use only after the flax straw remains under water for a specified time, sufficient for their separation from the other plant tissues surrounding them.

This primitive information, enriched with additional personally acquired experience, was gradually transmitted from generation to generation and practically applied, in ponds, rivers and later in tanks, to obtain various flax textile fibres, without real knowledge of the exact details of how this separation takes place and how to successfully control it.

A fragment of white woven cloth wrapped around a primitive tool was discovered by archaeologists in Cayonu (Turkey) near the headwaters of the Tigris river and examined by Gilian Wogelsang-Eastwood, a textile archaeologist at the National Museum of Ethnology in Leiden (The Netherlands). It was confirmed that the woven fragment is the oldest sample of textile ever found – about 10 000 years old – and it is a linen fabric. This fact helped to

prove that our ancestors living in the early Neolithic period (10 000–8000 BC) changed their way of life after millennia of hunting and gathering, and began to cultivate crops, establish villages, and adapting to a more sedentary way of life (Ersserberger, 1993).

The textile samples found in 1962/1963 and examined by M. L. Ryder (1965) were provisionally dated to 6000 BC and were found to be made of flax (Taylor, 1995). The history of flax also goes back to the prehistoric era and Egypt has been considered to be the historical cradle of dual-purpose flax cultivation and of the processing of its straw to produce textile fibres. Many thousands of years BC, the ancient Egyptians at the time of the pharaohs had already mastered flax cultivation (about 2000 ha) and excelled in processing its straw to obtain superior textile fibres that produced ultra-fine yarns, which were used for the production of diaphanous linen fabrics for the higher members of the priesthood and the female members of pharaoh families.

The extra-fine linen found in one of the pharaoh tombs, discovered at the beginning of the twentieth century, showed that the flax yarn used for the production of these very light fabrics is difficult to be attain in modern times. Some sources say that fine and even transparent linen apparels were worn by Egyptian queens and it was revealed that the fine yarn was obtained by enzymatic treatment of saliva in the human mouth.

Egypt's ancient flax technology was transmitted to other countries through Phoenician sailors trading in Egyptian linen. Unfortunately, this flax technology faded and disappeared with time from Egypt. What remains of it are the numerous mural inscriptions on the walls of the pharaonic temples and other monuments referred to in thousands of related papyruses discovered in modern times, but unfortunately still undeciphered.

When Spaniards brought cotton to Europe in the eighteenth century, cotton started to replace linen. Until the eighteenth century flax dominated as a raw material for textiles in Europe. In the nineteenth and twentieth centuries, especially after the First World War, cotton and artificial fibres (viscose) began to replace linen more and more intensively.

During the Second World War, the demand for flax textile fibres increased enormously because the United Kingdom, Germany and the Soviet Union had been almost completely deprived of their usual sources of flax fibre. The German military forces were widely using flax, for the first time in wars, in the manufacture of airplanes, parachutes and incendiary bombs. At that time, airplane wings were made of a solid wooden structure covered by a strong linen cloth. Parachutes' holding bands were made of very strong flax yarns specifically twisted and tightly woven. Linen fire hoses were essential to extinguish the fires caused by incendiary bombs. In response, the United Kingdom had to quickly produce more and more airplanes, parachute holding bands and linen fire hoses, over and above the usual military tarpaulins and other linen camping goods.

To replace and enlarge new sources of flax fibre supplies, the United Kingdom summoned its colonies and Commonwealth countries and other allies to grow as much flax as they could, and to process the straw to produce flax textile fibres. The United Kingdom bought the crop and the production in advance, to cover its military needs in the shortest possible time.

For this purpose, Egypt doubled its cultivation of flax, which necessitated the fast increase of its capability to process the additional flax crops to come. The annually cultivated area of flax in Egypt had always been positively proportional to the demand for fibres and did not exceed l4 000 ha, but during the Second World War, the cultivated area grew to 25 000 ha. The number of flax processing factories reached more than 60.

After the Second World War and since the 1970s many changes occurred. After many centuries of worldwide practice, the conventional method of flax straw retting was found to pollute the environment, and all the European retting centres were officially asked to either neutralize the retting effluents before draining them into the river or close down within a 2-year period. The majority of those retting centres found that neutralization of retting effluents would cost them more than they could afford, and decided to stop working altogether. Since the beginning of the 1980s, tank hot water retting, producing the highest textile grades of flax fibres, ceased in Europe. The reason was that for water retting 1 ton of flax straw needs 30–40 m^3 of water (20 m^3 hot water for retting and 10–20 m^3 water for washing).

European flax straw crops had to shift to the ancestral method of dew retting, which is unable to currently produce selected high textile qualities of flax fibres, like the ones that used to be produced by the more controllable method of tank hot water retting. The presence of high grade flax fibre on the world markets became sporadic and insufficient to cover the demand for such raw material, which was essential to the production of fine counts of flax yarns that sold for high prices.

Dexterous, highly experienced flax spinners almost disappeared and were partly replaced by younger generations that, initially, preferred to discard flax altogether and shift to other easier-to-spin textile raw materials.

Nowadays, however, the many earlier unsolved problems of flax (and other bast fibres) have been satisfactorily solved. These 'vegetal' fibres are now considered normal textile raw materials, having naturally and potentially stable, favourable textile characteristics. They only have to be properly extracted and produced in a pure intrinsic form.

Initially, the produced flax fibres had inconsistent textile characteristics depending on the different circumstances of their cultivation and the skill of the individuals who performed producing them. Often the nature of the plant itself and the prevailing weather conditions were blamed for the unstable specifications of the fibres.

With the advance of science, the first scientific explanation was given for the phenomenon of flax fibre separation when the straw was immersed in water for some time, resulting in the widely applied European flax extraction technology. According to this technology, the bacteria formed on the immersed flax straw develop and multiply and for their nutrition they secrete a specific enzyme that decomposes the pectin that ties the fibres together and to the other plant tissues. Once the pectin breaks down, the fibres are gradually liberated, starting from the root-ends and up to the crop-ends of the straw.

Information about common varieties of agricultural plant species, including flax and linseed, is given in Chapter 13, vol. 1: Developments in fibrous flax breeding and cultivation.

5.2 Flax plant morphology

The vitally important nutritive system of flax is composed of the following.

5.2.1 The root and rootlets (root-hairs)

The root of the plant is composed of an absorbing tissue, allowing the storage of all the water (soil solution) collected by the fine rootlets projecting in all directions and at different levels. This structure also has the function of fixing the plant firmly into the soil. The collected water is channelled upwards through the wooden cylinder to each leaf of the plant for its transformation into nutritive sap. The actual capacity of the rootlets is a limiting factor to the development of all other plant tissues and thus determines the final amount of any plant crop.

To be most efficient, each root needs to be surrounded by an even, specific free space, to allow the full expansion of its root-hairs without the interference of other rootlets belonging to other too closely positioned plants. Otherwise, the amount of available water will be shared between all closely positioned plants, which will only grow according to the percentage of the water each plant receives.

This is the reason why the maximization of the seed crop of closely positioned plants has been practically impossible. The vital free space needed to surround each plant has a maximum, which is equal to the furthest distance that the root-hairs can reach and an essential minimum without which the plant cannot grow normally.

5.2.2 The wooden cylinder

The central wooden cylinder is composed of a porous wooden hard tissue acting as an upright standing structure for the plant. It also acts as a duct to

bring up the soil solutions collected by the rootlets and stored in the root, to get them to the upper-side of each leaf to be transformed into the nutritive sap of the plant.

5.2.3 Flax fibres

Inside the plant, flax fibres are found as individually separated filaments of different lengths, which vary according to the height of the leaf they serve on the stem of the plant. Each filament is composed of a number of single tapered-ended fibre cells evenly joined lengthwise, so that each cell underlays 50% of the preceding one and overlaps 50% of the following one, tightly tied together all along, to form single filaments of the same regular fine thickness, but of different lengths.

A set of ten single filaments of the same length serves each plant leaf, running down from the height of the leaf to the surface of the soil. In each case, the ten filaments have lengths equal to the height of the served leaf on the plant.

The ten filaments do not stick together but are grouped together in bundles, circularly distributed around a supporting central wooden cylinder, and completely surrounded by a holding tissue, which fills the whole inner space between the wooden cylinder and the outer skin. These characteristics are responsible for the fine textile characteristics most favourable for the successful production of regular fine yarn, without major spinning problems.

In the plant, flax fibres are naturally and completely formed during the vegetative growth, as an essential part of the nutritive system. One of their functions is to receive and transport the already formed nutritive sap from the lower side of each leaf downwards to the holding tissue cells, which store it and use it to fulfil the needs of all the other plant tissues during their development.

5.2.4 The holding tissue

The holding tissue is completely formed of dynamic cells that have semi-permeable membranes to facilitate the easy exchange of nutritive sap; all the other plant tissues are formed of static cells that do not have semi-permeable membranes. The holding tissue is essential to all future developments in the plant tissues or their replacement in case of damage. It also tightly surrounds the different bundles of fibres transporting the already prepared nutritive sap from each leaf to easily absorb and store all the upcoming sap.

The stem of fibrous flax has a maximum length in green maturity stage. According to the results of many research works (Department of

Agriculture, Northern Ireland, 1985), the yield of flax straw and fibres is the highest in yellow maturity stage, but the highest quality fibre is obtained in green/yellow maturity stage of flax straw, when the fibre is fine and the level of lignin is lower than in yellow maturity stage.

Flax fibres are an important active part of the plant nutritive system and thus contribute to natural potentially excellent textile characteristics (quality), from the beginning of the vegetal growth phase, increasing in quantity with the growing plant's further development, until the end of the vegetal growth phase.

During any stage of growth, flax fibres have definite stable natural potential characteristics in relation to their thickness, tensile strength and suppleness. These characteristics are only apparent when the fibres are properly and smoothly extracted and obtained in a single homogeneous filament form, although the various filaments differ in length, because in each case they are equal to the height of the leaf they serve. It is worth adding that the ten single filaments in each of the ten-bundle filaments are of equal length.

In the plant, flax fibres are individually separated but grouped in bundles completely and firmly surrounded by the holding tissue. They have smooth glossy surfaces, which allow them to freely slide on each other when the stem bends in all directions.

The number of groups of flax filaments inside each stem is equal to the number of leaves appearing on this stem. Therefore the number of single filaments equals the number of leaves multiplied by ten.

The single flax filament diameter (thinness) has been mathematically calculated and found to be 0.250 tex.

The development of the cellulosic fibres from flax occurs progressively during growth through two main steps:

1. The elongation of the mother fibre cells (from several mm to cm depending on its position in the stem) results in a cell wall of high plasticity, due to a high level of neutral hydrosoluble polysaccharides, mainly β-1,4-galactan.
2. The progressive maturation of the fibres leads, on the one hand, to synthesis of cellulose and, on the other hand, to the reinforcement of the linkages in the cellular junction and middle lamellae with specific components such as the acetylated polygalacturonic acids or rhamnoglacturonan I-like polysaccharides, resulting in a supraorganization of the fibres into bundles.

At the maturity stage, the cell wall in the cortex is enriched in polygalacturonic acids of various degree of methylation depending on their localization in the primary cell walls or in the middle lamellae.

Such polysaccharides are easily degraded by pectinolytic enzymes (see Chapter 5, vol. 2: Enzymatic treatment of natural fibres). Conversely, the cell walls of the fibres are enriched in β-1,4 galactan, rhamnogalacturonan I

associated to acetylated polygalacturonic acids and rhamnogalacturonan II-like polymers.

The high heterogeneity of flax production is well known. There are lots of heterogeneities: in length, diameter, cellulose content in the fibre and polysaccharide cements of both tissue cortex and fibre differences in pectin composition according to the tissue cortex and fibres (Morvan, 1991).

In Russia, 13 pure lines of flax genetic collection were evaluated at N. I. Vavilov Institute in St Petersburg, for their vegetative period, yield, quality of fibre, as well as structure and composition of fibre. Correlation analyses showed that the strength of technical fibres was correlated to plant height, straw yield and number of bundles per stem section with diameter of elementary fibres. The fineness and flexibility were linked with the ratio between the areas of the fibre and xylem tissues and with the relative amount of encrusting components to cellulose in fibres (Brutch *et al.*, 2008).

5.3 Structure and chemical composition of flax

The group of fibrous plants including flax, hemp, jute, ramie, sisal and kenaf are the source of bast fibres. The main component of the fibres is cellulose, while secondary components are waxes, fats, hemicellulose, lignin and pectin (Konczewicz e*t al.*, 2006a, 2006b, 2007). The chemical composition of the fibres is presented in Table 5.1.

Table 5.1 Chemical composition (%) of lignocellulosic fibres

Fibre	Cellulose	Lignin	Pectins and hemicelluloses
Abaca	60–80	6–14	13
Bamboo	26–43	21–31	–
Cabuya	80	17	–
Coir	36–43	41–45	3–4
Cotton	83–99	6	5
Curaua	70–80	13	–
Flax	64–84	0.6–5	19
Hemp	67–78	3.5–5.5	17
Henequen	60–78	8–13	4–28
Isora	75	23	–
Jute	51–78	10–15	37
Kenaf	44–57	15–19	–
Nettle	53–82	0.5	0.9–4.8
Pineapple	80	13	–
Pita	80	17	–
Ramie	67–99	0.5–1	22
Sisal	60–80	6–14	13

Source: Kozłowski *et al.*, 2010.

Pectin plays an important role in the fibre as a component that binds the fibre into bundles and also determines the lustre and touch of the fibre. Pectins are macromolecular compounds of polygalacturonic acid. They are agglomerated in the middle blades mainly in the ground tissue. In fibrous plants, two pectin fractions can be found, i.e. fraction A which is soluble in water and fraction B which is water-insoluble. Technical flax fibre consists of cells bonded with each other by a lamella, which in turn consists of mostly pectin B with a small amount of pectin A. Fibre bundles, located in the phloem, form rings around the stem, of more or less compact structure, bundles of which are attached to adjacent tissues with a layer of pectin A.

Proper removal of pectin substances during preliminary processing determines the divisibility and, as a result, the fineness of the fibre and its suitability for spinning (Kozłowski *et al.*, 2010).

The easiest to remove in the retting process is pectin A, which is decomposed by bacteria and fungi, while pectin B remains within the fibre and is decisive for the compactness of the fibre. Excessive removal of pectin and natural waxes will cause the fibre to have an unpleasant dry and coarse touch. Complete removal of pectin will result in disintegration of fibre bundles into elementary fibres.

Waxes and fats are also important in terms of technological parameters. They determine soft touch, low friction and thus ease of moving the fibre. In flax fibre, waxes are present mainly in the outer part of the stem, in the epidermis, and in smaller amounts in fibre cells.

Lignin functions as an inlaying component within amorphous areas of cellulose and causes the cellulose to be rigid. In the elementary fibre, lignin occurs in the primary wall and outer part of the secondary wall. For processing the presence of lignin is undesirable as touch and flexibility of the fibre become worse. The presence of lignin means that the fibre is more easily breakable and mechanical parameters of tenacity and resiliency deteriorate. Moreover, the divisibility of the fibre lowers as a result of the presence of lignin.

The structure of elementary fibre and its microscopic, mechanical, physical and chemical properties also have effect on the overall quality of the fibre.

Single fibres that constitute plant fibrous tissue are referred to as elementary fibres, either when they are a part of a compact bundle or are loosely located within the cortex tissue. The structure of elementary fibres is similar in all lignocellulosic fibres.

The quality of the extracted fibre depends on the extraction method and the amount of elementary fibres, their distribution and binding within the bundles and between the bundles. In order to separate the fibres from other tissues, various mechanical treatments are used – decortication, hackling,

scutching, etc. Better results are achieved by preliminary processing such as water or dew retting, as well as enzymatic, chemical or physical processing of the fibre.

The average chemical compositions of flax stem and fibre are as follows:

Stem:

Cellulose 49–60%
Hemicellulose 10–25%
Lignin 17–23%, pectin 3–4%
Fats and waxes 1.5–3%

Fibre:

Cellulose 85–87%
Hemicellulose 7– 9%
Lignin 2.5–4%, pectin 1.5–2.5%

The differences depend on the kind of applied varieties, conditions of cultivation, date of harvesting, system of primary processing (decortication, dew retting, water retting, osmotic degumming), etc.

The structure and properties of flax in comparison to other bast fibres are presented in Tables 5.2 and 5.3.

Comparative study of the infrared (IR) spectrum has shown the presence of varying quantities of pectin, hemicelluloses and lignin in the fibres originates from various methods of flax straw degumming. The obtained data have revealed fluctuation of valence bands characteristic to flax and have shown the reliability of the Fourier Transform Infrared Analysis (FTIR) method for estimation of the degree of flax fibre fineness (Titok *et al.*, 2010).

Table 5.2 Morphological characteristics of bast fibres

	Single bast fibre (cell)				Cellulose microfibril	
Fibre type	Shape of cross-section	Length (mm)	Diameter (μm)	Wall thickness (μm)	Fibrillar bundle width (nm)	Microfibril width (nm)
Hemp	Flat/polygonal	10–30	5–50	5–10	25–80	3–18
Flax	Round-oval/ polygonal	20–40	10–20	5–10	30–80	3–18
Jute	Round-oval/ polygonal	1–6	10–25	2–5	40–90	3–12
Ramie	Flat	50–250	10–60	3–5	40–100	3–12
Sisal	Round-oval/ polygonal	1–5	10–40	3–5	20–50	3–8

Source: Pinnov and Fink, 1999.

Table 5.3 Degree of crystallinity and crystallite dimensions of native cellulosic fibres

Fibre	Content of cellulose (%)	Degree of crystallinity (%)		Crystallite dimensions (nm)	
		Fibre	Cellulose	Lateral	Longitudinal
Flax	79	44	56	4–5	12–24
Hemp	75	44	59	3–5	12.5–29
Jute	61	33	54	3–5	12–21
Cotton linters (scoured and bleached)	100	56–63	56–63	5–6	10–20

Modern tools for testing morphology and supermolecular structure of flax are:

- light microscopy,
- scanning and transmission electron microscopy (SEM and TEM),
- x-ray diffraction,
- nuclear magnetic resonance (NMR) spectroscopy.

More data on methods of testing and identifying natural fibres are given in Chapter 11, vol. 1: Identification of natural textile fibres.

5.4 Flax harvesting

During harvesting, flax is pulled, not cut. In 1960, the first self-propelled flax pulling machine was developed by Depoortere in Belgium (Fig. 5.1). The one raw machine has four pulling elements of 35 cm each, which means a total pulling width of 1.40 m. The two raw machines are provided with a double pulling machine with total pulling width of 2.40 m. When used in this process, self-propelled pulling machines (e.g., those made by Depoortere, Belgium) achieve polling yield from 0.5 to 1.25 ha/h. Then, the flax straw next is laid in the field in rows and the dew-retting process is conducted.

After pulling is over, the turning operation is carried out, which can be done in different dew retting times. Most of the machinery used for turning is also self-propelled. In the case of dew retting, 10 days after pulling deseeding has to be performed. Generally, two types of deseeding machines are used: a deseeding machine can be mounted on the chassis of the self-propelled flax pulling machine or a machine designed specifically for deseeding may be used. The deseeding has to be carried out when seed capsules are dry: 25% of moisture is allowed, but then the capsules have to be stored under cold ventilation to dry the moisture content to 12–15% (Depoortere, 1989).

5.1 Photographs of pulling, deseeding and turning machines used by Belgian company Depoortere. (a) Pulling machine, double row with cabin, (b) deseeding machine, double row 1, (c) turning machine, double row 2.

(Continued)

Flax harvesting from the field (unretted and dew retted) is performed by flax round balers, which are of two types: drawn round baler and self-propelled round baler. The drawn machine is cheap but its work is not so precise as that of the self-propelled round baler, which bales flax perfectly. During round baling,

5.1 Continued

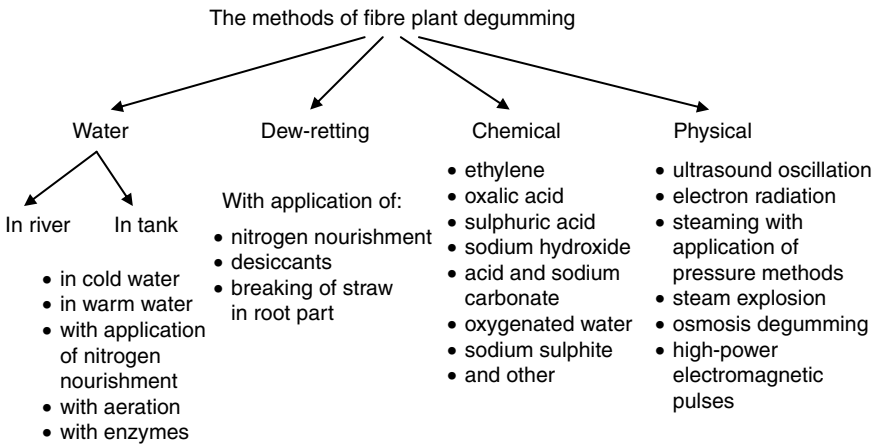
two ropes are rolled around a bale and their consumption ranges between 25 and 30 k/ha. The ropes have to be made from coarse flax, hemp or sisal fibres, never from polypropylene. Straw intended for water retting and for decortication is collected by a round baling system, whereas straw intended for dew retting is laid in rows in the field for 3–6 weeks and after that time the retted straw with humidity below 16% is rolled and delivered to a scutching mill. Currently, as already mentioned, in most European countries dew retting is widely used but crop loss, associated with this process, occurs around once every 5 years. Water retting, which consumes a lot of water (see above) and results in the formation of a very large amount of sludge (about 30 m^3 of sludge/ton of retted straw), is not applied nowadays (Dambroth and Seehuber, 1988).

5.5 Degumming

In order to separate fibre from the woody core, several methods for fibrous plant degumming are used. In this process, fibres are separated from adhesive substances like hemicelluloses, pectin, and partially from lignin, waxes and fats. The methods of fibre plant degumming are presented in Fig. 5.2. These methods of separation enable fibre to be obtained with the required length, fineness, purity, strength, optimal efficiency and homogeneity.

5.5.1 Retting of flax: water and dew retting

The biochemical process of retting can be performed under aerobic and anaerobic conditions.



5.2 The methods of fibre plant degumming.

Water retting

In the process of water retting, depending on temperature, environmental conditions (the presence or absence of oxygen), and sometimes nutrients, different bacteria take part, namely *Clostridium* sp., *Clostridium butiricum*, *Granulobacter pectinovorum*, *Clostridium felsineum*, *Clostridium guerfelli* and *Bacillus amylobacter*. In the case of water retting under anaerobic conditions, pectin materials and hemicelluloses which bond the fibres, decompose and form volatile fatty acids, mainly butyric (80%), responsible for the odour of retting liquor and artificially dried fibres. When sun-drying is performed, the volatile fatty acids undergo decarboxylation and decompose, thus after such drying, water-retted straw, as well as fibre dried in the air, does not have any odour (Jankauskiene *et al.*, 2006; Kozłowski, 1969, 1992; Schilling *et al.*, 1951.

Once this fact became common knowledge, all who performed the retting of flax straws had to stimulate the multiplication of the bacteria by heating the treatment water to 30°C or by adding active bacteria cultures to the water, or even by adding pure enzymes to hasten the decomposition; but in all these attempts the inconsistent specifications of the produced fibres persisted.

Recently, this interpretation of the reason for the separation of flax fibres was found to be scientifically incorrect. Thanks to a newly discovered, more controllable method of fibre extraction, it was discovered that within the flax plant, flax fibres have naturally stable excellent textile characteristics; they exist in the form of single filaments grouped in bundles surrounded by a holding tissue. They are not glued together and to the other plant tissues,

they are only grouped and held firmly by the holding tissue, consisting of dynamic cells having vulnerable semi-permeable membranes.

By smooth and complete elimination of the holding tissue, the flax filaments are gradually liberated in the form of individually separated filaments without affecting their natural potential textile characteristics, which were recently found to be suitable for spinning the finest yarns without any major problems, in addition to being consistent for every variety of flax.

The retting process may be carried out either without or with addition of urea in relation to the mechanical process, hackling, spinning, yarn bleaching, weaving, dyeing and fabric printing. Research carried out on a commercial scale included the comparison of retting process without and with addition of 0.5% urea in relation to the mechanical process, hackling, spinning, yarn bleaching, weaving, dyeing and fabric printing. As a result of urea application, in addition to the reduction of retting period (in this case above 34%), it appears that the most advantageous technological factors have been obtained at the optimum retting period of 52 h with the addition of urea and 79 h for the conventional retting process. Both hackled fibre, as well as grey and bleached yarns produced from these optimum retting periods had the best parameters, but the parameters from 52 h retting with addition of urea are more advantageous than those obtained from the conventional retting.

The most advantageous factors for the finished fabrics have also been obtained from the optimum retting periods. The tensile strength of the fabrics produced from the longer retting periods is lower than that from the optimum retting periods both with and without addition of urea. The fabrics produced from fibre retted by the conventional process experienced during different finishing processes higher losses than those manufactured from fibre retted with addition of urea (Probulski *et al.*, 1967).

Dew retting

During dew retting various fungi take part in this process, such as *Alternaria alternata*, *Alternaria linicola*, *Cladosporium herbarum*, *Fusarium nivale*, *Cephalosporium* sp. and other, mainly pectinolytic and cellulolytic species. Flax straw lies on the field for about 2 months. Dew, sometimes rain, starts to develop the fungus activity mentioned above.

After dew retting two factors have a great influence on fibre quality:

- the level of moisture for a good conservation, without risk of over-retting in bales, which degrades the fibre strength;
- the alignment of swath to avoid picking up the neighbouring swaths and crushing the extremities of the stems in the baler.

The harvesting operations in field retting can preserve quality in several respects:

- homogeneity of dew retting and fibre alignment influence processing yield;
- degree of retting has an effect on divisibility of fibre and strength of the yarn;
- cleanness of the straws helps with avoiding irregularities after the dyeing of yarn or fabric.

There are two problems in the case of traditional use of fibre after water retting:

- pulling binding, where the problem consists in obtaining a good tossing in the shives for colour and the homogeneity of retting;
- pulling-spreading (mostly used for green flax harvesting) is associated with the same problem as that encountered in the case of field retting, since swaths regular in thickness and alignment will give straw homogeneous in colour and make it easy to pick up it for baling (Kozłowski, 1992).

Osmotic degumming

The discovery of a more controllable and non-polluting method of vegetal fibre extraction, using regular water and taking advantage of the natural forces of fluid diffusion – osmosis and the effective force of the resulting osmotic pressure generated inside the cells that have semi-permeable membranes – made it easy to gradually and smoothly extract the flax fibres without affecting their natural potential for excellent textile characteristics.

In Egypt, with the involvement of the Institute of Natural Fibres, Poznan, Poland, and the FAO/ESCORENA European Cooperative Research Network on Flax and other Bast Plants, a new, more controllable method of vegetal fibre extraction has been investigated, using osmotic pressure which enabled the avoidance of the pollution caused by bacteria and fungi. This method is capable of resulting in more homogeneous pure flax fibres in single filament form which, once implemented, would fully satisfy the world demand for fine flax fibres.

Osmotic degumming consists in the extraction of fibres based on physical laws: water diffusion, osmosis and osmotic pressure. Using physical laws, especially those concerning osmosis, when the inner part of a fibrous plant is in contact with water, the extraction of fibres can occur with no effect on their natural properties. The fibre obtained by this method is delicate, thin

and has colour adequate to the quality of raw material used (Konczewicz and Kozłowski, 2007; Konczewicz *et al.,* 2006a, 2006b).

Osmotic degumming enables homogeneous fibre to be obtained and allows for objective evaluation of the amount and quality of fibre contained in the raw material. This is especially important when evaluating progress in breeding new cultivars of fibrous plants. Osmotic degumming can be accelerated using ultrasound processing. From the point of view of industrial application of osmotic degumming method to bast fibrous plants, it is worth mentioning that fibres obtained by using the above method can be employed for producing better textiles (yarn, fabric or nonwoven) and composite products.

Osmotic degumming gives homogeneous long flax fibres with fineness of – 0.91 (tex) and breaking tenacity of – 62.79 (cN/tex) (Kozłowski *et al*., 2010a). This more controllable method of vegetal fibre extraction takes advantage of natural physical laws of fluid diffusion, osmosis and osmotic pressure, to consistently produce flax fibres of stable and more homogeneous textile characteristics, capable of successfully competing against all other textile raw materials. It is reliable, as it is based on the following scientific principles:

1. The tissue surrounding the fibres inside the plant is completely formed of dynamic cells, which have semi-permeable membranes, while all other parts of the plant (the wooden cylinder, the fibres and the outer skin) are formed of static cells, which do not have semi-permeable membranes.
2. According to the natural physical laws of fluid diffusion, osmosis and osmotic pressure: when two fluid media of different water molecules concentrations are separated by a semi-permeable membrane, the water molecules in the medium of higher water molecules concentration will continuously move to the medium of lower concentration by crossing the semi-permeable membrane, which only allows a one-way crossing, until the concentrations in the two media become equal.
3. When flax straw is submerged into fresh virgin water, each dynamic cell in the plant represents a medium of lower water molecules concentration, separated by semi-permeable membrane from the surrounding fresh water, which represents the medium of higher water molecules concentration.
4. Under these conditions the water molecules in the surrounding treatment water continuously move and enter each dynamic cell, through its semi-permeable membrane.
5. As a result, increasing outwardly osmotic pressure is generated on the inside of the semi-permeable membranes of each dynamic cell, causing the gradual rupture of its semi-permeable membrane when the osmotic pressure becomes more than these membranes can stand and

the contents of the dynamic cells are then dispersed into the surrounding water.

6. This gradual destruction of the dynamic cells, forming the holding tissue firmly surrounding the fibres, continues until the complete release of the fibres, as long as the difference in water molecules concentration, inside and outside these dynamic cells, is kept at its maximum, by renewing the treatment water every time it becomes ineffective when saturated or polluted.
7. To follow up the course of the fibre separation, samples of the treated straw are frequently tested by trying to pull out the fibres sideways from a piece of straw from the root-end to discover the degree of fibre separation.
8. Water molecules enter the flax straw through the central wooden cylinder and attack the dynamic cells layer after layer, starting with the first circular layer adjacent to this wooden cylinder up to the last circular layer adjacent to the outer skin.
9. During the extraction operation, all static cells forming the wooden central cylinder, the fibres and the outer skin are only wetted to saturation without being affected.
10. Once all the dynamic cells, forming the entire holding tissue, are eliminated, the fibres are totally liberated and appear individually separated; they are also be in a pure form and clearly have natural potential for superior high textile qualities.

Enzymatic degumming

Enzymatic treatment allows for faster and simultaneous degumming of fibre from the woody part and decomposition of pectin in the fibre itself. This leads to shortening of the degumming time and improvement of fibre quality, mainly by higher divisibility and fineness of fibre strands (Kozłowski *et al*., 2005, 2006a, 2006b).

Enzymatic treatment (mostly with use of pectinolytic enzymes) enables the application of preparations that undergo complete biological degradation in sewage which leads to an eco-friendly technology as well as water and energy savings (Foulk *et al*., 2009; Guebitz *et al.*, 2006; Jayapriya *et al*., 2010; Kozłowski *et al.*, 2009).

The commercial offer to improve the retting process by using enzymes has been put forward by Novo Industry, Rohm and Haas, Enzylin, Bioprogress Maceraze, Pectopol U and other companies.

Flaxzyme offered by Novo Industries for degumming of flax straw is used at a concentration 3–6 mL per 1 L of water at temperature of 40°C. The enzyme solution can be recuperated many times. Flaxzyme consists of polygalacturonases, methylesterases, pectin lyases and, in smaller quantities,

chemicellulase and cellulase. Enzymatic treatment can also be applied to green decorticated fibre. In the case of decorticated flax fibre the following parameters were obtained: fineness 3.6–4.7 tex, strength 6.8–8.8 cN/tex and good softness and feel (Kozłowska, 1989). For more about enzymatic treatment and about the mechanism of enzymatic action is presented in Chapter 5, vol. 2: Enzymatic treatment of natural fibres.

New process of plant fibres degumming using electric resonance technology

A novel plant fibre degumming technology has been developed that consists in the use of electric resonance electrodes immersed into a water pool, possibly with the addition of specific pectolytic enzymes to the water, to accelerate the process.

The special electrodes used for degumming fibre crops in a pool, develop an electric field, without producing electric current, but propagating a particular resonance signal in the water.

The retting process occurs by combining two factors: (a) the utilization of a device called EL3 that generates signals at frequencies ranging from 50 Hz to 12.8 MHz, (b) the addition of a special mixture of selected enzymes.

The well-dried biomass bale should be fully submerged in the pool for a period of a few days. A continuous retting system is easily conducted; the electric resonance produced by the electrodes eliminates bacterial development.

The advantages of the technology are:

- in the case of flax, the seeds can be harvested separately, before baling of the stems;
- the possibility of processing also well-dried biomass;
- the process produces neither bad odour nor contaminated water, in contradistinction to the traditional pond retting system, which at present is not accepted in most countries;
- the colour of fibres is not dark (as often occurs in the case of the dew retting process or full immersion retting); fibres are clear and brilliant, and they are characterized by higher quality;
- cutting and baling in the field can be performed when the crop is fully matured and dried, which enables decreasing the size of the retting processing plant and reducing manpower.

In order to make the separation of fibres easier and faster, specific pectinolytic enzymes, dissolved in an appropriate amount of water, are used.

The optimal temperature for degumming is between 25°C and 30°C. Lower temperatures will slow down the time of processing. In optimal

conditions of treatment, an increase of water temperature is observed due to molecular movements induced by the resonance. In water volume of 5.5 cubic metres and with the power system used, two pairs of immersed electrodes are needed. Electrodes should be located under the water level, one in front of the other, but they should not be in contact with the walls and with the bottom of the container, nor with the bale of biomass. Each electrode used has the size of 55 cm × 15 cm × 5 cm.

After processes involving the electrodes and enzymes are over, it is necessary to completely dry the biomass in order to preserve and, later on, to process the material for the extraction of fibres. This drying process could be energy consuming, if not properly done. Possibly, energy from solar panels and dry air forcing technologies should be used after the opening of wet bales to speed up the process and save electric and thermal energy.

If the stems could be properly cut and orderly baled by machine at harvest, all the process could be made easier and the final product quality could be better. Note: special electrodes were developed by Italian physicist Professor Mario Pandolfo; see the technical indications provided by Giovanni Mignoni, Pietro Cappelletto and other experts (Bozzini and Mignoni, 2010).

5.6 Scutching

Scutching is the next operation after harvesting and retting, which is aimed at separating bast from woody core. For this operation retted straw is prepared as parallel layer stalks. Next the flax layer is gripped between two belts – U Belt and T Belt. During the transit, the flax tops are broken by 2 × 5 pairs of jagged cylinders: the first 5 pairs with rough teeth, the second five pairs with fine teeth. After this, broken part of the stalks is taken over by two other belts, which continue the transit. The scutching removes broken stalks and remaining shives and also short fibres obtained.

The typical scutching machines consist of two drums 4 m long for scutching the foot of the straw and two drums 4 m long for scutching the top.

The shives and short fibre dropping under the drums are pneumatically sucked up and put on the shaker which removes the loose shives.

At the end of the shaker the short fibre is transported to an automatic tow press or tow scutching machine. The scutched long flax fibre goes out of the end of these scutching machines and is laid down in handfuls, which are tied into bundles (16–20 kg). These bundles are pressed into 100 kg bales and sent to the spinning mill.

The scutched flax is in the form of coarse fibre strands (bundles of many fibres joined together by hemicelluloses and residual pectin and also some woody impurities). The fibre strands consist of 15 fibre bundles held together in lattice formation and they are roughly parallel. The quality of the scutched

fibre generally is estimated by organoleptic method (features such as lustre, colour, handle, alignment of the strand, etc.) as well as by objective methods such as measuring the strength, fineness, length of fibres, level of impurities and also freedom from naps and knots.

5.6.1 Decortication of green straw

There are so many disadvantages of water retting and dew retting, which were mentioned before, that many research centres have attempted to develop direct extraction of the fibre from the straw by breaking or tearing off raw straw by the process called decortication. Obviously the quality of such fibres (bast) is very low and such fibres are not useful for textile application without enzymatic or chemical treatment. Decortication is cheaper and faster in comparison to different degumming methods. One of the new concepts is the treatment of the decorticated bast by osmosis and enzymes and intensifying these treatments by exposure to ultrasound. A photograph of the decorticator developed at the Institute of Natural Fibres (Polish Patent 199630, Kozłowski *et al*., 2008; Kubacki, 2010) is shown in Fig. 5.3. This system is very promising from an economic viewpoint.

5.3 Decorticator, LENKON: photograph of the decorticator developed at the Institute of Natural Fibres, Poznan, Poland (Polish Patent 199630, Kozłowski *et al*., 2008; Kubacki, 2010).

Summing up, there are some important rules for the production of high-quality fibrous flax, followed by degumming and extracting high-quality flax fibres, suitable for spinning.

- The choice of a proper soil with pH 6.5–8.0. Flax should not be grown on the same plot of land more frequently than once in 4 years and the preferable interval is 7 years. Sowing should be carried out as soon as favourable conditions occur. In Central Europe this can be done in the second week of March and preferably before the first week of April. Later sowing is not recommended, because the yield and quality are lower. Young plants survive to –3°C and for a short time even at –6°C.
- The use a suitable compound fertilizer to provide approximately 20–22 kg N, 20–25 P_2O_5 and 80–90 kg of K_2O per 1 ha. It is also recommended to apply 4–6 kg of $ZnSO_4$ dissolved in 400 L of water per 1 ha, as well as 0.3–1 kg of borax.
- The choice of a suitable variety.
- Establishing the optimum sowing density (about 1800–2000 plants/m^2; 125–140 kg seeds/ha.
- Good crop of flax free from weeds.
- Freedom from lodging.
- Pulling at the stage of green (green/yellow) straw (straw yield between 5 and 7 tons/ha).
- In the case of dew retting – turning the straw in the field at least twice.
- Collecting the retted straw and storing at a uniform humidity below 16%.

In the case of good dew retting, scutching gives a yield of 15–20% of long fibre and tow 6–3% (Sultana, 1991).

5.7 Hackling (combing)

Hackling of scutched flax fibres is a very important process in the preparation of fibres to effective quality for spinning. In the linen industry three types of hackling (combing) machines are used: very old Russian system, Mackie system (Northern Ireland) and Linificio system (Italy). Lummus Mackie developed a hackling machine that was mechanically simpler and more efficient than the old Russian system. The patented system opens the combing sheets, moves the flax pieces into position and then closes for the hackling process to commence. The sheets then are opened again allowing the flax to be carried to the next stage. This system achieves a consistency in the rate of hackling which is ensured by the inclusion of an inventor control motor to regulate the rate of hackling.

The corner motion is by a pneumatic mechanism, which ensures a quick and efficient method of transferring the flax pieces from one side of the frame to the other, while at the same time repositioning the flax piece to ensure a complete hackle. The holder facilitates the hackling of a larger amount of flax per lift.

Dust and dirt extraction is an important part of the hackling process. The above system comprises a doubler, four drawing frames and a roving frame.

The commonly used system of processing (hackling) of the scutched fibres (e.g., Linmack machine of Mackie International) is presented in Table 5.4.

The hackling machine consists of two sides or 'machines' to treat the top end of the fibre strand and the root-end. The fibre bundles held in holders travel along the 'root side' of the machine, where the shorter root-ends of the fibres are combed or hackled between the pinned sheets of the machine. At the other end of the machine, the fibre is turned around so that when travelling along the 'top side' of the machine the tops of the fibres are combed, care being taken to see that there is no part of the fibre left untreated – this is achieved by an automatic pull-through at the change-over end.

The hackling machine therefore produces two types of fibres:

1. Long straight fibre bundles – 'line fibre',
2. Short tangled fibre – 'tow fibre'.

The yield of line fibre is defined as:

$$\text{Yield of line fibre} = \frac{\text{weight of line fibre produced}}{\text{weight of scutched fibre fed}} \times 100 \qquad [5.1]$$

The yield of hackled fibre is usually in the range 55–65% and depends on the degree of hackling, on fibre quality and purity. The production yield (capacity) of the hackling machine is normally between 40 and 65 kg/h (Ross, 1989).

Table 5.4 System of hackling of the scutched flax fibres

Flax line	Flax tow (hackled noils)
Preparing	*Carding*
Doubler	
1st Drawing passage	
2nd Drawing passage	*Preparing*
3rd Drawing passage	
Roving	*Finisher*
Line roving machine	
Wet ring spinning	*Dry ring spinning* *Wet ring spinning*

Source: Mackie International, 1994.

5.8 'Cottonization'

'Cottonization' is a process by which the flax fibre is made shorter and finer with the lowest possible level of impurities. There are several methods to make flax fibre 'cottonized'.

- Generally the flax fibres used in wool and cotton spinning come from classical scutching. Next they are proceeded to adjust their length and purity according to spinning system which also provides with the possibility of blending with other fibres (Sultana, 1991).

5.8.1 'Cotton' type flax fibre

'Cotton' type flax fibre has the following properties: staple length – unbleached 30–35 mm, bleached 20–30 mm, linear density unbleached 1.5–2.9 tex, bleached 1.0–2.0 tex, impurities below 0.5% (Kozłowski, 1996).

'Cottonized' flax can be processed in the same way as cotton (US Patent 666696 – a new mechanical treatment process for flax). The decortications can be used to separate the woody component, pectin and other impurities, which can be removed by chemical or cooking process of a fibre bundle that is then converted into a shorter staple fibre. Fibre bundle with a 10% of moisture content is carded and a fine fibre strip or sliver is created. The flax tow is carded on Mackie card and the sliver has only 5% of woody impurities. The sliver is then drawn 5 m/min with 16 doubling. Next it is padded and fed into an intermediate steamer followed by further wet processing, but finally drum dried and delivered into a card can coiler.

At the first stage impregnation and subsequent steaming takes place at 60°C and pressure of 2 psi (13.79 kPa), after which washing is carried out in a solution of 35 mL/L NaOH, 50° BE (6 g/L cottoclarin OI surfactant, 3 g/L securon 540 complexing agent × 2 g/L Defindol EX deaerator). Liquor pick up is 72% and the sliver is then deposited in a steamer for 30 min and treated with saturated steam at 100% followed by drying at 140–150°C. This system is then used to bleach the sliver with intermediate steaming at 20°C and again 2 psi (13.8 kPa). The bleaching liquor composition is 0.15 g/L magnesium sulphate, 8 mL/L water glass 38°Be 5 g/L sodium hydroxide sol, 50°Be 6 g/L cottoclarin OK, 2 g/L securon 540, 2 g/L Belsoft 200, plasticizer, 60 g/L H_2O_2 –35% (Lennox, 1998).

5.8.2 'Wool' type flax fibre

The thinness of 'wool' type flax fibre is a maximum of 30 nm (for the yarn 23 tex, length of the staple 40–120 mm, impurities below 1%, homogeneous color).

'Wool' type flax fibre is used for ring, wrap, friction–DREF 2 spinning, mainly for the production of blended linen yarns, also for fabrics, knitting, apparels, socks, shoes, household goods and upholstery fabrics.

5.9 Spinning

Flax fibres after extraction and hackling can be used for spinning. Both short and long fibres can be used, but different techniques are used for spinning short (tow and noils) and long fibres.

5.9.1 Problems with spinning linen

The established belief has been that flax fibres naturally have heterogeneous textile characteristics, making their spinning a complicated and difficult job, successfully achieved only by highly skilled spinners. For this reason more than 75% of flax yarns usually produced worldwide have been of easier-to-spin, coarser counts whose production has stopped lately due to lack of demand. The more astute spinners who have been producing different specialized fine counts of flax yarns still exist, but are loudly complaining about the scarcity and irregularity of the flax raw materials available.

The latest thorough scientific review of the plant's detailed morphology and fibre extraction operation, sustained by practical experience in flax cultivation, flax straw processing and flax fibre spinning, have successfully led to the firm confirmation of the following:

1. The stability of flax fibres has favourable natural potential textile characteristics.
2. It is possible to maximize the production of either the fibres or the seeds by using two distinctive methods of flax cultivation. The maximization of the fibre crop is successfully obtained through optimizing the number of slender branching single stemmed plants, by giving each sowing seed only the minimum necessary vital free space around it aimed at preventing the plants from giving birth to tillers or many branches and sub-branches. The maximization of the seeds is successfully obtained through minimizing the number of plants and encouraging them to give birth to the greatest number of tillers, branches and sub-branches, to bear the greatest number of capsules containing seeds, by giving the plants the maximum necessary vital space around each plant.

Flax yarns have an irregular thickness that is often welcomed by weavers as an attractive and idiosyncratic feature. Their users have considered the characteristic irregularities in the textures of the linen fabrics produced as a welcome change to the monotonous regularity of other more uniform fabrics. These linen irregularities have even been industrially

imitated with other homogeneous textile raw materials to get the same irregular effect.

5.9.2 Developments in linen spinning

Fineness and regularity in length are two important factors for flax spinning. The good ripeness of the straw brings a lower heterogeneity between the fibrils of the top and bottom (Sultana, 1991).

The first flax spinning machines could produce only coarse yarns and could not compete with handmade 'workshop linen yarns'. The first step in splitting flax fibre bundles, not only in the hackling process, but also in further stages of spinning, was done in France. In particular, progress was made by chemist Gay-Lussac at the Engineering College in Paris, who had adopted the chemical treatment to flax spinning. Further development in changing the length and thickness of flax fibres resulted in mechanical and chemical treatments in preparation stages of spinning including boiling or bleaching roving, and in wet yarn formation on the spinning frame. In this way, despite difficulties for the spinners, these yarns up to tex 8 (Nm 125) could be produced on an industrial scale. Unfortunately, the cost of spinning fine yarns is relatively high, mainly because of the low speed of spinning and the low automation of the process (Kozłowski *et al.*, 1994).

There are remarkable differences in the speed of spinning and there is an urgent need for progress in the field of flax fibre processing. A similar conclusion can be drawn from the comparison of spinning costs. It is obvious that only high value end fibre products can be spun at low speed where there are high costs of the yarn production. One hundred per cent linen fine fabrics and apparel made of hackled flax fibre may compete with cotton and man-made fibres despite low speed of spinning and high cost of the yarns. These linen fabrics and goods can compete because they provide high comfort and hygiene as well as elegance. Unfortunately, many excellent advantages are not enough to solve the competition problems of short flax fibre. By the use of traditional spinning technology only coarse yarn or yarns of medium density can be spun from short fibres (tow). Even worse, carded linen yarns are rigid, irregular in every aspect and have low elongation. This is why that for many years short flax fibre lost its market in household and other uses. It should be mentioned that in the traditional flax fibre technology short flax fibre represents about 50% of total flax fibre production. Due to overproduction of short fibres, the new method of making this fibre more clean (impurities of fibre from retted straw make up less than 0.5%, with fibres more fine and homogeneous) is very promising. Such a method was developed by using Rieter machines, which provide more clean and homogeneous short fibres. This system can also be used for cleaning green decorticated fibres, which then can be used for the production of, for example, cigarette paper or

natural linen nonwovens and also coarse, thick technical yarns, for example, string. Among traditional linen yarns, 100% linen yarns for knitting specially softened in or after traditional spinning can be produced. The features of these pure linen yarns are similar to these of traditional weaving yarns and in addition they have favourable qualities for rigidness and softness which make them suitable for knitting. They suit perfectly the requirements for comfortable and hygienic apparel for hot, humid climatic conditions. Also blended linen yarns (with cotton, wool and man-made fibres) are on the market. Tables 5.5, 5.6 and 5.7 present the properties of blended linen yarns, spun by conventional cotton ring spinning systems (Kozłowski *et al.*, 1994).

The features of blended linen yarns spun by long staple ring spinning system are presented in Table 5.8, and those spun by rotor spinning system in Table 5.9.

The rigidness of flax fibre and the low flax fibre elongation forced the development of wrap spinning technology, for example the application of flax blended Parafil yarns in woven and knitted products.

The Parafil spinning system frames are characterized by:

- The length range of processed fibres, including short flax fibres after appropriate preparation.
- The linear mass range of manufactured yarns, including fundamentally the whole range of fineness for yarns made of flax and its blends.
- Lowering of energy consumption in the process of spinning.
- Significantly higher speed of spinning.
- Considerably lower labour consumption of the process – sliver spinning among others (Manys, 1989).

Data on blended short and long staple Parafil linen yarns are presented in Tables 5.9 and 5.10.

Friction spinning of linen blended yarns spun by DREF 2 and DREF 3 spinning machines are presented in Tables 5.11 and 5.12. These yarns are mainly applied to the manufacture of upholstery yarns and knitted apparel (Kozłowski *et al.*, 1994).

Summing up, the main features of the new short flax fibre types, characterized by shorter length, lower thickness, low content of impurities and sometimes improved surface properties, provide the possibility of producing the yarns mentioned above. Narrowing the staple and fineness diagram, reducing impurities and increasing softness are tasks for the future development in processing of short flax fibres. The design of apparel according to fashion expectations may change, but the flax fibre will remain untouched by mode and fashion. Now we have knowledge about producing new homogeneous and fine flax fibres, also functionalized for special application areas including composites, working in different extreme conditions. New linen woven

Table 5.5 Blended linen yarns spun using conventional cotton ring spinning systems (flax tow)

	Flax 10% Cotton 90%	Flax 20% Cotton 80%	Flax 15% PAC 85%	Flax 35% PAC 85%	Flax 15% Viscose 85%	Flax 30% PES 70%	Flax 30% PES 70%	Flax 30% PES 70%
Linear mass (tex)	50	50	49.7	48.2	50	45.4	47.3	51.4
Linear mass irregularity (%)	–	–	1.2	1.1	–	2.7	4.1	4.4
Strength (cN/tex)	11.5	10.3	18.2	14.4	10.3	17.3	19.5	15.7
Strength irregularity V (%)	13.7	15.6	11.5	12.7	10.4	9.5	12.4	16.4
Elongation (%)	6.1	6.0	19.4	18.5	15.2	20.3	20.0	
Twists (n/m)	453	443	428	407	409	486	525	516
Twist irregularity V (%)	4.2	4.4	5.4	2.2	5.9	4.9	9.1	

Note: PAC, polyacrylic fiber; PES, polyester fiber; V, variation.
Source: Krmela, 1993.

Table 5.6 Blended yarns spun by conventional cotton ring spinning technology (30% flax, 50% viscose, 20% cotton) (Rieter)

Linear mass (tex)	64	64	31	31
Linear mass irregularity CV (%)	18.4	19.2	22.0	25.0
Breaking strength (cN/tex)	11.5	11.1	10.0	9.6
Elongation (%)	6.0	6.5	4.8	5.2
Faults (n/1000 m)				
Thin places	20	30	280	500
Thick places	600	980	2000	500
Naps	480	650	1900	2450

Source: Krmela, 1993.

and knitted textiles are in leading collections over the world, the main trend of which is to improve comfort and hygiene features, like low electrostatic properties, natural blocking of ultraviolet (UV) radiation, and giving the best sleeping conditions on 100% flax bedlinen (Kozłowski *et al*., 1994).

There are a number of advantages and disadvantages from the spinning point of view arising from the properties of flax fibres.

The well-known properties of flax fibres are high hygroscopicity, high heat absorption, low static resistance and non-allergic behaviour. These features provide comfort and hygiene of apparels and bedclothes, as well as bedlinen. However, technologically disadvantageous properties of flax fibres exist too:

- long and irregular fibres;
- linear mass of fibres – high and irregular;
- resistibility against splitting;
- rigidness;
- low elongation;
- high content of impurities;
- different colours;
- different mechanical strength.

These disadvantages of flax fibres make the competition against cotton and man-made fibres very difficult and sometimes impossible, especially in the spinning area (Kozłowski *et al*., 1994).

In countries with the highest level of textile technology, more than 70% of flax fibre processed is used in the manufacturing of clothing. The area of clothing manufacture is open to proposals for new types of flax yarns produced by means of new techniques. Some examples of spinning techniques for flax are given below:

- Flax-adapted processing with the use of non-flax conventional techniques of spinning (with mechanical and physico-mechanical processes at the pretreatment stage) is presented in Tables 5.13 and 5.14.

Table 5.7 Blended linen yarns spun by conventional wool spinning system (flax combings)

	Flax 11% Wool 22% PES 41% PAC 26%	Flax 11% Wool 22% PES 41% PAC 26%	Flax 15% PAC 85%	Flax 35% PAC 47% Wool 18%	Flax 15% Viscose 17% PAC 68%	Flax 25% Viscose 17% PAC 58%	Flax 26% Viscose 14% PAC 60%	Flax 30% PAC 70%	Flax 30% Viscose 70%
Linear mass (tex)	48.6 × 1	48.9 × 2	32.1 × 2	32 × 2	54	50	55	51	68
Linear mass irregularity (%)	1.8	1.3	2.1	2.7	2.48	2.6	4.4	2.3	5.4
Strength (cN/tex)	13.1	15.5	13.8	6.5	12.9	13.0	9.9	9.9	7.3
Strength irregularity V (%)	12.5	9.3	12.1	10.1	3.9	4.2	5.0	5.4	7.5
Elongation (%)					12.8	10.7	14.9	12.6	11.0
Twists (n/m)	434	180	–	–	360	340	326	330	334
Twist irregularity V (%)	6.5	7.2		6.7	3.3	3.5	1.9	3.6	4.3

Source: Krmela, 1993.

Table 5.8 Rotor blended linen yarns

	Linear mass		Breaking strength	
Blend	Tex	CV%	cN/tex	CV%
10% Flax/90% Cotton	30	3.5	8.5	13.5
20% Flax/80% Cotton	42	3.5	8.0	13.5
30% Flax/70% Cotton	50	3.5	7.5	14.0
40% Flax/60% Cotton	64	3.5	7.0	14.0
10% Flax/90% Cotton	30	3.5	6.5	13.5
20% Flax/80% Cotton	42	3.5	6.5	13.5
30% Flax/70% Cotton	50	3.5	6.5	13.5
10% Flax/90% PES	30	3.5	12.0	13.5
20% Flax/80% PES	42	3.5	12.0	13.5
30% Flax/70% PES	50	3.5	11.5	13.5
40% Flax/60% PES	64	3.5	11.5	13.5

Source: Krmela, 1993.

Table 5.9 Wrapped yarns spun by spinning frame Parafil 2000

		Breaking strength			
Type of yarn	Linear mass (tex)	cN/tex	V%	Elongation (%)	Wrapping material (%)
Flax-PL combed yarns					
Flax-PL combed yarn (fibre broken to 220 mm)	86.2	9.4	22.0	4.5	5.5
Flax-PL combed yarn (fibre broken to 200 mm)	75.8	7.5	18.0	1.9	5.5
Flax combed blended yarns					
Flax-PL combed blended yarn, 30% flax, 70% PES	56	20.2	18.0	17.9	6.2
PL combed blended yarn, 30% flax/70% viscose	100	11.5	13.9	9.7	2.3
PL combed blended yarn, 30% flax/70% viscose	50	11.5	19.0	17.9	4.6
PL combed blended yarn, 20% flax/30% PES/50% PAC	86	11.6	14.8	14.1	5.5

Source: Krmela, 1993.

Table 5.10 Wrapped yarns spun by spinning frame Parafil 1000

Specification	Linear mass		Breaking strength		Elongation (%)	Wrapping material (%)	Number of wraps per 1 m	Spinning speed (nm/min)
	Tex	V%	cN/tex	V%				
40% Flax/30% PES/30% PAC	78.7	–	9.7	–	14.5	6.3	330	53
30% Flax/20% PES/50% PAC	98.0	3.5	10.2	14.7	14.1	4.9	130	80
30% Flax/70% PES	54.5	0.2	19.1	13.5	20.0	5.0	130	80
20% Flax/40% PES/40% Viscose	57.7	2.1	14.4	13.0	14.7	5.0	130	80
20% Flax/80% PAC navy blue	51.4	2.9	9.6	20.2	12.7	5.0	130	80
20% Flax/80% PAC beige	55.4	2.5	10.5	12.8	14.6	5.0	130	80
An experiment on manufacturing crewel to be used in knitted top fabrics: 30% Flax/20% PES/50% PAC	450.6	—	14.6	8.5	14.7	3.7	150	120

Source: Krmela, 1993.

Table 5.11 Friction linen blended yarns (spun by spinning machine DREF 3)

Blend		Linear mass			Strength			Elongation (%)	Twist (n/m)
		Tex	Nm	V%	daN	cN/tex	V%		
DREF 3	Core: PAN 38 mm Coat: PES 50%, flax 50% 150 m/min	51.09	19.57	0.7	0.63	12.39	13.5	18.3	–
DREF 3	Core: PES 38 mm Coat: cotton 60%, flax 40% 150 m/min	53.44	18.71	1.1	1.10	20.51	9.4	11.4	–
DREF 3	Core: flax 20%, PES 25%, PAN 55% Coat: PES 50%, flax 50% 150 m/min	82.57	12.11	1.8	0.71	8.65	13.7	9.9	–

Note: PAN, polyacrylonitrile fibre.
Source: Krmela, 1993.

- Flax adapted to be processed using non-conventional spinning techniques (with mechanical and physico-mechanical processes at pretreatment state).

Some new forms of spinning techniques for flax, presented above (excluding traditional ring spinning), have overcome barriers in its processing such as slow speed of ring yarn manufacturing from flax fibres, limited assortment of flax yarns and narrow range of using mixed systems of raw materials in yarn.

The data presented above show that flax fibre has been accommodated for almost all the modern techniques of spinning of recent yarns. New forms of flax make it possible to manufacture new flax yarns of different characteristics in composition with all other natural and chemical fibres used in the production of yarns for household goods and clothing.

Some of the new forms of flax fibre have already been included in commercial offers, while some others, most likely, will be commercially offered, too.

While describing the new phenomenon in flax processing, one must stress the following: just like in the case of traditional flax technology, mechanical or physico-chemical processes used in obtaining new standards of flax quality must be accompanied by the use of highly effective spinning techniques in order to raise the spinning quality of flax fibre.

Nevertheless, the ultimate effect, that is, fineness of the yarn obtained and quality of the final product, associated with fineness, depends on the anatomical structure of elementary flax fibre and on the structure of cellulose and its accompanying substances which in turn depend on vegetation conditions.

Table 5.12 Friction linen blended yarns (spun by spinning machine DREF 2)

		Linear mass		Strength		Elongation		Twist	
No.	Blend	Tex	Nm	V%	daN	cN/tex	V%	%	n/m
4675	Core: fil. PES 167 dtex Coat: flax 20%, PES 25%, PAN 55%, 150 m/min	99.70	10.03	1.6	0.94	9.43	4.0	21.1	–
4675	Core: fil. PES 167 dtex Coat: flax 20%, PES 25%, PAN 55%, 150 m/min	161.45	6.19	1.1	0.78	4.83	20.9	13.3	–
4675	Core: fil. PES 167 dtex Coat: flax 20%, PES 25%, PAN 55%, 170 m/min	152.00	6.58	1.0	1.28	8.42	6.3	20.2	–
4689	Core: fil. PES 167 dtex Coat: flax 20%, PES 25%, PAN 55%, 170 m/min	105.26	9.50	1.0	0.95	9.03	5.6	19.7	–
4689	Core: fil. PES 167 dtex Coat: flax 20%, PES 25%, PAN 55%, 130 m/min	159.06	6.29	1.1	0.71	4.44	16.8	12.5	–
	Core: fil. 167 dtex Coat: flax 100 %, 130 m/min	108.34	9.23	0.4	1.04	9.60	8.0		–
	Core: fil. PES 167 dtex Coat: flax 100%, 130 m/min	149.48	6.69	1.0	0.85	5.68	21.3	6.3	–

Note: fil. PES, filament polyester fiber.
Source: Krmela, 1993.

Table 5.13 Spinning techniques – ring spinning for short and long staple fibres

Ring spinning for short staple fibres	
Length of fibre (mm)	17–28
Fineness of fibre (tex)	0.6–1.6
Breaking strength (cN/tex)	34.0–18.0
Impurities (%)	0.0–0.7
Degree of polymerization, DP	2.600–2.200
Degree of whiteness	½ – ¾
Ring spinning for long staple fibres	
Length of fibre (mm)	52–110
Fineness of fibre (tex)	0.75–2.2
Breaking strength (cN/tex)	25.0–19.0
Impurities (%)	0.2–1.5
Degree of polymerization, DP	3.200–2.000
Degree of whiteness	up to ¾

Source: Manys, 1991.

Table 5.14 Spinning techniques – rotor spinning, friction, friction/combined spinning

Rotor spinning – open end (OE)	
Length of fibre (mm)	24–32
Fineness of fibre (tex)	0.4–0.8
Breaking strength (cN/tex)	18.0–14.0
Impurities (%)	0.0–0.1
Degree of polymerization, DP	2.000–3.000
Degree of whiteness	up ¾
Friction spinning – OE DREF 2	
Length of fibre (mm)	38–64.3
Fineness (tex/Nm)	1/1000–2.64/388
Impurities contents (%)	0.2–1.1
Degree of polymerization, DP	2.700–3.000
Degree of whiteness	–
Friction/combined spinning DREF	
Length of fibre (mm)	20/150
Fineness (tex/Nm)	0.8–1.5
Impurities (%)	0.3–4.0
Degree of polymerization, DP	2.200–3.200
Degree of whiteness	
Hollow spindle (wrapping)	
Short staple system, for example, Parafil 1000	
Length of fibre (mm)	84–112
Fineness of fibre (tex/Nm)	0.8–1.7
Breaking strength (cN/tex)	15.0–27.0
Impurities (%)	0.6–1.2
Degree of polymerization, DP	2.000–2.600
Degree of whiteness	–

(*Continued*)

Table 5.14 Continued

Long staple system, for example, Parafil 2000	
Length of fibre (mm)	24–36
Fineness of fibre (tex/Nm)	0.7–1.8
Breaking strength (cN/tex)	19.0–23.0
Impurities (%)	
Degree of polymerization, DP	1.900–2.300
Degree of whiteness	up to ¾

Source: Manys, 1991.

New spinning techniques have been developed that are used to obtain new flax fibre types and 100% new fibre-containing yarn, as well as blended yarns. For instance, these fibres were adapted to the production of 'cotton-like' flax fibres (see 'cottonization') by spinning techniques such as OE (open end), wrap, friction DREF 3, for production of 100% linen and blended linen yarns for fabrics, knitting, especially suitable for apparel, socks, shoes, household goods and upholstery fabrics (Manys, 1991).

5.10 Bleaching, dyeing

Generally raw linen fabrics and knitting are grey and coarse. They should be bleached, dyed, printed and finished to be of appropriate quality and performance for the needs of apparel and textiles for the home. After these processes, they become more attractive and soft, and their area of potential application is enlarged. These treatments are widely used in the linen textile industry. The following subsections present some data on bleaching, dyeing, printing as well as finishing of linen fabrics.

5.10.1 Bleaching

Until the turn of the nineteenth and twentieth centuries, our ancestors generally used open space and sun operation for bleaching flax fabrics. For this purpose they put linen fabrics in supported positions above the field, towards the sun and exposed them to high humidity conditions by spraying water on the fabrics. The UV radiation from the sun (wave length shorter than 400 nm) decomposed water on the fabric surface to create active oxygen '*in statu nascendi*', part of which formed ozone O_3. Bleaching of linen fabrics by using this process took a long time, but it was not hazardous to the environment. To improve this bleaching process, alkaline potash (from burned wood) and organic acids coming from fermentation of starch and milk (buttermilk) were used. This traditional bleaching process was known as crafting or grassing. After the discovery of chlorine (1774) and the development of hypochlorites at the end of nineteenth century, calcium hypochlorite (bleaching powder) came into use. After 1920 chlorine gas and acidified

hypochlorite were used. In the early 1930s the modern procedure of boiling in peroxide was introduced.

The major bleaching agents for textiles are hydrogen peroxide (most widely applied), sodium hypochlorite, sodium chlorite and peracetic acid (oxidative bleaching agents). Silk and wool are bleached by using, for example, sodium dithionite, the reductive agent, belonging to a group called often 'hydros' (Holme, 2005).

The history of the achievements in the scope of bleaching of cellulosics are presented in Table 5.15, while major organic stabilizers used in peroxide bleaching are given in Table 5.16.

It is important in bleaching that there is no negative influence on the depolymerization of cellulose, achieving high whiteness and moderate weight loss. The principal operations in industrial modern linen bleaching are alkaline boiling or scouring, acid chlorination, sodium hypochlorite solution bleaching, hydrogen peroxide bleaching and sodium chlorite bleaching. Worth

Table 5.15 Nine major milestones in the bleaching of cellulosics

Process	Approximate date of significant usage
Sulphuric acid scour	1756
Chlorine bleach	1790
Le Blanc process for soda ash	1791
High pressure kier	1815
Rope washer	1830
Enzyme desizing	1900
Peroxide bleaching	1925
Continuous peroxide bleaching	1939
Fluorescent brightening	1950

Source: Pratt, 1994.

Table 5.16 Organic stabilizers (sequestrants) that have been used in peroxide bleaching

Gluconic acid
Glucoheptonic acid
Aminocarboxylates
Nitrilotriacetic acid (NTA)
Ethylenediaminetetraacetic acid (EDTA)
Diethylenetriaminepentaacetic acid (DTPA)
Organophosphonates
Aminotri (methylenephosphonic acid) (ATMP)
1-Hydroxy ethylidene-1,1-diphosphonic acid) (HEDP)
Ethylenediaminetetra (methylenephosphomic acid) (EDTMP)
Diethylenetriaminepenta (methylenephosphonic acid) (DETPMP)

Source: Lewin, 1984.

mentioning is the intensive development of the use of molybdenum catalyst for effective bleaching (without chlorine components) (Holme, 2005). There has been growing awareness that the use of chlorine compounds can result in the formation of hazardous dioxins resulting in the elimination of chlorine bleaching and the utilization of mainly hydrogen peroxide with sodium silicate (Kernaghan *et al.*, 1992a).

Single bath full bleaching of flax fibres using an activated sodium chlorite hexamethylene tetramine system for the bleaching of flax fibres is based on the activation of sodium chlorite by hexamethylene tetramine (HMTA) in the presence of an anionic wetting agent. The bleached flax fibres are assessed for critical properties, namely whiteness index, loss in fibre weight, tensile strength, content of carboxyl and carbonyl groups. Optimal bleaching conditions for the flax fibres are $NaClO_2$ 5 g/L, HMTA 0.25 g/L and wetting agent 1 g/L, temperature 90°C, time 3 h and liquor ratio 1:50 (Zahran, 2005).

Biobleaching of flax fibres by degradation of lignin with *Phanerochaete chrysosporium* and *Trichosporon Cutaneum* R57 is also used. The enzyme complex secreted by the filamentous yeast *Trichosporon Cutaneum* R57 can be successfully applied to bleaching of flax fibres and fabrics. Also *Phanerochaete chrysosporium* produces enzyme that shows a similar behaviour. After the treatment, the fibre increased its lightness with a stronger lignin degradation and removal during processing.

A sodium chlorite bath for scouring and bleaching of linen fabric in a one-step process activated by glyoxal as an organic reducing component with two aldehyde functional groups was investigated as well. This treatment was tested under a variety of conditions and the following properties were monitored: degree of whiteness, loss of fabric weight, wettability, carbonyl group content, carboxyl group content, percentage of sodium chlorite decomposition, and tensile strength of fabrics. The optimum conditions were found at 60°C, pH of 10.35 with aqueous solution containing 5 g/L $NaClO_2$, 6 mmol/L of glyoxal, wetting agent 2 g/L for 120 min at the liquor ratio 1:30 (Zahran, 2010).

A new improvement of linen bleaching was made by the Technical and Natural Sciences Research Development Innovation Institute at the 'Aurel Vlaicu' University of Arad, Romania, according to which the acceleration of bleaching can be achieved by using a catalyst of the formula Na_2 [Mo_2O6 (C_2O_4)]·4 H_2O.

5.10.2 Dyeing

All fibres and fabrics usually have to be dyed. The use of natural dyestuff for dyeing fibres and fabrics dates back to very early times and is associated with the human understanding of beauty. Primitive man, while watching nature, dreamed about capturing its colours. He found them in roots, berries, branches, barks, leaves, flowers, fruits, minerals, shells and insects. Skill

mastering in plant usage and particulars of knowledge of the use of auxiliary chemical substances were a secret art in the past.

Dyeing with natural dyestuff s (of plant and insect origin)

In ancient Egypt dyeing was at an advanced level. The first information about dyeing of linen concerned early Dynastic Egypt and was reported by Plinius (AD 79). The earliest methods of dyeing used mordant and mineral dyes. This knowledge was adopted by the Israelites and Phoenicians from the Egyptians and then by the Greeks and Romans.

The discovery of America and its exploration showed the independent dyeing development on high cultural level in Mexico and Peru. Thanks to sea routes to East India the following dyes became known: cochineal, indigo and dyes from trees such as *Fernambuco Caesalpinia brasilensis* and *Sanders terocarpus Santalinus*. Dye producers keep secret their knowledge and protected it from becoming commonly known.

Natural dyes can also be derived from plants, minerals, insects and shellfish, although they have many limitations in terms of fastness and brilliancy of shade.

The archaeological findings indicate that dyes from natural sources have been used to colour textiles for the last 6000 years. Dye research conducted within an ethical and ecological framework has long been the focus of artists, conservationists, chemists, etc.

A dyestuff is a pigment or colouring matter dissolved in water or some other vehicle so that it may be able to stain, more or less permanently, the substance to which it is applied. Natural dyes are classified into two groups – substantive and adjective dyestuffs:

- *Substantive dyestuffs* dye the fibres directly. These kinds of dyes were probably the first type of dyes used by early people. Substantive dyes usually rich in tannins come from bark, leaves and fruit. This group includes also vat dyes – indigo *Indigofera tinctoria* and woad *Isatis tinctoria.* The colouring matter of vat dyes is not directly soluble in water. The colour develops on exposure to oxygen present in air. In indigo and woad the dye matter is the substance indican.
- *Adjective dyestuffs* need mordant to fix permanently to the textile fibres. Metallic salts of alumina, chromium, iron, and tin help to create affinity between the fibre and pigment. Mordant often produces a much stronger colour on fibres. Examples of such dyes are: madder, logwood, cochineal, fustic.

Some dyes do not have a solubilizing group. In such cases a temporary solubilizing group is introduced in the dye at the time of application. A classical example of such a dye is indigo. Indigo is water-insoluble. After

application, the reduced form of the indigo is converted back to its original form by air oxidation of the material. This process is known as 'vatting', and indigo is classified among vat dyes.

In scientific papers and books on colour, the primary colours are identified as red, blue, yellow and green (Schmidt-Przewozna, 2001).

Blue

Indigo (*Indigofera tinctoria*) is the king of dyes, originally closely related to life and culture. In Egypt, deep blue was reserved for the daughters of the pharaohs who painted their breasts with blue and gold. Indigo-coloured garments have been found in Thebes. In most traditional cultures blue was the colour associated with class status. Blue was also obtained from woad (*Isatis tinctoria*). This plant contains the same blue colouring matter as indigo, but in lower concentrations. Woad was probably the first blue dyestuff used in America. From the thirteenth century many farmers and dyers in Germany, England, France and Italy grew rich on woad production. The woad vat was basically similar to the indigo vat requiring care in controlling the fermentation process by which the colouring agent was reduced to its soluble form. Nowadays, this dye is little used in England and France by artists and designers, although the designer Oliver Lapidus showed an indigo collection at a fashion show in Paris in 1999. The fabrics were dyed at the Lambert workshop in France.

Red

In European culture red was reserved for noblemen. In ancient times purple was obtained from shells. Historically, Poland was renowned over a long period for the source of one very important dyestuff, Polish cochineal (*Porphyrophora polonica* L.). From the Middle Ages until the end of the sixteenth century this insect was used for dyeing silk with an alum mordant. It thrived on the perennial knawel (*Scleranthus perennis* L.), which can be found in parts of northern Europe including Poland. The precious dyestuff was obtained from the cysts of the insect. The insect is a root pathogen, therefore it was necessary to dig the plant out to gather the cysts. Poland exported the dyestuff to Italy, the Netherlands, France and other parts of Europe, and also to Turkey. The high point of this trade was in the fifteenth century. In the middle ages dyers used Polish cochineal and also kermes (*Kermes vermilio* Planchon); sometimes both dyestuffs were used together. There are sometimes mistakes in sources referring to these insects as the differences between the two dyestuffs are not always realized. After the discovery of America, the use of both of them declined as they were replaced by the richer dye insect, cochineal (*Dactylopius coccus* Costa), found in Mexico and Central America. Other important red dyes were the plant dyes madder (*Rubia tinctorum* L.) and safflower (*Carthamus*

tinctorius L.), both of which were planted commercially. Other, more local, dyestuffs were also used in Polish workshops. These included the fugitive dyestuff obtained from bilberry or blueberry (*Vaccinium myrtillus* L.) and the colourant extracted from the stems of the nettle (*Urtica dioica* L.), which were collected for use in the autumn when they are red. Dyers throughout Europe imported red dyestuffs from other parts of the world and Polish dyers were no exception: brazilwood was imported from southern India and Malaysia (*Caesalpinia sappan* L., sappan wood) and, after the discovery of the Americas, from parts of Central and South America (*C. echinata* Lam and other species). Red sandalwood (*Pterocarpus santalinus* L.), like sappan wood, was imported from southern India (Schmidt-Przewozna, 2005).

Yellow

The colour of the sun, symbolizes growth and possibility. The principal yellow of the ancients was reserved only for the emperors. The yellow dye for textile manufactures was obtained from Weld *Reseda luteola* L. It is possible to obtain a good yellow from turmeric (*Curcuma longa* L.). The main producer of this plant was India, but it was also cultivated in China, South East Asia and tropical countries. Yellow-dyeing plants are common in nature. It is possible to create a full palette of yellows by cultivated, wild and forests plant. Other sources of yellow with considerable tinctorial properties are: onion *Allium cepa*, dyer's chamomile *Anthemis tinctoria*, babery *Berberies species*, birch *Betula species*, heather *Calluna vulgaris*, safflower *Carthamus tinctorius*, drug fumitory *Fumaria officinalis* L., common horsetail *Equisetum avense* L., heart's-ease *Polygonum persicaria* L., sorrel *Rumex acetosella* L., common yarrow *Achillea millefolium* L., dandelion *Taraxacum officinale* L., dyer's coreopsis *Coreopsis tinctoria* L., coreopsis *Coreopsis grandiflora* L., French marigold *Tagets species* L., dyer's greenweed *Genista tinctoria* L. and many others.

In some parts of the world, natural dyes are still used to colour traditional textiles. In India, Eastern and Central Asia, South America and Africa natural methods of dyeing are still alive in residual forms. The world tendencies aim towards their recovery (Schmidt-Przewozna, 2001, 2005, 2007, 2009).

Dyeing with synthetic dyestuffs

Until the 1950s only about 10% of the linen fabric produced was dyed. In the twentieth century, natural dyestuffs application in the textile industry almost disappeared and was replaced by chemical dyestuffs. After the 1980s it was observed that especially fine apparel was dyed.

Direct dyestuffs were the first important synthetic class, developed in the nineteenth century. By the early twentieth century the vat, azoic and sulphur classes of dyestuffs as well as reactive dyestuffs were also applied to dyeing linen. Most commercial dyestuffs for linen are similar to those used for

cotton, viscose and rayon – most of them are applicable to all lignocellulosic fibres. The simple method of dyeing involves exhaustion of dyestuff from electrolyte solution. These dyestuffs belong to a group of direct dyestuffs.

Vat dyestuffs represent the reference standard of fastness and resistance to most agents. In practice, linen fabrics are impregnated by pad mangle using vat dyestuffs. Also these vat dyestuffs are used for dyeing linen yarn, which is dyed in hank form and also in pack form.

The reactive class of dyestuffs achieve retention by formation of a covalent bond with the cellulose. This reaction fundamentally proceeds via hydroxyl group of cellulose, ionized under mild alkaline conditions to form linkage between dye molecule and substrate. There are two possibilities for this reaction mechanism: (a) nucleophilic substitution or (b) nucleophilic addition.

Printing

For decoration purposes especially for wall coverings, domestic furnishings, tea towels, upholstery, curtains and blinds made from linen, special dyestuffs are used. Very little printing of the expensive apparel weight fabrics is performed. Obviously absorbency and penetration are potential problems with linen (Kernaghan *et al*., 1992b).

Nowadays, there is growing awareness regarding environmental pollution by textile effluents, for example, from dyeing. There are many dyestuffs, especially those containing heavy metals, that are forbidden. In Europe, the REACH (Registration, Evaluation and Authorization of Chemicals) system regulates the use of many chemicals and some dyestuffs as well. On the other hand, increasing awareness concerning environmental issues contributes to the return to natural dyeing and many research centres throughout the globe carry out research on making natural dyeing systems more effective and economically reasonable.

5.11 Finishing

Commercial modified glyoxal-based resin is used for the chemical end finishing of linen and ramie fabrics in wet fixation. This technology significantly reduces the tensile and tearing properties and decreases the resilience of the fabrics and results in a considerable increase in wet recovery angle (Kim, 2005).

Butanetetracarboxylic acid (BTCA) and citric acid are used to replace formaldehyde releasing agent in cellulosic fabric finishing. BTCA can esterify hydroxyl groups of cellulose to produce a graft copolymer of cellulose (Bertoniere, 1999).

Liquid ammonia treatment is applied in large scale production for improving quality of cotton/linen warp/weft blended fabrics. This treatment improves the dimensional stability, abrasion resistance, appearance after washing and creasing recovery (Dornyi, 2007).

Liquid ammonia treatment was first used by Norwegian company TEDECO in the 1970s to upgrade woven cellulosic fabrics. Then it was used in Japan for cotton and also for linen. The treatment for linen was developed by the Institute of Natural Fibres (Poland) in cooperation with Nissihinbo Industries Inc. (Japan). The best results were obtained when liquid ammonia and low formaldehyde emission resins were used. The 'Super Soft Peach Phase' (SSP) imparts the following properties to non-iron products:

- low shrinking after washing,
- increased wrinkle resistance,
- increased elasticity,
- increased strength,
- softness to touch,

Liquid ammonia penetrates cellulose very rapidly and forms complexes with its hydroxyl groups after breaking hydrogen bonds in crystalline as well as in amorphous regions of the fibres. Ammonia molecules do not dissolve cellulose, but they are capable of increasing the distances between the cellulose chains through penetration into crystallites. The mechanism of the chemical reaction is not fully recognized; however, it should be mentioned that in finished linen fabrics a small content of nitrogen was found. Probably ammonia was bonded to the carboxyl groups of cellulose chain. A significant increase in intensity of the absorption band at 895.78 cm^{-1} suggests the formation of $CONH_2$ species (Kozłowski and Helwig, 1996; Kozłowski and Manys, 1996).

Liquid ammonia treatment is similar to the mercerization process, which is very widely used for cotton. The mercerization process (with sodium hydroxide) improves some properties of linen, namely it increases strength, dyeability, water sorption and lustre. After the mercerization process, linen fibres are untwisted and become almost round in cross-section with almost complete disappearance of the convolution. This treatment involves a short exposure to aqueous solution of NaOH which results in the transformation of native cellulose I crystal structure into the regenerated cellulose II crystalline modification (Kozłowski and Helwig, 1996; Kozłowski and Manys, 1996).

5.12 Recapitulation

The present situation in the flax market can be summarized as follows: the main demand for flax fibres is for the superior high grades suitable for the easy production of fine fibres for flax yarns with no major spinning problems. Since then, the great majority of the world producers of flax fibres have been facing the dilemma of either improving the specifications of their flax fibres to meet the high level required, or totally stopping production due to the almost complete absence of demand for inferior quality textile flax fibres.

Recent efforts aimed at finding a solution to the alarming situation of flax fibres resulted in establishing the following facts:

- In general, flax straws potentially contain very fine fibres, which naturally have superior high textile qualities; however, they become apparent only when these fibres are properly and smoothly extracted from the other plant tissues surrounding them, and are obtained in pure, intrinsic, individually separated single filament form, which have stable more homogeneous textile characteristics, inherent to each variety.
- The only method of vegetal fibre extraction that steadily produces pure intrinsic flax fibres of obvious natural excellent textile characteristics, is the newly discovered osmotic pressure method that liberates the fibres smoothly, without affecting their natural excellent textile characteristics and without polluting the environment.

To maximize the yield of the fibres, flax cultivation should be aimed at getting the greatest number of fine, slender, unbranched stems through increasing the density of population of the plants by giving each plant the minimum necessary free vital space around it and by evenly distributing the sowing seeds. In Egypt, the maximum potential number of fine stems per square metre has been practically increased to 2500.

To maximize the crop of seeds, flax cultivation should be performed in such a way that the greatest number of capsules is obtained, which is attained by giving the plants the maximum vital free space around each plant, through a regular distribution of the sowing seeds, to allow the plants to give birth to the greatest number of tillers and as much branching and sub-branching, to bear the greatest number of flowers which would produce, after pollination, the greatest number of seed-containing capsules.

Specific free space around each plant is essentially needed to allow the full expansion of its root and rootlets, in all directions and at different levels, in order to collect all the water that touches them, without interfering with other nearby plant rootlets. Otherwise, the nearby plants will share the amount of available water and each one of the plants will only grow according to the amount of water it acquires. The crop maximization of any plants spaced too closely is practically impossible.

Plants grow according to a number of influential factors, some of which are natural factors that are out of our human control, while the other ones can be effectively controlled. If any of these factors is partially absent, the final overall effect will be limited to the level of presence of this factor, in accordance with the physical law of the effective capacity of a wooden barrel being equal to its shortest stave.

In flax cultivation, if the required free space around each plant is not respected, the actual crop of each plant crop will be limited to the actual

percentage of the required free space around each plant. If, for example, the required free space around each plant is 10 cm and the actual free space around a plant is only 4 cm, the optimum growth and crop of this plant will not exceed 40% of its natural potential capacity, even if all the other influential factors are 100% present.

In equal weights, thinner flax straws contain relatively more fibres than the thicker ones, and need less time to be properly extracted from other surrounding plant tissues. The best average diameter of flax straw varies between 0.7 and 1.0 mm. In this case the total yield of fibre can reach the level of about 35–37% of total mass.

5.13 Conclusions and future trends

The emerging applications of flax fibre, as a fabric and as a non-woven, are in the areas of more eco-friendly composites that are used in modern building materials and in the construction of automobiles. It is also possible to modify the properties of flax fibres and produce nano-flax fibres. Currently, there is ongoing discussion regarding genetic modification of flax, for example, in creating '*in statu nascendi*' polyhydroxyalkanoates, which are naturally created polyesters. The new techniques and technology of processing flax fibres give opportunities to obtain very fine yarn and thin non-iron flax textiles.

5.13.1 Conclusions

The new improved aspects of flax are due to the innovations applied to its cultivation and to the extraction of its fibres. The maximization of flax crops to obtain either seeds or fibres, and the stabilization and improvement of fibre specifications, have been the results of first-hand scientific approaches, which updated and enriched the available knowledge about the morphology of this ancient plant, and about the impact of the influential natural and human factors on its behaviour during growth and subsequent development.

From now on, flax manufacture will be much improved. During cultivation, its behaviour is under full control and can practically be directed to economically and beneficially maximize the production of excellent textile fibres or rich oily seeds through two completely different forms of flax sowing seed distributions: for the maximization of fibres, each plant should be surrounded by the minimum necessary free vital space; for the maximization of seeds, the plants should be surrounded by the maximum necessary free space.

For the first time, the extraction of flax fibres can be completely mastered and scientifically industrialized, in accordance with known natural forces acting according to physical laws, leading to the steady production of pure, more homogeneous flax fibres that show natural potential superior high textile quality, suitable for the production of the finest possible yarns, without major spinning problems, at reasonable competitive production costs.

As a result, flax industries can look forward to a healthy revival and guard against future unexpected negative situations.

Useful flax knowledge, on the basis of Egyptian experience, in terms of flax cultivation, flax straw processing, linseed oil production, flax tow twine production, production of particleboard and flax hardboard production, is presented in Tables 5.17, 5.18, 5.19, 5.20 and 5.21.

Table 5.17 Useful information – flax cultivation

Item	Rate	Unit
Preparation of the field	4 labour hours	Per hectare
Sowing of seeds for fibres	2500 seeds	Per square metre
Sowing of seeds for seeds	1000 seeds	Per square metre
Mechanical sowing of seeds	¾ labour hours	Per hectare
Hand spreading of seeds	4 labour hours	Per hectare
Artificial irrigation	600 cubic metres	Per hectare
Artificial irrigation	2 labour hours	Per hectare
Chemical weed control	½ labour hour	Per hectare
Manual weed control	100 labour hours	Per hectare
Mechanical spreading of fertilizer	½ labour hour	Per hectare
Manual spreading of fertilizer	2½ labour hours	Per hectare
Amount of fertilizer	Nitrogen 80 units Super- phosphate 300 kg Potassium 350 kg	Per hectare
Mechanical harvest	2 labour hours	Per hectare
Manual harvest	140 labour hours	Per hectare
Mechanical deseeding and tying beets	6 labour hours	Per hectare
Manual deseeding and tying beets	160 labour hours	Per hectare
Weight of imported sowing seeds	5 g	Per 1000 seeds
Weight of local sowing seeds	9.5 g	Per 1000 seeds
Weight of deseeded straw of 1 mm in thickness	0.5 g	One stem
Weight of deseeded straw of 3 mm in thickness	1.5 g	One stem
Volume of flax straw with seeds	18 cubic metres (9.5–12.0 m^3 in Europe)	One metric tonne
Volume of deseeded flax straw	25 cubic metres (11.0–14 m^3 in Europe)	One metric tonne
Volume of flax seeds	1.5 cubic metres	One metric tonne
Even spaces around each stack	3 times its height	Stack height
Flax straw stacking	2 labour hours	Per metric tonne
Stacking of flax seeds in bags	¾ labour hour	Per metric tonne
Permitted humidity for flax straw	10% (16–18% in Europe)	
Permitted humidity for flax fibres	12%	
Permitted humidity for flax seeds	8%	
Straw loading on lorries	3 labour hours	Per metric tonne

Source: Allam, 2008.

Table 5.18 Useful information – flax straw processing

Item	Rate	Unit
Straw sorting	16 labour hours	Per ton
Filling of retting tanks	¾ labour hour	Per ton
Emptying of retting tanks	2 ½ labour hours	Per ton
Setting of wetted straw in cones for even dryness	10 labour hours	Per ton
Mechanical breakage and scutching using turbines	50 labour hours (20 h in Europe)	Per ton
Manual breakage and scutching	300 labour hours	Per ton
Sorting and classification	50 labour hours (20 h in Europe)	Per ton
Pressing of long filament flax fibres in bales of 250 kg	1½ labour hours	Per ton
Pressing of tows in bales of 200 kg	2 labour hours	Per ton
Pressing of fibre waste in bales of 158 kg	2½ labour hours	Per ton
Loading of pressed flax fibre bales	1 labour hour	Per ton
Unloading of pressed bales	¼ labour hour	Per ton

Source: Allam, 2008.

Table 5.19 Linseed oil production

Item	Rate	Unit
Linseed sieving	3 labour hours	Per ton cleaned
Crushing and extracting the oil	72 labour hours	Per ton in containers

Source: Allam, 2008.

Table 5.20 Useful information – flax tow twine production

Item	Rate	Unit
Flax tow carding	20 labour hours (10 in Europe)	Per ton of sliver
Sliver drawings to needed weight	15 labour hours	Per ton of sliver
Spinning and twisting into twine	50 labour hours (30 h in Europe)	Per ton of twine

Source: Allam, 2008.

Table 5.21 Useful information – production of particleboard

Item	Rate	Unit
Cleaning of flax shives	2 labour hours	Per ton cleaned
Shive mixing and material gluing	4 labour hours	Per ton of boards
Sanding and finishing the boards	5 labour hours	Per ton of boards

Source: Allam, 2008.

5.13.2 Future trends

The growing fibre flax in the European Community has increasing potential, although to a limited extent. The total flax fibre market is 17–21 thousand tons. The conditions to obtain the indicated growth are as follows:

(a) restructuring of the entire linen industry;
(b) research on the improvement of the quality of flax fibres (higher fineness, higher fibre content);
(c) enlarging the area of utilization (Coby, 1990).

The main reasons for fibrous flax expansion and flax technology modernization in the twenty-first century are:

- The exhaustion of resources of the major organic chemicals (coal, gas, oil) which dictates the trends towards organic renewable raw materials both for energy and products.
- In the long-term future polymers for textile and non-textile applications might be of plant origin (fibrous plants and wood) supplemented by algae and autotrophic bacteria which can use carbon dioxide directly to synthesize polymers (e.g., bacterial cellulose).
- Fibrous flax and oil flax are perfect plant polymers.
- Rapidly growing ecological trends and ecological awareness create the demand for healthy clothing, environmentally friendly raw materials and technologies.
- The urgent need for sustainable agricultural development is well known and flax could be cultivated in the areas extending from one arctic circle to the other.
- The other reasons for flax expansion in the current century are the valuable new products and by-products associated with flax, for example, reinforced polymers, bio-components based on flax (or other bast plants and derived fibres) instead of glass fibres.
- 100% biodegradable packaging materials based on, for example, waste short fibres, also special pulp and paper (e.g., cigarette paper).
- Brand new products based on linseed (food and nutrition products rich in omega 3 and omega 6 fatty acids as well as in lignans and cyclopeptides; bio-oils for nutrition, cosmetics and also for energy).
- Possibility of cultivation of flax and other bast fibrous plants on soils polluted with heavy metals as alternative crops for non-food purposes and soil reclamation.
- New fibre processing technologies developed in the last decades are similar to those in the so-called yarn revolution which occurred some years ago in the area of cotton, wool and man-made fibre industries.

This requires new types of flax fibres that are cleaner and highly homogeneous. The shortening, splitting and cleaning necessary to spin flax by modern techniques are far beyond the reach of traditional fibre assortments. These new types of flax fibres can be blended with all natural and man-made fibres. The newest 'dream concept' in the area of flax fibre production is to develop one integrated process which should include harvesting, deseeding and fibre extraction performed by one machine in the field at harvest time. It is likely that by using genetic modifications (GM) this dream can come true.

- The new research achievements and progress made in the field of flax production and processing have also resulted in the improvement of the competitiveness and coexistence of flax with other fibrous materials (both natural and man-made ones).

Summing up, new types of flax fibres have been developed that are characterized by a controlled level of lignin and pectin, adopted to conventional and unconventional spinning and other processing systems (e.g., non-ovens), and linen blended yarns and new fields of the application of flax fibre and by-products emerged, namely healthy linen apparel, protective and anti-UV and anti-electrostatic clothing (see Chapter 4, vol. 2), knits such as socks, pullovers, healthy nonwovens and upholstery, bedlinen, biocomposites, degradable packaging, and linseed-based new products rich in omega 3 and omega 6 fatty acids as well as rich in lignans (like Bioflax – see Chapter 11, vol. 2).

These data give excellent confirmation that the Latin name '*Linum usitatissimum*' given to flax by Carl Linnaeus in the 1770s is well-deserved (Kozłowski, 1996).

5.14 Sources of further information and advice

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6

Bast fibres: hemp cultivation and production

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Abstract: Hemp (*Cannabis sativa*) has been utilised as a source of fibre for the production of a wide range of textiles- and fibre-based products. This chapter first reviews the hemp plant and its cultivation for the production of hemp fibre. It then discusses the ways in which the hemp straw is retted and mechanically processed to obtain both long and short fibres. The chapter then describes fibre cleaning and carding in preparation for spinning and the spinning process.

Key words: *Cannabis sativa*, cultivation, retting, fibre extraction, spinning hemp.

6.1 Introduction

Fibre production from hemp (*Cannabis sativa*) has been conducted over many centuries, for end uses from textiles, ropes and sails, to matrices for industrial products in the modern age. It has been cultivated and utilised in a diversity of countries in many parts of the world, in both the northern and southern hemisphere.

6.1.1 Classification of hemp plants

The name 'hemp' is a term most commonly used in connection with the *Cannabis sativa* plant, its components (seeds, stems, leaves) and any products (foods, fibres or biomass) extracted and manufactured from them. The term is also used as a name applied to a large number of other fibre bearing plants and their products, such as Sisal hemp (*Agave rigida*) or Manilla hemp (*Musa textalis*), but these plants and fibres are neither related nor associated with *C. sativa*. The widely accepted common usage of the singular term 'hemp' in the English language as a description of a plant or product, for example hemp crop or hemp fibre, is in reference to the plant source *C. sativa*.

6.1.2 A brief history and background of hemp

Hemp (*C. sativa*) has been widely cultivated as a source of bast fibres and/or seeds. The plant is native to India and Persia, but over the last 6000 years

it has been cultivated in nearly all temperate and tropical countries of the world and is likely to be one of the oldest non-food crops known (Schultes, 1970; Vavilov, 1926). Historically, hemp fibres were used for the production of rope, cordage, fabrics and paper, and the debris of such industries is a significant source of archaeological evidence of hemp production. For example in the British Isles hemp fibres were widely used in the production of ship rigging and sail cloth from AD 800 and retting sites have been used to identify the locations of production (Schofielda and Wallerb, 2005).

Hemp seed has been primarily produced for the oil that can be crushed from it and subsequently used in culinaries and manufactures such as soaps, paints, lubricants and cosmetics. The residue seed meal has been used as an animal feed.

During the sixteenth, seventeenth and eighteenth centuries the major fibre crops found growing in Europe, North America and Russia were hemp and flax (Aber, 1980; Pounds, 1979). However, during the nineteenth century the expanding cultivation of cotton and jute caused the world area of hemp to decline, while during the twentieth century the widespread production and adoption of synthetic fibres as an alternative to natural fibres caused a further decline in hemp crop production (Atkinson, 1964). Additionally, during the twentieth century the production of hemp was made illegal in many countries as part of controls on the recreational use of cannabinoids.

After the Second World War the major producer of hemp fibre was the Soviet Union, particularly in Russia and the Ukraine. In the latter hemp fibre production peaked at 3000 km^2 in the early 1950s (Holoborod'ko, 1995). Large areas were also grown in Korea, China and France. In the late twentieth century a number of countries, particularly the European Union (EU) and Canada, stimulated the production of low-cannabinoid hemp for industrial end uses. As a result the area of hemp production expanded in the EU and Canada, and its cultivation developed and expanded in other countries such as Australia and Chile.

In the first decade of the twenty-first century the production of hemp is primarily found in Canada, China and the EU, especially since the latter's expansion to include a number of East European countries formerly part of the Soviet bloc. The presence of hemp in Europe is partly because of the allied production of flax fibre and partly the result of the agricultural subsidies for farmers and the financial support paid to hemp fibre extractors. However, the area of cultivated hemp in the EU has been in significant decline over the first decade of the twenty-first century, with the total cultivated area declining from 39 000 ha in 1998 to 11 900 ha in 2007 (Table 6.1).

However, this decline in hemp production is mostly the result of declining production in Germany, Spain, the UK, the Netherlands and Finland, and this has been partly offset by an expansion of hemp production in Poland and Belgium.

Table 6.1 Hemp production area of major world hemp producers by country 1998–2007

	1998	1999	2000	2001	2002	2003	2004	2005	2006	2007
Austria	1000	300	300	800	300	400	400	300	500	0
Belgium	0	0	0	0	0	0	0	200	1100	1400
Canada	2400	14 205	5485	1312	1530	2733	3531	9725	19 458	6132
China	15 000	11 300	9700	12 300	14 800	10 000	20 100	11 000	9600	17 800
Finland	1300	300	200	100	0	0	100	0	100	0
France	9700	10500	7700	7000	7700	9500	8400	9300	8100	7400
Germany	3500	4000	3000	1900	2000	2600	1700	2000	1200	800
Hungary	N/A	N/A	N/A	N/A	N/A	N/A	500	300	200	0
Italy	200	200	200	200	200	900	900	200	200	400
Netherlands	1100	900	800	1000	2100	1500	0	100	0	100
The Poland	150	50	50	150	100	100	100	100	800	1100
Romania	3100	1300	500	600	1000	1200	N/A	N/A	1300	100
Spain	19 900	13 500	6100	800	700	700	700	900	0	0
UK	2500	2500	2200	3300	1400	2400	2000	1300	1700	600

N/A = not available.
Source: Eurostat (2008), Chaudhary (2008), FAO Stat (2009).

There is a significant amount of production in Canada but this is primarily seed hemp rather than fibre hemp. However, the areas grown fluctuate significantly from year to year (Table 6.1). China is the world's largest producer of hemp (*C. sativa*), but the production of ambary hemp, which is otherwise known as Kenaf (*Hibiscus cannabinus*), is also widespread in China and can inflate the perceived 'hemp' area in Chinese fibre crop production statistics (Anon, 2008). A number of other countries, such as Australia and North Korea, have developed the production of hemp over recent years, but there are few reliable statistics on hemp production in these countries.

6.2 The hemp plant

Hemp is a plant from which bast fibres can be extracted from the stems of the plants. However, the plant differs widely in its growth and development characteristics according to genetic type and the location.

6.2.1 Botany of hemp

Hemp (*C. sativa*) is a tall, slender annual, with a vigorous growth habit, of the Cannabaceae family. The plant is most commonly recognised by the shape and pattern of its leaves. The first seedling leaves are sessile leaves, but all subsequent leaves have a petiole (Fig. 6.1). The first pair of true leaves have a single elliptic blade with serrate margins, a leaf of the second pair of true leaves has three serrate leaflets emerging from the petiole, a leaf from the third pair of true leaves has five serrate leaflets, and so on. This pattern continues up to between 9 and 12 leaflets (Stearn, 1970). Phyllotaxis is opposing in young

6.1 Hemp seedlings.

hemp plants, but as flowering begins phyllotaxis becomes alternate and the leaflet number declines (Heslop-Harrison and Heslop-Harrison, 1958).

Hemp plant stems are furrowed externally, hollow and can range from 5 to 25 mm in diameter. When grown at a high plant population they do not branch, and so when grown for fibre production the seeds are planted closely together (Bennet *et al.*, 2006; Schafer and Honermeier, 2006). The height achieved by a hemp plant can range from 1 to 5 m at harvest but this depends on growing conditions, husbandry and cultivar (Schafer and Honermeier, 2006).

Hemp is a short day plant; its flowering is delayed by long days and hastened by short days. In general hemp flowers in the autumn when the photoperiod falls to between 12 and 13 h/day and only flowers slowly at day lengths above 14 h/day, but it depends on the cultivar and geographical location (Borthwick, 1947). Some cultivars are adapted to maturity in short days in southern latitudes, while others are adapted to bearing seed in the comparatively longer days of northern latitudes (Bredemann *et al.*, 1956).

Hemp is primarily dioecious; the pollen-bearing parts are found on one plant and the seed-bearing flowers on another. The male (staminate) and female (pistallate) plants are indistinguishable prior to flowering. The male inflorescences can be first identified by the development of round, pointed flower buds with five radial segments, while the female inflorescences can be identified by the presence of a calyx (Clarke, 1980). The proportion of male to female plants can vary widely from 40% to 60%. The male inflorescences go on to be strongly branched with few or no leaves, while the female inflorescences are leafy and unbranched. The life expectancy of the males is limited to a point soon after anthesis, while the female plants live 3–5 weeks after anthesis until the seed is fully ripe. As a result of the latter, where hemp has been grown for fibre production using manual labour the male plants have been harvested, by hand pulling, approximately 3 weeks prior to the female plants (Kirby, 1963).

Monoecious cultivars, such as the cultivar Felina, have been developed and are commercially produced, and they may have the advantage because of their uniform maturity in comparison to dioecious types but they tend to exhibit lower fibre yields. Monoecious types are suited to dual-purpose seed and fibre crops rather than fibre crops alone.

Hemp seeds are achene fruits; a single seed is contained within a shell and tightly encompassed in the thin wall of the ovary. The seed is typically an ellipsoid shape 2–7 mm in length and 2–4 mm wide at maximum diameter. The seed can appear in varying colours from a light brown to dark green and it contains about 35% oil and 25% crude protein (Dempsey, 1975).

6.2.2 Morphology of hemp stems

In cross-section, hemp stems can be thought of as consisting of two major parts. First there is the tissue outside the vascular cambium, often referred

to as the 'bark' or bast, and second the tissues inside the vascular cambium which are often referred to as the 'core'.

The outer bark contains three component parts: epidermis, cortex and phloem. The epidermis is covered with a thin layer of wax, which is a barrier preventing excessive moisture loss and provides some protection for the plant. During the retting process bacteria will access the stem via this layer. The cortex consists of circular cortical cells that contain pectins but it is not significantly lignified (McDougall *et al.*, 1993).

The phloem contains the primary bast fibres and sieve tubes, and it is sometimes referred to as the bast layer. This layer contains bundles of fibres and runs the full length of the plant stem. The length of the ultimate fibres is reported to range from 2 to 60 mm, with a mean length ranging from 40 to 50 mm. The fibres tend to be thicker near the bottom of the stem in comparison to the top. The phloem has been noted to also contain secondary bast fibres that arise from the cambium (Kundu, 1942). The length of the secondary bast fibres is reported to be <5 mm. Primary bast fibres can range from 20 to 35 microns, while secondary bast fibres are significantly narrower at 15 microns (Kundu, 1942).

The inner core constitutes the cambium and the xylem. The cambium consists of tissue that separates the layer of fibres from the woody xylem tissue. The xylem consists of vessel members, ray and paratracheal cells and libroform fibres (Easu, 1965); surrounding the pith cavity is a central hollow that runs the length of the stem. The libroform fibres are approximately 0.5–0.8 mm in length and about 25 microns wide.

6.2.3 Chemical composition of hemp stems

The distribution of chemical constituents of hemp stems varies significantly between the outer bast and the woody core. Hemp fibres are 60–70% cellulose, 15–20% hemicelluloses, 2–4% lignin, 2–4% pectin and 1–2% fat and wax (Kymäläinen *et al.*, 2001). The chemical composition of hemp woody core is similar to that of hardwood with approximately 40% cellulose, 20% hemicelluloses and 20% lignin (McDougall *et al.*, 1993; Thomsen *et al.*, 2005).

6.3 Hemp cultivation

The cultivation hemp involves agricultural activities from the planting of the seed in a suitable location through to the harvesting of the crop stems. The production of the fibre is dependent on successful agricultural production, while the production of fibre of a desirable quality for a specific end use depends on understanding the impact of crop husbandry on crop yield and quality.

6.3.1 Hemp breeding and cultivars

The form taken by hemp plants and any fibre is highly dependent on the growing conditions, particularly climate and soil type, and cultivar of hemp grown. Prior to the mid-1930s hemp crops were commonly cultivated from wild forms of the plant (Kirby, 1963). Although there were very few efforts made to genetically breed and select the plant for cropping, the selections were grouped into northern latitude (40–55° latitude) and southern latitude types (0–45° latitude) (Hoffmann, 1961). Northern types tend to require lower temperatures for growth and they are characterised by a short and branched growth habit and produce high seed yield. These types are adapted to the length of summer days in northern and central Europe and will produce seed there. Southern types require higher temperatures and have a tall, unbranched growth habit and mature later with a comparatively low seed yield but high fibre yield.

Modern breeding programmes, particularly in Europe, have developed a number of hemp cultivars for cultivation according to end use: fibre hemp and seed hemp. Simultaneously, varietal development has also produced hemp varieties that contain a low level of the tetrahydrocannibinol (THC) compound, thus allowing their widespread cultivation for either seed or fibre production. In the EU only these low THC cultivars may be grown and are listed in Table 6.2.

Modern fibre hemp cultivars have a tall, unbranched growth habit and a relatively low seed yield (De Meijer, 1995). A significant effort in fibre hemp breeding has developed the fibre content of fibre varieties from 12% to 15% up to 25% to 33% (De Meijer, 1995; Hennink, 1994). However, there can be a significant amount of variation between the performance of cultivars in differing seasons and conditions (Tables 6.3 and 6.4) (Bennet *et al.*,

Table 6.2 List of cultivars approved for cultivation in the EU

Asso	Beniko	Bialobrzeskie
Cannakomp	Carma	Carmagnola
Delta-Ilosa	Delta-405	Dioica 88
Epsilon 68	Fedora 19	Felina 32
Félina 34	Férimon	Fibranova
Fibrimon 24	Fibrimor	Fibrol
Fibroseed	Finola	Futura 77
Kompoliti	Kompolti hibrid TC	Lipko
Lovrin 110	Moniseed	Monoica
Multiseed	Red petiole	Santhica 23
Santhica 27	Santhica 70	Silesia
Silvana	Szarvasi	Tiborszállási
Tygra	Uniko B	Uso-31

Table 6.3 Effect of cultivar and sowing rate on the stem fibre yield and fibre content of hemp (*Cannabis sativa*)

Cultivar	Seeds sown (m^{-2})	Fibre yield (t ha^{-1})	Fibre content (%)
Fedora 19	200	1.43	19.6
	400	1.79	20.8
Felina 34	200	1.34	21.2
	400	1.92	23.1
Uniko B	200	21.0	24.0
	400	2.82	26.2
Futura 77	200	1.79	19.3
	400	2.67	23.6
Kompoliti	200	2.37	27.0
	400	3.48	27.4

Source: Cromack (1998).

Table 6.4 Effect of cultivar and seed sowing density on the stem yield of hemp (*Cannabis sativa*) (t/ha)

	Seeds sown, 1994		Seeds sown, 1995	
Cultivar	200/m^2	400/m^2	200/m^2	400/m^2
Fedora 19	7.1	8.6	10.6	9.4
Felina 34	6.3	8.3	8.7	10.7
Uniko B	8.8	10.8	12.9	9.3
Futura 77	9.3	11.2	12.2	11.5
Kompoliti	9.2	12.7	12.8	11.6

Source: Cromack (1998).

2006; Cromack, 1998), and so cultivar selection may need to be based on pre-production trials at the intended locations.

6.3.2 Hemp crop environment

The first stage in the process of producing hemp fibre is the production of a hemp crop. As with all plant fibres, the environment and manner in which the crop is established and managed has a dominant influence on the quantity and quality of fibre to be found in the hemp stems.

Although it is not possible to give a prescription for hemp crop cultivation in all circumstances, because of the numerous types of hemp and the wide range of soils, climate types and agricultural systems in which the plant may be grown, there are certain aspects of hemp crop husbandry practice that can have a significant influence on the quality of hemp fibre produced and are critical to note if successful cultivation is to be achieved (van der Werf *et al.*, 1996).

Climate and soil

Hemp is well adapted to be grown in many regions of the world, including most temperate, tropical and sub-tropical zones. Hemp crop growth has been found to peak at an average daily temperature of approximately 14°C, but growth occurs over a wide range of 5.6–27.5°C (Duke, 1982). Importantly, the crop is not very frost tolerant. Fibre hemp varieties require 4–5 frost-free months from planting to produce a harvestable crop, while hemp seed types require 5–6 months to produce mature seed.

The availability of suitable moisture (rainfall, irrigation and humidity) throughout the growing season is vital to the production of fibre hemp crops. Hemp plants develop a significant root system that will extend to extract water from approximately 140 cm deep in well-structured soils (soils that allow unrestricted rooting). It has been reported that hemp will achieve a peak yield at soil moisture levels of 80% of soil field capacity (Slovonov and Petinov, 1980). Although hemp will tolerate dry conditions it will not 'thrive' in drought prone soil types (e.g., very light coarse sands) and climates, unless irrigation is employed. Duke (1982) suggests that hemp grows best in areas receiving 970 mm of rainfall per year, while during the growing season maximum fibre yields have been found to occur in crops receiving 535 mm consisting of precipitation (rainfall and irrigation) and soil moisture. In Europe, studies suggest that hemp requires 500–700 mm of available moisture for optimum yield (Bosca and Karus, 1998). Hemp has been successfully cultivated with the use of irrigation (Dempsey, 1975; Lisson and Mendham, 1998), but at present it is mainly cultivated in locations with sufficient rainfall.

Hemp can be grown on a very wide range of soil types, and it is well suited to be grown on soils of many textures and structures. Hemp grows best on well drained, loamy soils with high levels of organic matter (van der Werf, 1991). The crop is not well suited to drought prone soils (e.g., coarse sandy soils), nor is it suited to very heavy soils (e.g., predominantly clay soils) as these soils are often slow to warm up in the spring, prone to waterlogging and they are not always able to accommodate mechanical harvesting, especially in high rainfall years (Vessel and Black, 1947). Hemp can tolerate a soil pH as low as 5.0, but a pH around 6.0 is suggested (Bosca and Karus, 1998; Duke, 1982).

Establishment

The method and timing of crop establishment, at any one location, are significant factors determining the yield and quality of any fibre ultimately produced. The two factors most important to consider are the time of sowing and the density of sowing.

Hemp should be sown once the risk of very hard frosts has passed, making it a late sown spring crop. For example, in Europe this ranges from early April through to early June which would give a harvest date between mid-August and the end of September. A number of studies have investigated the effect of sowing date on fibre yield and quality. Overall the major impact of sowing date is primarily on fibre yield as a result of delayed sowing reducing crop biomass (Table 6.5), rather the size of the fibrous layers in the plants. However, a delay in the sowing of a fibre hemp crop, without an appropriate delay in harvesting date to allow the crop to reach the required stage of maturity, could result in fibres being harvested at an earlier stage of maturity. This will then have a subsequent impact on fibre quality, particularly fibre fineness and fibre strength. In practice, late maturing varieties of hemp should be sown earlier in a planting sequence, while early maturing varieties should be sown later to ensure the required stages of maturity are achieved in the available growing season.

The plant density of a hemp crop, as determined by the seed sowing density, has a significant impact on the properties of the fibres in the plants. Seeding rates for hemp crops can be found to vary widely from 50 to 500 seeds/m^2 in older recommendations (Dempsey, 1975), but recent recommendations range from 150 to 400 seeds/m^2 (Amaducci *et al.*, 2008; van der Werf *et al.*, 1995). Tables 6.3 and 6.4 illustrate the interaction of cultivar and seed rate on the stem yield of fibre hemp in two growing seasons. Overall a high plant density produces a better crop with increased early growth competition with weeds, and high plant densities will produce crops with a higher proportion of stems and lower proportions of leaves and seeds. However, higher densities do not increase crop biomass beyond a maximum. A higher plant density will also decrease the diameter of stems and increase the percentage of fibre and reduce the percentage woody core (van der Werf, 1991).

Seed should be drilled at a depth of 2.5 cm in rows spaced 12–50 cm apart depending on requirements (van der Werf *et al.*, 1995).

Table 6.5 The effect of date of sowing on hemp stem yield (t/ha) at 15% moisture content in two growing seasons in Ireland (1998 and 1999)

Sowing date	Season 1	Season 2
Late March	15.3	12.5
Mid-April	11.2	10.9
Early-May	10.5	8.3
Mid-May	8.4	6.7
Standard error	1.12	0.99

Source: Crowley (2001).

Husbandry

Hemp is a fast growing crop and the provision of adequate supplies of plant nutrients is very important. It has been estimated that to produce a tonne of hemp material 15–20 kg of nitrogen, 4–5 kg of phosphorus and 15–20 kg potash are required (Bócsa *et al.*, 2000). The phosphorus and potassium may be available to the crop from adequate reserves in the soil or it may be applied at or prior to planting. The application of nitrogen fertiliser is an important input into hemp crops as it is needed to produce an economic crop yield and for stem uniformity, with the latter important for accommodating efficient crop post harvest processing. However, high levels of nitrogen fertiliser (200 kg N per ha) can significantly increase stem variability in comparison to moderate levels (100 kg N per ha). It can also increase stem diameter above a desired range (Ranalli, 1999) or lead to self-thinning and a reduction in the number of live plants (van der Werf *et al.*, 1995). In Europe nitrogen fertiliser rates typically range from 80 to 150 kg N per ha.

An attractive feature of hemp is that its fast growth habit means that it can compete with weeds with little or no pesticide use (Dempsey, 1975; van der Werf, 1991). However, if the crop establishes slowly, perhaps because it is planted too early, hemp stands may suffer from weed ingress.

Hemp production does not at present incur many agriculturally significant pests or diseases, despite a number of diseases and over 300 insect pests being known to affect the crop (McPartland, 1996a, 1996b; McPartland *et al.*, 2000). The most important disease of hemp is grey mould (*Botrytis cinerea*), which attacks crops under conditions of cool temperatures and high humidity and has been observed in the UK (Cromack, 1998). In practice the most significant pest of hemp crops is birds feeding on seeds and grazing birds such as pigeons attacking seedlings. However, pest problems are not widespread. Although the crop is generally free of pests at present it may be that if the crop is more widely cultivated then pest and disease problems will become more widespread.

Harvesting and cutting

When hemp is grown for the production of long fibres, similar to long flax fibre, the crop is harvested when the staminate plants have finished flowering but before the seed has ripened, also known as technical maturity (van der Werf, 1991). After the stems have been cut they are laid on the ground in windrows for field retting, and to maximise the recovery of long fibres during stem breaking and fibre breaking the hemp stems are maintained in parallel alignment (Bruce *et al.*, 2005). Such a system is very similar to the process used to harvest flax for linen production, except that the plants are cut rather than pulled. Depending on the location and planting date, the

6.2 Cut hemp field retting in rows.

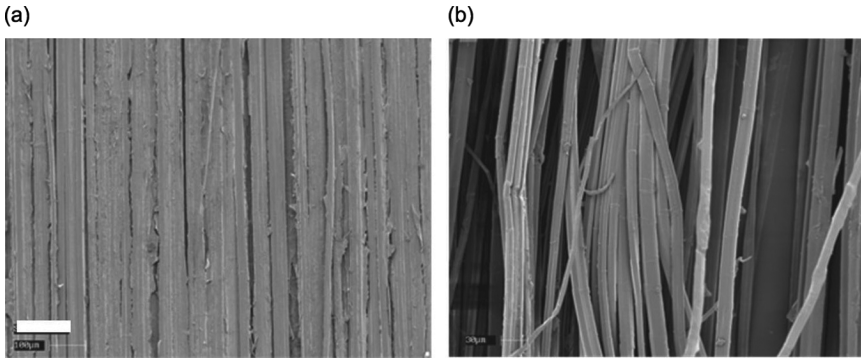
hemp crop harvest normally occurs in the late summer to early autumn, for example in August and September in northern Europe.

Alternatively, hemp can be cut with a disc or reciprocating knife-type mower (Bednar, 2008) and then left on the field to ret in rows (Fig. 6.2). The crop can then be turned to ensure the whole stems are retted and dry evenly (<18% moisture content) and then baled as a hay crop. This method of harvesting has the advantage of being very efficient and does not require specialist machinery (Chen *et al.*, 2004). However, the method is not suited to crops grown for long fibres as the orientation of the stems is not maintained, but the system is suited to the production of short fibres for industrial end uses or textiles.

The fibre yield of a hemp crop will continue to increase until the plant reaches full maturity and senesces (van der Werf, 1991). However, as the plants proceed towards seed maturity the fibres in the stems become coarser and weaker, and greater levels of lignin are deposited. Increasing lignification has been suggested to potentially reduce the yield of fibre mechanically recoverable from the hemp straw (Struik *et al.*, 2000). For end uses that do not require well retted and separated fibres these mature fibres may be adequate, but where fine fibres are required the crop may need cutting at an earlier development stage.

6.4 Retting

The quality and quantity of hemp fibres yielded from a crop is very strongly determined by the degree of retting that the fibre has undergone. The retting process involves the removal and breaking down of the 'gummy'



6.3 Electronmicrographs of longitudinal section of hemp bast layer: (a) unretted hemp fibres and (b) retted hemp fibres. (Source: Used with permission from De Montfort University.)

substances, particularly pectins, which bind the fibres together in bundles (Fig. 6.3) and to the plant core. The retting process can be undertaken chemically or biologically. In the case of the latter retting is achieved with the action of colonising enzymes or the action of specialised enzymes secreted from colonising fungi and bacteria. The definition of retting is '*the subjection of crop or de-seeded straws to chemical or biological treatment to make the fibre bundles more easily separable from the woody part of the stem*' (Farnfield and Alvey, 1975).

Retting is of critical importance to the extraction and separation of fibres from the core of hemp stems, as it significantly affects the yield and quality of fibre achieved after the subsequent processing of the straw. In practice hemp stems may be retted in several methods:

- field, dew retted,
- water retted,
- enzyme retted,
- chemical retted.

The chosen method will primarily be determined by location and the requirements of the target end use.

6.4.1 Field retting

After the crop has been cut the stems are left spread over the surface of the field for 2–8 weeks (Fig. 6.2), depending on the location, the degree of retting required and any consideration for subsequent land use such as the planting of the next crop. During this period of time the spread stems are normally turned a number of occasions to evenly expose the stems to light,

weather and temperature conditions. Importantly with this method of retting, there can be a significant year to year variation in the duration of time required to achieve satisfactory retting as a result of varying weather conditions. The retting of hemp stems in the field is achieved by the application of moisture (rain and dew), warm temperatures, high relative humidity and still air in diurnal episodes. The stems are judged to be sufficiently well retted by examining the colour of the stems for an even dark grey colour throughout and/or by manual handling to examine the ease and cleanliness with which the woody core and fibres separate.

During the field retting process microorganisms attack the hemp stems and break down the cementations that bind together the ultimate fibres in bundles. The microorganisms will secrete enzymes that will degrade pectins from the outer middle lamella lignified inner middle lamella; proteins from the protoplasm of plant cells, starches, fats and waxes, and tannins. It is the most widely practised method of retting hemp and it is the source of most hemp fibre produced in Europe and the UK. Critical to the success of field retting is the presence of satisfactory temperatures and moisture, and so it can only be conducted in suitable geographical areas.

A major advantage of field retting is its agricultural efficiency and low cost, as the technique does not require the crop to be moved from the field in which it was grown, and it can be fully mechanised to minimise labour input. Additionally, the technique is low input in comparison to water and chemical retting as it requires nothing more than the mechanical field operations.

The major disadvantages of field retting hemp arise from a comparative lack of control on the process by the hemp grower. The retting can be variable both within and between crops as a result of fluctuating weather and climate patterns, and crop variation. Subsequently the fibre extracted is often variable in fineness, length, strength and colour within a sample in comparison to chemical and water retted hemp fibre. Other drawbacks of this method include the occupation of land until the retting is complete, which may impact on the planting of a subsequent crop, and the fact that hemp fibre extracted from dew retted crops tends to be dusty such that it can pose a health risk to operators during subsequent processing.

6.4.2 Water retting

Water retting often produces the best separated and therefore finest hemp fibres, in a short period of time (Table 6.6). The traditional method of water retting hemp involved steeping the stems in running water by submersion in ditches, streams or rivers but this has largely been replaced by the method of water retting in tanks through which water flows (Di Candilo *et al.*, 2000, 2009). Water retting tanks can be sealed or open depending on the climate, and may be arranged in a series or cascade layout. In the cascade design a

Table 6.6 Chemical composition of water retted hemp fibre at different times with and without the addition of bacterial inoculum

	Treatment time (h)	Cellulose (%)	Pentosans (%)	Soluble lignin (%)	Insoluable lignin (%)	Extractives (%)	Ash (%)
Untreated		82.0	8.4	2.2	0.7	4.4	2.3
Water retted without bacterial inoculum	1	85.4	6.3	2.2	0.7	3.2	2.2
	2	86.4	5.8	2.0	0.7	3.2	1.9
	3	86.4	5.2	3.0	0.6	3.2	1.6
	4	86.6	5.0	3.2	0.6	3.2	1.4
	5	87.4	4.7	3.2	0.6	3.1	1.0
	6	87.3	4.5	3.7	0.4	3.1	1.0
Water retted with bacterial inoculum	1	85.5	6.5	1.5	0.7	3.2	2.6
	2	87.0	5.5	0.6	0.6	3.2	2.0
	3	88.6	4.8	2.1	0.6	3.2	0.7
	4	88.6	4.6	2.4	0.6	3.1	0.7
	5	88.4	4.4	2.9	0.5	3.1	0.7
	6	88.9	4.1	3.1	0.5	2.8	0.6

Source: Di Canandilo *et al.* (2009).

series of three to four interconnected tanks will have water passed through at a rate that will achieve one change of water in the system every 2 days. The use of cold water in tank-based water retting will result in the retting process taking only 7–14 days to complete, but this can be reduced further by heating the tanks to between 30°C and 40°C. It is not uncommon in all methods of water retting to find the inclusion of chemical additives to enhance the process and offset some of the environmental impacts of the process. The latter water retting has been widely practised in Europe and can still be found conducted in some countries.

The primary advantage of this method of retting hemp is that it can be controlled and evenly applied to hemp stems, subsequently producing a superior quality of fibre in comparison to field retting. Additionally, it is not dependent on weather conditions, so enabling fibre hemp to be grown in locations that may not possess a suitable climate for field retting, and in its basic form it is a relatively simple technology that can be used in locations without access to chemicals. Furthermore the process is relatively quick in comparison to field retting (Table 6.6) and ensures the field is available for subsequent cropping more rapidly after cutting.

On the other hand, the process suffers from a very high level of water consumption per ton of straw and produces a significant amount effluent as a waste-product. Water retting is also very malodorous. Additionally, it is necessary to dry the fibre after the water retting has been completed which can incur significant energy costs.

In order to combat some of the problems associated with high levels of water consumption and effluent, aerobic bacilli are added to the retting liquor and the tanks are aerated. This gives slightly alkaline liquor, lowers the concentrations of volatile acids and reduces the odour given off during retting. In practice it also enables the water to be recycled through the water retting system indefinitely and the bacilli do not attack or compromise the fibre. Despite water retting producing some of the best quality hemp fibre and the advances made with the technique to reduce the potential environmental impact of effluent, the use of water retting in Europe has largely been terminated as a result of environmental concerns and the high cost of artificially drying the fibre after retting.

6.4.3 Chemical retting

Chemical retting methods have been described and used for retting hemp stems (Kostic *et al.* 2008; Wang *et al.*, 2003), and are similar to those used for chemical treatment of flax. Chemical retting is commonly performed using a sodium hydroxide (NaOH) solution, with or without the use of chelating agents such as ethylenediaminetetraacetic acid (EDTA). Other chemicals, such as sodium carbonate (Na_2CO_3) or sodium sulphite (Na_2SO_3), can also

be used in retting solutions. Many of these chemical mixtures are similar to those used for degumming other plant fibres, such as ramie. The main factors determining the effectiveness of chemical retting solutions include:

- chemical concentration,
- solution acidity (pH),
- stem to liquor ratio,
- temperature,
- duration of treatment,
- agitation degree.

This method posses all the advantages of water retting over field retting, but the retting process can be further accelerated from the few days required for water retting to just a few hours.

Although the results achieved from chemical retting of hemp have generally been very good, in terms of fibre quality and fibre yield, the method is generally cost prohibitive for many end uses and is not widely commercially viable. The results of chemical retting are generally good (Table 6.7).

6.4.4 Enzyme retting

A significant amount of research has been focused on retting hemp with enzymes by attempting to replace the bacteria which facilitate fermentation in water retting with enzymes in tanks. The objective of this technique is to ultimately produce a fibre quality equivalent to that achieved with water retting but without the problems of effluent waste and malodour.

A number of enzyme mixtures have been tested at the laboratory level and found successful at retting hemp fibres, although variations in performance have been noted (Sultana, 1992). With enzyme retting no field retting is required and the time for land occupation by the hemp crop is shortened to that of growing the crop without interference to subsequent cropping. Additionally the method is attractive because it allows retting to take place all year around and eliminates the risks associated with field retting.

Table 6.7 Chemical composition and weight loss of hemp fibre treated with NaOH at room temperature (RT) or boiling temperature (B)

		Cellulose (%)	Hemicellulose (%)	Lignin (%)	Weight loss (%)
Untreated		76.12	12.28	5.65	
5% NaOH	RT	83.63	7.49	2.61	4.4
5% NaOH	B	89.16	1.93	2.07	25.5
18% NaOH	RT	86.18	3.83	2.44	14.1

Source: Kostic *et al.* (2008).

The major disadvantage of this method is the potential high cost of retting, as a result of the cost of the enzymes and the equipment needed.

6.5 Fibre extraction

The extraction of fibres from hemp stems is commonly achieved by the mechanical processing of the straw, although historically manual decortication has been widely practised. The purpose of this processing stage is to completely extract the fibres by entirely separating them from the woody core of the hemp stem, which is often referred to as 'hurd'. Prior to processing, after retting, it is necessary that the straw be dried to a suitable moisture content to stop the retting process and to enable the straw to be stored without deterioration. This drying process enables the fibre bundles in the stem to contract and become partially loosened away from the woody core.

Although the objective of extracting, separating and cleaning the fibres from the other parts of the hemp plant is universal in hemp fibre production, the exact method of fibre extraction employed will ultimately depend on the necessary condition of fibre (length, fineness, etc.) required for the end market and users. Conventionally, hemp fibres were extracted in a long fibre (50–60 cm) form, with a significant quantity of by-product short fibre, using breaking and scutching methods similar to those used for the production of long flax fibre (Fig. 6.3). However, as new markets have developed for hemp fibres, particularly in industrial products, decortication methods of fibre extraction that aim to produce a single quality of hemp fibre have been developed (Tables 6.6 and 6.7).

6.5.1 Breaking and scutching

The first stage of mechanical processing is the breaking process whereby the straw is mechanically treated to break and loosen the woody core from the fibres. Typically the stems are fed into a series of fluted and smooth rollers, either horizontally or vertically arranged, which crush and break the straw along its length. The smooth rollers crush and break the stems along their length, while the fluted rollers break the woody core into small pieces which then drop out of the breaking unit. The fluted rollers are typically arranged in a series with declining pitches so as to break the woody core into increasingly smaller pieces.

Overall, the effectiveness of the breaking stage is almost entirely dependent on the degree of retting of the hemp straw. Well-retted hemp stems can be broken with ease and the content of hurd remaining at the end of the process is significantly reduced, to less than 40%, and the fibre is suited to further mechanical processing that will yield fibres suitable for spinning for

textiles. The processing of unretted material results in a comparatively less successful removal of hurd from the straw and the final fibre is only really suitable for use in paper manufacture.

The second stage of mechanical processing is the scutching stage, which has the objective of completely removing all the remaining hurd adhering to the plant fibres, which was loosened by previous retting and mechanical breaking. The process also has the effect of opening the fibre, making it finer and softened. The fibre is scutched by being beaten between turbine blades, bladed wheels operating against a curved plate. The broken fibre is held vertically in a continuous belt that passes the fibres between a pair of turbines, rotating in different directions. The turbines are operated in two pairs so that after passing through the first pair of turbine blades the fibres are reversed to pass through the second pair facing in the opposite direction. The primary output of this process is the long (line) fibres, but during the process some fibres are broken and contained in the scutching waste. These fibres are extracted from this waste and cleaned as short (tow) fibres for use in textile products, coarse yarns or ropes. The greater the degree of retting the straw the lower is the yield of tow fibre of total processed fibre.

After scutching, the tow fibres that have been removed during the process are separated from the hurd and other impurities (dust, leaf material, etc.) by passing them through some form of cleaning unit. The tow fibres may then be further processed to open the fibres in preparation for the anticipated end use. They may also be dried to the required moisture content if needed. The key factors of hemp tow fibre production are fibre processing efficiency, and fibre qualities such as fineness, colour and fibre length.

6.5.2 Decortication and fibre cleaning

Decortication

Although the use of breaking and scutching lines has been widely employed for the production of hemp fibre, particularly for the production of high-quality textile fibres, alternative methods of extracting hemp fibres and subsequent cleaning have now been developed. The objectives of decorticating the hemp straw, and subsequent opening and cleaning of the extracted fibre, are the same with these alternative methods as for traditional breaking and scutching, but the actual mechanical methods employed are different. In particular the fibres produced from these processing techniques are significantly different and make the fibres much less suited to the manufacture textiles but are well suited to the production of technical fibres. A significant difference between the scutching and hackling methods and the alternative decortication methods is that the harvested stems in the latter do not have to be kept in a single orientation, with all the stem bases kept together and all stems parallel, to ensure

the effective extraction of the long fibres. Instead the stems can be harvested and the stems allowed to randomly orientate in the harvested bales.

The first stage in these processing methods is the opening of the bales of hemp straw and their feeding into the decortication machine. During this first stage of the processing the hemp straw can be cut with a guillotine or bale cutter into shortened lengths (400–500 mm) to ensure efficient straw feeding and uniform subsequent processing. The opened and cut straw is then metered into the decortication equipment with either a box feeder or metering box device, or the straw can be transferred in an air flow to a decorticator.

As with breaking and scutching the process of decortication has the objective of separating the outer layer of fibres of the hemp stems from the woody inner core, while also applying some opening actions to the bundles of fibres. The principal methods used to conduct a decortication operation are swing-hammer mills, with and without cleaning, and roller decortication. Hammer mills consist of a cylinder containing a rotor equipped with hinged beaters (hammers) that rotate at high speeds and decorticate the hemp straw, fed vertically into the top of the cylinder, by working it against the internal surface of the cylinder casing. Decorticated material passes down the cylinder pneumatically in an axial direction. Some hammer mill decorticators are equipped with a screen on the bottom surface of the cylinder to allow woody core extracted from the hemp straw to be separated from the decorticated fibre. The major advantage of these decorticators is that they operate at a very high throughput – 6–8 t straw per h – and can decorticate both unretted and well-retted hemp straw. When cleaning is introduced into the hammer mill it is possible to achieve high levels of cleanliness and up to 60% of hurds can be discharged at this stage, making subsequent cleaning significantly easier. However, the major disadvantage of this processing technique is that fibre decortication is achieved by the impact of the hammers acting on the surface of the straw. Given that the fibres are contained in an outer layer on the straw this has the potential to cause mechanical damage to the fibres, which could result in inconsistency in the extracted fibre and limit its use to nonwoven products and industrial textiles, rather than fine short fibre hemp-based yarns.

An alternative to hammer mill based decortication is the use of roller decorticators (Fig. 6.4). Using this method opened hemp straw is fed through a series of fluted crimping rollers that will break and fracture the inner woody core and separate the fibre bundles. The rollers are operated in a horizontal series of 7–10 rollers, and the pitch of the rollers is reduced from one roller pair to the next to allow for the reduction in the width of the straw flow. Hurds fall out below the rollers as the straw passes through the series. The advantage of this system is that the fibres are not damaged by the decortication process and it is possible to extract long fibres after this method, but the proceeding fibre cleaning operation will have significantly more cleaning to conduct as fewer hurds are removed during this process.

6.4 Roller decorticator behind guard.

The major drawback of this system of decortication is that it generally only accommodates a low throughput of straw: no more than 1 t/h. Also, roller decorticators are less able to decorticate unretted straw and they are less effective as the straw moisture content exceeds 15%.

Fibre cleaning

After the straw has been decorticated, the resulting mass will be a combination of fibre and broken hurds, and the next stage(s) must separate these two component parts. The type and amount of cleaning required will depend on the cleanliness of the material after decortication and the cleanliness levels required by the end users of the hemp fibre. After hammer mill decortication, with a cleaning screen, in its cylinder the level of hurd in the fibrous mass is likely to be lower than with roller decortications. Consequently, the output of the latter will require more cleaning than the former. Several types of cleaner can be employed in various combinations and sequences to achieve desired fibre cleanliness, but in general fibre cleaners operate on the principle of either a tambour turbine, step cleaner or comb shaker table.

Picker

A picker is a hard faced cylinder covered in pins or beater blades that rotates to strike a downward motion against fibres fed into the beating cycle by feed rollers, typically via a feed belt (Fig. 6.5). As the fibres are fed into the machine they first become arched by the rotating action of the cylinder and the fibres

6.5 Picker cleaner.

are then conveyed between the cylinder and a bar grid screen at the base of the machine. The hurd and non-fibrous particles drop through the grid while the fibre adheres to the cylinder. The main advantage of these machines is that they are able to process quantities of fibre in excess of 1 tonne/h.

Step cleaner

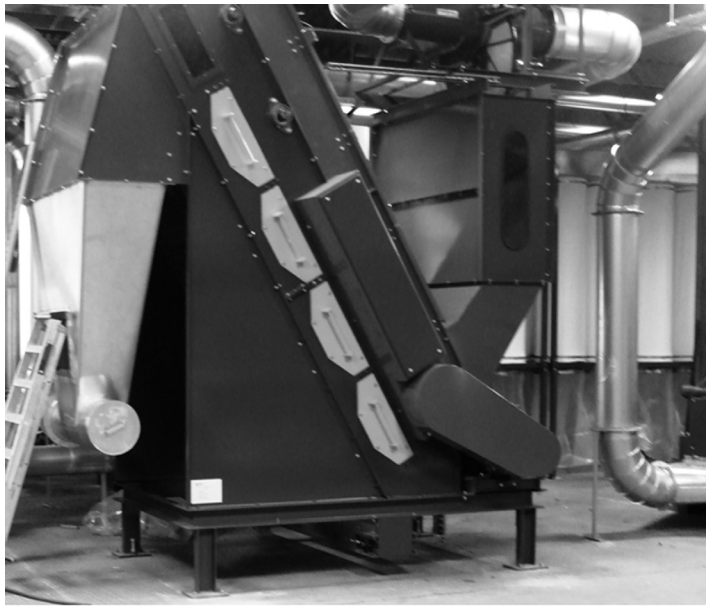
A step cleaner is called so because of the arrangement of its beater cleaner components. Up to six drums are positioned in an ascending manner like stairs, with bar or wire grids beneath each drum. These drums are hard faced but have beater arms which loosen the fibres fed into the lowest end of the machine (Fig. 6.6). As the drum rotates the fibre is passed from one beater to the next by the air flow generated by the rotating drums, while the loosening action of the drum on the fibre results in an excellent separation of the fibre from the hurds, which fall through the grids beneath the drums. Commonly the bar or wire grids have adjustable gaps widths to accommodate different particle sizes.

Step cleaners are highly efficient and are normally used for the production of fine fibres, and they can process fibres at a high throughput of up to 5 tonne/h.

Shaker table

A shaker table is a simple cleaner unit that is frequently used for the final cleaning stage of fibre. The comb shaker constitutes a grid positioned at a

(a)



(b)

6.6 Step cleaner. Side view (a) and rear view showing rollers and beaters (b).

slight gradient with rows of oscillating teeth over the top of them, to operate as a comb reciprocating the fibre up the gradient. The hurd is loosened from the fibre and falls through the grid bars. Although relatively effective as a cleaner the major disadvantage of this method of cleaning is the slow throughput of the unit.

Fibre opening

The objective of fibre opening is to separate the bundles of fibre into smaller aggregations of fibre or into single fibres. The purpose is to increase the fineness of the fibre material, while simultaneously removing any non-fibrous material, particularly dust and small pieces of hurd.

Opener

A fibre opener consists of one or more opening cylinders into which fibre is fed by feed rollers. The fibre opening cylinder is 'clothed' with needles, pins or garniture with different pitches. As the fibre is fed in the rotating cylinder the clothing pull the fibre over several carding plates to separate, parallelise and comb the fibres. Finally, the fibres are sucked off pneumatically. The process will primarily separate the fibres and increase the fineness of the fibre. A major advantage of this type of opener is that it is relatively low cost in comparison to carding and it can be a flexible process that can utilise a number of needle, pin or wire arrangements to produce fibre of a very specific fineness.

Carding

Using a card to process hemp fibre extracted using decortication rather than breaking and scutching of straw is identical to that used to open short fibre hemp tow. However, the production of hemp fibre for technical rather than textile end uses is a lower value market and the requirements for finer fibre less. As carding is an expensive process it is less suited to opening this hemp fibre.

6.6 Hemp fibre spinning

Conventionally there have been two principal types of hemp fibre – long line fibres and shorter tow fibres – produced during the extraction of the fibres from the stems and which can be spun into yarns. The long line fibre is usually spun to produce a relatively coarse yarn, while tow fibre hemp may be used to produce finer yarns and it is often blended with other fibres prior to spinning.

6.6.1 Long fibre spinning

Preparation of long fibre hemp

Prior to the hackling process it is common to find hemp fibres are processed to increase the fineness of the bundles of fibre produced during scutching. A softening of hemp fibres can be achieved with the use of ribbed rolls that will soften the hemp fibres as they move between the rolls. Further softening may be achieved during this stage with the application of a softening emulsion to the hemp fibres prior to treatment.

A further preparatory stage of processing the scutched fibres is also the cutting of the fibres to a standard length in preparation for processing on the hackling frames. The process also removes the unevenness often found at the tangled tips and roots of the scutched hemp.

Hackling

After the preparation of the scutched long fibres is complete, the next stage in the processing of long fibre hemp is hackling, a combing operation. This process is conducted to disentangle and straighten the fibres, separate and then split up the fibre bundles without significantly reducing the length of the long fibres. The process also has the objective of cleaning the fibres to remove any remaining hurd, dust and short fibres and tow.

The process is conducted with the use of a hackling frame. In this process two pieces or 'hands' of scutched hemp are clamped in a holder that moves the fibre along a channel, between vertically moving and opposing sets of pins of increasing fineness. This use of increasing pin fineness combs the coarse fibre bundles into separate finer bundles. As the fibre is moved around and between hackling frames it is turned over so that fibres are worked uniformly through the frame.

During the hackling breakage of some long fibres occurs. This hackled tow is collected and subsequently used in tow short fibre yarns.

After fibres have been processed through the hackling frame the fibres are assembled into a sliver. This is done on a machine that is a modified drawing frame. Each frame consists of four horizontal belts separated from each other. Hackled line fibres are converted into a continuous sliver by laying them end-over-end.

Drawing and doubling

The operation of drawing and doubling has the purpose of separating the bundles of fibres in the slivers and to regularise the slivers, and it is the final stage of processing prior to spinning a yarn. Five consecutive drawing and doubling frames would be typical, with the last stage introducing an element of twist into the process. Critical to the production of a uniform

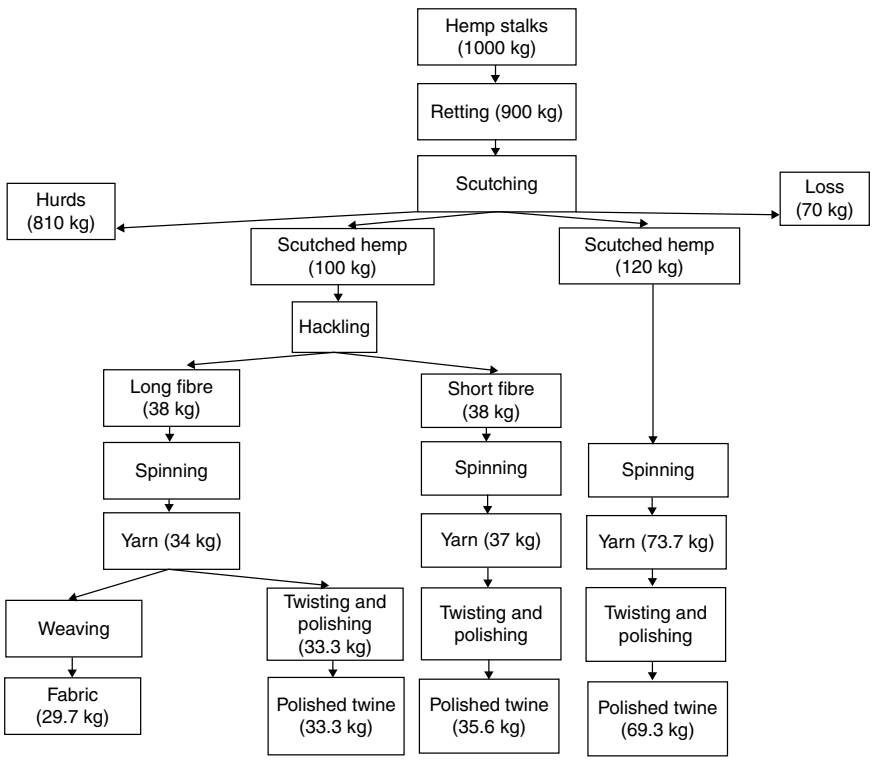
rove for spinning is the selection and combining of slivers of an even length and weight for blending during the drawing stages. Long fibre hemp rove weights are typically 2.5–5 g/m.

Spinning

Long fibre hemp rove maybe spun using either a wet spinning or a dry spinning technique. Wet spinning is significantly more costly and now dry spinning is the primary method of spinning hemp. The majority of hemp yarns are produced for weaving and consist of yarns of a count weight of 3.5–5 Nm. After spinning hemp yarn is wound on cone winders. A flow diagram of the processing of fibre hemp is shown in Fig. 6.7.

6.6.2 Short fibre spinning

During the scutching and hackling operations a certain amount of short fibre is removed as part of the extraction and preparation of the long fibres.



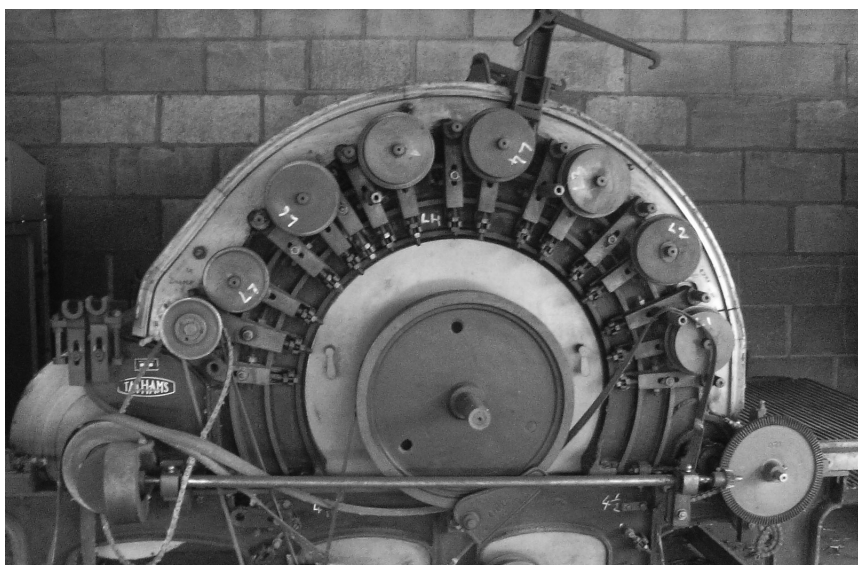
6.7 Long fibre hemp production flow chart and yields. (Source: Sponner *et al.,* 2004.)

This fibre typically falls into one of two types: scutched tow or hackled tow. This fibre can be processed in preparation for spinning into a wide range of yarns, depending on the quality of the hemp being processed and uniformity of fineness, length and cleanliness. However, unlike long fibres hemp tow fibre tends to be processed and spun on more conventional fibre preparation and spinning equipment.

Carding

The first processing stage for short fibre hemp prior to spinning is carding, which may be completed in a single or multiple phases. Overall, the objective of the carding process is to break down the long fibres into ultimate or split compound fibres, untangle and open lumps of fibres, remove any remaining dirt and very short fibres and form a carded sliver. The process consists of the fundamental processes of 'working', 'stripping' and 'doffing' and a considerable degree of fibre opening and compound fibre breakdown can be achieved with retted hemp fibre on a breaker or finisher card (Fig. 6.8). In addition, the carding process will parallelise fibres, as a result of the directional bias of the carding machine, and it provides an opportunity for blending different qualities of hemp or hemp with other fibres.

The carding process of hemp can include a first carding stage that is conducted on a 'coarse' card, with large cylinders and rolls and stronger card wires than the main stage. This process will clean out the remaining foreign matter to be found in the fibre, cards the fibre and forms a sliver. A



6.8 Single stage breaker card machine – open side view.

complementary second stage card (finisher card) then unwinds the slivers prior to being fed into a card condenser and main cylinder that disentangles the fibres further. The fibre is then formed into a sliver at the drawhead prior to doffing. If hemp fibres are to be blended together or hemp fibres blended with other fibres, this can be done at entry into this final card where different coils can be fed into the card at the required ratio. The subsequent carding and drawing and doubling processes then blend the fibre further. Alternatively, hemp fibres may be carded in a single stage card and any required hemp fibres blended prior to and/or during carding without the formation of an intermediate sliver.

Combing, drawing and doubling

Depending on the intended quality of yarn anticipated the final sliver may undergo a combing process after carding but prior to subsequent preparation. Combing will further parallelise the fibres and remove any remaining impurities and short fibres. Combing is only necessary for the production of finer yarn counts, while coarse and medium count yarns can be spun directly from the finisher sliver on spinning frames. Any slivers intended to be combed after carding are normally passed through a draw frame first to limit the losses during the combing operation. After combing the slivers will undergo a further three draw frame passes prior to being doubled and then spun. Drawing attenuates the sliver to decrease the mass of fibre per unit length of the sliver. Drawing frames similar to those employed for the processing of line fibres can be used, but with a shorter drafting reach, although the use of gill drawing frames can be used as an alternative for producing slivers for coarser yarns. Intersecting gill frames are less likely to be used with hemp processing than flax due to the cost involved. After the drawing operation 'doubling' superimposes several slivers, processed together, to improve sliver regularity.

Spinning

Hemp fibres are most commonly dry spun, but alternative methods are also used.

Dry spinning

Short-fibre hemp prepared for spinning as a sliver is typically spun using a dry spinning method, rather than wet spun, on frames similar to those used for spinning long fibre hemp but with a shorter drafting zone. As a result of the fibres still existing in groups, although sub-divided to some extent, and as they are drafted together there is a limit to the yarn fineness achievable of approximately 65 tex using this method. The final yarns tend to be soft and open structured.

Alternative spinning methods

Spinning methods used in the production of long-staple fibres, flax and hemp are relatively specialised activities and have not received the attention of developments in innovation received by more widespread short-staple systems. The majority of recent developments in spinning systems have been directed towards the likes of warp spinning, ring spinning, open-end spinning and friction spinning and the use of cotton and short staple man-made fibres. However, there has been some development of the production of short-staple hemp fibre of a length suitable to be processed either alone or in blends with other short staple fibres.

Over the last 10 years there has been an increasing interest in the use of hemp fibre in blends with cotton and man-made fibres in fabrics, woven and knitted, mainly driven by the attractive potentially low environmental impact of hemp fibre production. In dry spinning, hemp can be blended with cotton and most types of man-made systems as well as wool. In the cotton spinning system flax fibres should have length of 20–60 mm, while in wool spinning systems hemp fibres should be between 60 and 120 mm. In general, the potential number of options for spinning hemp fibres on short-staple systems with other fibres, and in what quantities, is very high. For the production of yarns for use in apparel fabrics the most important blend is hemp fibre with cotton and polyester, at percentages from 10% to 60%. Its blending with viscose is also important. Blends of hemp with wool are easier to achieve and can produce coarse yarns of a relatively high composition, up to 90%.

Winding

Immediately after being spun yarns are transferred to large containers, typically cones. This process is known as winding and is usually completed at high speed to produce a package of uniform density without disturbing the structure of the yarn. Although the process is not productive itself, winding provides an opportunity for quality control and the removal of faults. Fine hemp yarns, of a count between Nm 1 and Nm 6, spun from long hemp fibres and from short fibres of a count of Nm 1 to Nm 15 are normally wound onto comb winders. Thicker hemp yarns, of a count between 0.20 and 1 Nm, are wound onto cross winders of a significant weight, up to 10 kg.

Once packaged the yarn is then ready for the preparatory treatments of warping and warp sizing, assuming that the yarn is intended for weaving.

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7

Silk fibres

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Abstract: This chapter deals with one of the important natural fibres: silk. The chapter focuses on silkworm rearing, cocoon production and different types of silk. The properties of silk fibres such as tensile behaviour, optical properties, visco-elastic behaviour and thermal properties are discussed in detail. In addition, the structure of silk fibre (for example crystal structure and morphology) is discussed. A brief introduction is presented on the current status of the silk industry and its future growth. Silk reeling operations, types of reeling machines, silk throwing and fabric production details are also presented. Being a natural fibre, silk has number of applications in different fields and these applications are discussed in detail. The concluding part of the chapter discusses future trends in silk production with respect to production in various countries, and new avenues of applications of silk are discussed to inform the reader about the latest trends in silk production.

Key words: silk fibre, mulberry silk, non-mulberry silk, silk properties, applications of silk.

7.1 Introduction

Silk is one of the oldest fibres known to man. Silk is an animal fibre produced by certain insects to build their cocoons and webs. Although many insects produce silk, only the filament produced by the mulberry silk moth, *Bombyx mori*, and a few others in the same genus, is used by the commercial silk industry (Jolly *et al*., 1979). The silk produced by other insects, mainly spiders, is used in a small number of other commercial capacities, for example weapon and telescope cross-hairs and other optical instruments (Spring and Hudson, 2002).

Over the centuries, silk has been a highly valued textile fibre. Its qualities of strength, elasticity, softness, absorbency, affinity for dyes and adaptability to various forms of twisting continue to satisfy various market demands. Despite facing keen competition from man-made fibres, silk has maintained its supremacy in the production of luxury apparel and specialized goods of the highest quality (Robson, 1998). Silk has been used as a textile fibre for over 4000 years. Because of its high (tensile) strength, lustre and ability to bind chemical dyes, silk is still considered a premier textile material in the world today (Zarkoob *et al*., 2000).

Silk fibres from silkworms have been used in textiles for nearly 5000 years. The primary reasons for this long-term success are the unique lustre,

tactile properties, durability and dyeability of silks. Silk fibres are remarkable materials displaying unusual mechanical properties: strong, extensible, and mechanically compressible (Matsumoto *et al*., 2006). Silk is rightly called the queen of textiles for its lustre, sensuousness and glamour (Reddy, 2009). Silk's natural beauty and properties of comfort in warm weather and warmth during colder months have made it useful in high-fashion clothing. Silk fibres have outstanding natural properties which rival the most advanced synthetic polymers, yet the production of silk does not require harsh processing conditions and hence, widespread investigations are being undertaken even for artificial synthesis of silk fibres (Chen, *et al*., 2003).

7.2 Silk industry

Silk production, including sericulture, is well known as a highly employment-oriented and low capital intensity activity ideally suited to the conditions of a labour-abundant, agro-based economy. Sericulture is both an art and science in terms of raising silkworms for silk production. Silk as a weavable fibre was first discovered by the Chinese empress Xi Ling Shi around 2640 BC and its culture and weaving were guarded secrets for more than 2,500 years. Silk was a profitable trade commodity in China. Traders from ancient Persia (now Iran) used to bring richly coloured and fine textured silks from Chinese merchants through hazardous routes interspersed with dangerous mountainous terrains, difficult passes, dry deserts and thick forests. China is the largest producer of silk and the biggest consumer of raw silk and silk fabrics in the world and India is the second largest producer of raw silk after China (Datta and Nanavaty, 2005). An analysis of trends in international silk production suggests that sericulture has better prospects for growth in the developing countries rather than in the advanced countries. Silk production in temperate countries like Japan, South Korea, Russian Federation, etc., is declining steadily not only because of the high cost of labour and heavy industrialization in these countries, but also due to climatic restrictions imposed on mulberry leaf availability that allows only two cocoon crops per annum (Gangopadhyay, 2008). Although silk is produced in more than 20 countries, the major producers are in Asia and sericulture industries have also been established in Brazil, Bulgaria, Egypt and Madagascar for their labour-intensive advantage.

7.2.1 Silkworm rearing and cocoon production

Sericulture is the rearing of silkworms for the production of raw silk. The major activities of sericulture comprise food-plant cultivation to feed the silkworms which spin silk cocoons and reeling the cocoons for unwinding

the silk filament for value-added benefits such as processing and weaving. Although there are several commercial species of silkworms, *Bombyx mori* is the most widely used. Sericulture is ideally suited for improving the rural economy of the country, as it is practised as a subsidiary industry to agriculture. Recent research has also shown that sericulture can be developed as a highly rewarding agro-industry.

7.2.2 Types of silk

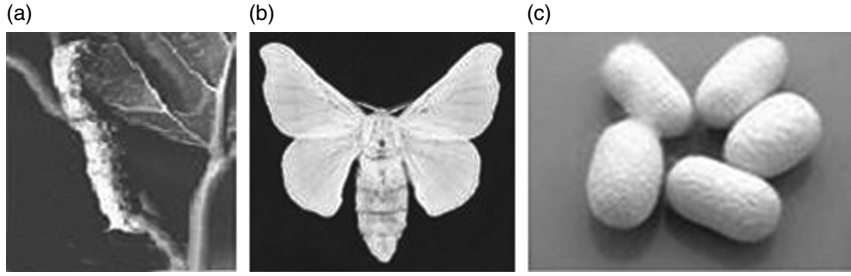
There are five major types of silk of commercial importance, obtained from different species of silkworms which in turn feed on a number of food plants. Except mulberry, other varieties of silks are generally termed non-mulberry silks. India has the unique distinction of producing all of these commercial varieties of silk.

Mulberry

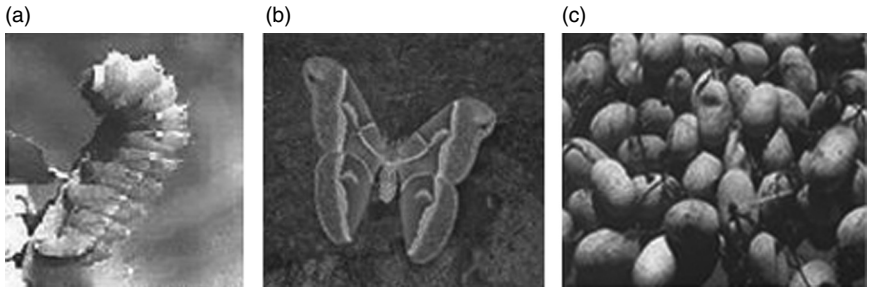
The bulk of the commercial silk produced in the world comes from this variety and the silk is referred to as mulberry silk. Mulberry silk (Fig. 7.1) comes from the silkworm, *Bombyx mori* L., which feeds solely on the leaves of the mulberry plant. These silkworms are completely domesticated and reared indoors. In India, the major mulberry silk producing states are Karnataka, Andhra Pradesh, West Bengal, Tamil Nadu and Jammu & Kashmir which together account for 92% of the country's total mulberry raw silk production.

Tasar

Tasar (Tussah) is a copperish coloured, coarse silk mainly used for furnishings and interiors. It is less lustrous than mulberry silk, but has its own feel and appeal. Tasar silk (Fig. 7.2) is generated by the silkworm *Antheraea mylitta* which mainly thrive on the food-plants Asan and Arjun. The silkworms are



7.1 Mulberry silk: (a) worm, (b) moth and (c) cocoons.



7.2 Tasar silk: (a) worm, (b) moth and (c) cocoons.

7.3 Oak tasar silk: (a) worm, (b) moth and (c) cocoons.

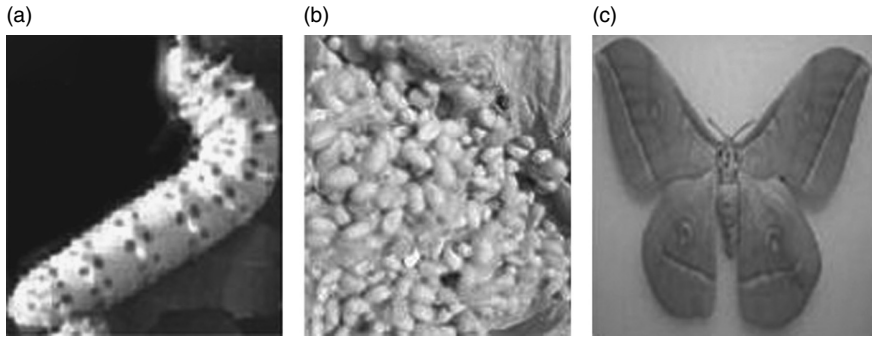
reared on the trees in the open. In India, tasar silk is mainly produced in the states of Jharkhand, Chattisgarh and Orissa, besides Maharashtra, West Bengal and Andhra Pradesh. Tasar culture is the mainstay for many a tribal community in India.

Oak tasar

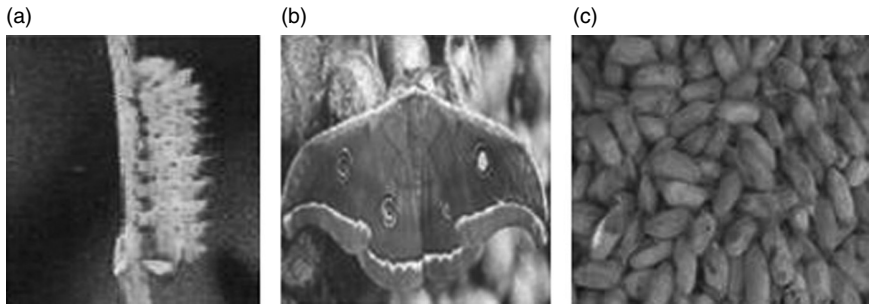
Oak tasar is a finer variety of tasar generated by the silkworm, *Antheraea proyeli* J (Fig. 7.3) in India which feed on natural food-plants of oak, found in abundance in the sub-Himalayan belt of India covering the states of Manipur, Himachal Pradesh, Uttar Pradesh, Assam, Meghalaya and Jammu & Kashmir. China is the major producer of oak tasar in the world and this comes from another silkworm which is known as *Antheraea pernyi*.

Eri

Eri is also known as endi or errandi. It is a multivoltine silk spun from open-ended cocoons, unlike other varieties of silk. Eri silk (Fig. 7.4) is the product of the domesticated silkworm, *Philosamia ricini* which feeds mainly on castor leaves. Ericulture is a household activity practised mainly for protein-rich pupae, a delicacy for the tribal. Resultantly, the eri cocoons are open-mouthed and are spun. The silk is used indigenously for preparation



7.4 Eri silk: (a) worm, (b) moth and (c) cocoons.



7.5 Muga silk: (a) worm, (b) moth and (c) cocoons.

of *chaddars* (wraps) for own use by these tribals. In India, this culture is practised mainly in the north-eastern states and Assam. It is also found in Bihar, West Bengal and Orissa.

Muga

Muga is a golden yellow coloured silk, which is the prerogative of India and the pride of Assam state. It is obtained from semi-domesticated multivoltine silkworm *Antheraea assamensis*. These silkworms (Fig. 7.5) feed on the aromatic leaves of som and soalu plants and are reared on trees similar to that of tasar. Muga culture is specific to the state of Assam and an integral part of the tradition and culture of that state. Muga silk is a high value product used in products such as sarees, mekhalas and chaddars.

7.2.3 Silk reeling

Introduction

Silk reeling is the process of unwinding the silk filaments from the cocoons and the process by which a number of cocoon baves are reeled together to

produce a single thread. This is achieved by unwinding filaments collectively from a group of cooked cocoons at one end in a warm water bath and winding the resultant thread onto a fast moving reel. Raw silk reeling may be classified by the direct reeling method on a standard sized reel, the indirect method of reeling on small reels, and the transfer of reeled silk from small reels onto standard sized reels on a re-reeling machine. The last technique is primarily applied in modern silk reeling processes (Mahadevappa *et al.*, 2001).

Various silk reeling devices

Many types of silk reeling machines are in use. The major structural features of the sitting type reeling machine, the multi-ends reeling machine and the automatic reeling machine are shown in Table 7.1.

Hand spinning wheel

The hand spinning wheel is a primitive spinning apparatus that is operated by two hands – one to drive the wheel and the other to feed in cocoons. One end of the reeling thread is wound onto each wheel, while cocoons are boiled in a separate pot.

Charka type reeling machine

The charka type is in use in India. This machine is operated with separate work motions in reel driving and cocoon feeding to reeling ends by two men per machine. Each machine has three ends or more to a reel, which is the

Table 7.1 Comparisons of structural features of various reeling machines

Kinds of machine	Sitting type reeling machine	Multi-end reeling machine	Automatic reeling machine
No. of reeling ends per basin	4 (2 to 8)	20 (10 to 40)	20
End-groping apparatus	Hand-driven	Semi-automatic	Automatic
End-picking apparatus	Hand-driven	Hand-driven	Automatic
Cocoon supplying apparatus	None	None	Equipped
End-feeding apparatus	None	Equipped (hand feeding)	Equipped (machine feeding)
Stop motion	None	Equipped	Equipped
Traverse guider	Equipped	Equipped	Equipped
Temperature of reeling bath (°C)	65–80	30–45	30–45
Reeling velocity (metres per minute)	180–250	50–80	120–200

same size as the large wheel of the re-reeling machine in order to save the re-reeling process (direct reeling method).

Sitting type reeling machine

There are two kinds of sitting type reeling machines, foot operated and motor-driven. The motor-driven reeling machine is not equipped with the stop motion attachment. There are obstacles to the production of good quality raw silk as the raw silk thread is wound too rapidly to maintain good quality control.

Multi-end reeling machine

The multi-end reeling machine eliminates the disadvantages of the sitting type reeling machine by increasing the number of reeling thread ends per basin and reducing the reeling speed. The operator must stand when running this machine as the number of reeling threads per basin increases by 20-fold. This is also called a 'standing type reeling machine'. Reeling efficiency is unchanged. Quality is better due to reduced speed.

The multi-end reeling machine is composed of a driving part, groping ends, picking ends, standby bath, reeling part, jetboute, stop motion, traverse guide, small reels, steam heating pipes and clutches.

The cooked cocoons contained in the tubs are carried into the groping ends portion of the reeling machine. From there, cocoons are moved into the picking ends apparatus. After correctly processing, the cocoons go to the standby bath for cocoon feeding. They are picked up by the reeler and fed to the reeling thread. During this step a number of cocoons will be dropped thus reducing the ratio of reeling cocoons per thread. The normal speed of cocoon feeding by a skilled reeler is around 16 times per minute. The reeling thread passes through the jetboute, silk button, first guider, second guider, third guider, fourth guider, traverse guide, and then is wound onto the small reels (Lee, 1999).

The cocoons dropped during the reeling process are gathered and reprocessed starting from the groping end section. The croissure of reeling thread is made between second guider and third guider, and the length of croissure is not for twisting of thread but for cohesion of thread by rubbing of composed filament. Typically, one set of multi-end reeling machines consists of ten basins with each basin having 20 ends or reels.

- *Basin*: The basin is rectangular with well-rounded corners and edges. It is only 10–12 cm deep. It is commonly made of dark coloured porcelain. The basin is subdivided into sections, each intended for a specific job such as brushing, end gathering of baves, stocks in reserve and waste collection.

- *Reels*: The reels of the multi-end reeling machine have a circumference of 75 cm. The frame of the reel is made of light metal or plastic. The reels are fitted into reel carriers and driven by a transmission shaft by connecting gears.
- *Traverse guide*: To ensure narrow and long web on the hank of the reel, a cam type traverse assembly has been fixed. This will make a convex surface in the hank, which is wound on the reel. The centre part of the hank is higher than the two axes.
- *Thread button*: Porcelain button thread-guiders are used for removing any dirt adhering to the thread passing through the tiny aperture in the button.

Automatic reeling machine

In raw silk production, the continuing increase of labour costs has mandated automation. Around 1950, the automatic reeling machine was invented, which controls the number of reeling cocoons per thread. Shortly thereafter, it was replaced by a second automatic reeling machine, which could automatically control the size of the reeling thread.

The automatic reeling machine mechanizes the processes of groping ends, picking ends, cocoon feeding to reeling thread and separation of dropped end cocoons during the reeling process (Sonwalker and Krishnaswamy, 1980). The efficiency of the automatic reeling machine compares favourably with the manual multi-end reeling machine.

The automatic reeling machine, though built to replace manual reeling, still requires manpower for problems with the reeling thread, which must be corrected by hand. A moderate amount of cooked cocoons are carried to the newly cooked cocoon feeder and then removed into the groping end part.

The end groped cocoons go to the picking end part and the correctly picked end cocoons are dispensed to the cocoon supplying basket which continuously rotates around the reeling basin on an endless chain belt. Usually, the reeling method is classified as the fixed cocoon feeding system and the moving cocoon-feeding system.

In the case of the fixed cocoon feeding system, the correctly picked end cocoons in the rotating cocoon baskets are poured into the arranging basin and here the picked end of each cocoon is hung on the end holding reel. When the size detector of the reeling thread indicates the feeding of cocoons, the picked end cocoons on standby are fed to the reeling thread by a feeding spoon. The reeling thread fed by picked end cocoons passes through the jetboute, silk button, first guider, second guider, third guider, fourth guider, denier indicator, fifth guider and traverser, and then it is finally wound onto small reels. The end dropped cocoons are placed into the

cocoon flowing tunnel by the remover plate. They are carried into the pupa separating drum. However, more reelable cocoons are poured into the end groping part by the conveyor belt and reels-finished cocoons are placed into the dropped pupa case for parchment layer cocoons.

In the case of the moving cocoon feeding systems, the correctly picked end cocoons are contained in the moving cocoon basket equipped with cocoon feeding apparatus. They are fed by the feeding fork of the cocoon basket, which moves simultaneously around the reeling basin. The denier indicator of the reeling thread indicates the feeding motion of the cocoon. After cocoon feeding, the reeling path of the moving cocoon feeding system is the same as that of the fixed cocoon feeding system.

Generally, one set of the automatic reeling machines has 400 ends, while one basin has 20 ends. The operating efficiency of the automatic reeling machine is easily affected by cocoon qualities, drying and cooking machinery as well as the quality of reeling water.

7.2.4 Silk throwing and fabric production

The first process of manufacture through which raw reeled silk must pass corresponds in some ways to the carding, combing and spinning in cotton and wool. In silk manufacturing it is called throwing. It is a process in which the silk strands are twisted together with other silk strands to form a thicker, stronger, multi-threaded yarn. Throwing produces a wide variety of yarns that differ according to the number of strands and the amount and direction of the twist imparted.

When the silk arrives at the throwing mills it is usually in the form of skeins just as it came from the filatures. Throwing naturally does not include the common processes of carding and combing, for the reason that the reeled silk is already in the form of thread. The only difficulty with it is that it is altogether too fine and delicate for use. Throwing is essentially a process of cleaning, doubling and twisting the single fibres as they come from the filatures. To do this requires several processes, most of which require different machines. The different processes used in throwing are discussed in brief in the following sections:

- *Opening bales, assorting skeins, and scouring*: The first process includes opening the bales containing the skeins, assorting according to sizes, colours and qualities of fibre, and laying up the skeins in piles of about 5 lb each. Each of these piles is weighed carefully, placed in cotton canvas bags, and then taken to the soaking room. Here the bags containing the raw silk are placed in tanks of warm water in which considerable soap has been dissolved. The temperature is usually regulated at about

90–100°C, and the silk is allowed to remain here for 10 or 12 h. This soaking softens the natural gum of the silk and makes it possible to unreel the silk from the skein with little difficulty or breakage.

- *Drying*: When the soaking has been concluded, the bags of silk are removed and the silk is placed in a drying machine which extracts the moisture by whirling the goods in a rapidly revolving, circular, sieve-like can. The centrifugal force of the rapid revolutions throws most of the moisture out of the skeins. Another drying method in common use, but one taking a longer time, is simply to hang the skeins on poles in a steam-heated chamber.
- *Softening*: When the skeins are fairly dried by either process, they are twisted, rolled and rubbed either by hand or by machinery so as to soften any stiff or hard spots left after the soaking. When this is completed, the silk is ready to be wound on spools, or bobbins as they are called.
- *Silk winding*: Each skein is then carefully placed on a reel and made ready for unreeling. The tiny silk fibre is unrolled from the skein gently, yet at a high speed. The winding apparatus here, as in nearly all other mechanisms used in textile industries, is fitted with apparatus that automatically stops the machine if anything goes wrong. Hence if the silk fibre coming off the reel should break, the machine will stop. This makes it possible for an operator in a silk winding room to take care of a great number of reels and bobbins. All that needs to be done is to replace empty reels with new skeins, to take away the full bobbins, and to attend to the difficulties causing breakage or stoppage of the machines.
- *Throwing or twisting*: The full bobbins are now taken to other machines that twist and combine the silk fibres into silk threads of various sizes. In making organzine, the single fibre is given a twist of several turns an inch before it is combined with others. The machine that combines the fibres is called the doubling frame, and the machine that twists the thread is called the twister. In some of the latest models of throwing machinery, the doubling and twisting is done on the same machine. These machines are so made that the number of turns to be given to the thread per inch can be exactly regulated. After the machine is once set and started, all that the operator needs to do is to replace empty bobbins with full ones from time to time and take away the twisted yarn bobbins when full. The doubling and twisting machinery is also equipped with automatic stop motions. If a bobbin runs out, or if a thread breaks, that part of the machine stops at once until the operator has attended to the difficulty. One operator in a modern plant can watch a great number of spinning threads. Types of twisted yarn include:
  - Poil: A silk yarn formed by twisting raw silk. The twist may be very slight or exceed 3000/m.

  - Tram: A silk yarn formed by doubling two or more silk threads and then twisting them slightly, generally to 80–150 turns per metre (TPM).
  - Crepe: Silk yarn made by doubling several raw silk threads and twisting them to very high levels in the range of 2000–4000 TPM.
  - Organzine: A silk yarn formed by doubling two or more yarn and twisting them in the opposite direction to that of the twist of the individual ends.
  - Grenadine: A silk thread formed by doubling two or more ends of poil, and then twisting them in a direction opposite to that of the individual poil ends. Grenadine is three to four times more tightly twisted.
  - Cordonnet: A thick silk thread obtained by doubling and throwing several tram ends in the direction opposite to the twist of the individual tram ends.
- *Stretching*: After the twisting, the silk threads are run through another machine called a stretcher. In this machine the thread is first passed through a bath of soap and water and then drawn over rollers which stretch the thread at every point where it is larger in diameter than it should be. The process equalizes the diameter of the thread so that it becomes uniform throughout. Such inequalities result from uneven tension in the various threads in the doubling or twisting machines. After the stretching, the silk is reeled into skeins about 50 in long, containing, according to the size of the thread, from 500 to 2500 yards.
- In the manufacture of silk fabrics, generally the warp and weft yarns are dyed before weaving. Such goods are said to be 'yarn dyed'. From the throwing mills and dye houses, the silk is taken to the weaving mills to be made into cloth. The process of weaving is very similar to cotton fabric weaving except for minor modifications on the looms.
- During the process of warping, the bobbins holding the warp are sent to the warping room. About 400–500 bobbins are placed on a frame called a creel. From the creel the thread is unwound upon a warper reel in the proper lengths which, for broad silks and dress goods, usually run from 300 to 600 yards per piece. If different colours are used, they are all properly arranged in order and number at this point.
- From the warper's reel the warp is wound onto the warp beams in sections, usually about 30 in number for yardwide cloth. Cloth of this width will require from 9000 to 21 000 warp threads, the number depending upon the size of the warp used and the size of mesh desired. The process of winding the warp requires about one day's work of one skilled operator. Every layer of silk that goes onto the beam is separated from the rest by a sheet of stiff paper the width of the beam. This paper prevents the warp from becoming entangled on the beam.

- Next, the warp ends are passed through the loom harnesses, every thread being passed through its proper heddle eye. The ends are passed similarly through the reed, and then all is ready for the loom.
- The process of threading the harness is usually shortened in a weaving mill by leaving in the harness and reed the last part of the warp of the piece previously woven, and then tying or twisting the ends of the old warp to the new. The new warp can then be pulled through into the harness at once. This process can be carried out much more quickly than when the new warp threads are to be passed through the heddle by hand. After the new warp has been passed through, the old ends are cut off, and the new ends knotted together in clusters to prevent their slipping back again.

7.3 Microstructure and appearance

Silk fibres (*Bombyx mori*) spun out from silkworm cocoons consists of fibroin in the inner layer and sericin in the outer layer. Each raw silk thread has a lengthwise striation, consisting of two fibroin filaments of 10–14 lm each embedded in sericin. The chemical compositions are, in general, silk fibroin of 75–83%, sericin of 17–25%, waxes of about 1.5%, and others of about 1.0% by weight. Silk fibres are biodegradable and highly crystalline with well-aligned structure. It is known that they also have higher tensile strength than glass fibre or synthetic organic fibres, good elasticity and excellent resilience. Silk fibre is normally stable up to 140°C and the thermal decomposition temperature is greater than 150°C. The densities of silk fibres are in the range of 1320–1400 kg/m^3 with sericin and 1300–1380 kg/m^3 without sericin. Silk fibres are also commercially available in a continuous fibre type.

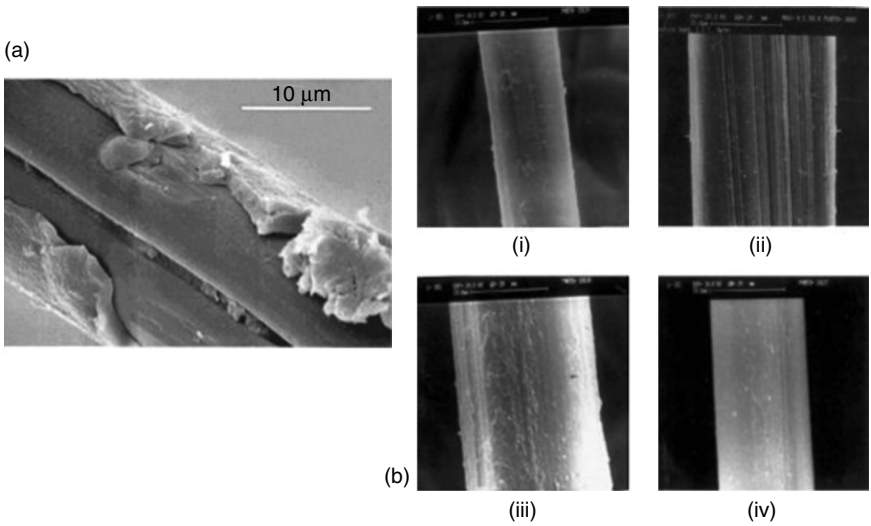
7.3.1 Longitudinal view

Scanning electron micrographs of longitudinal views of undegummed and degummed silk fibres are presented in Fig. 7.6a.

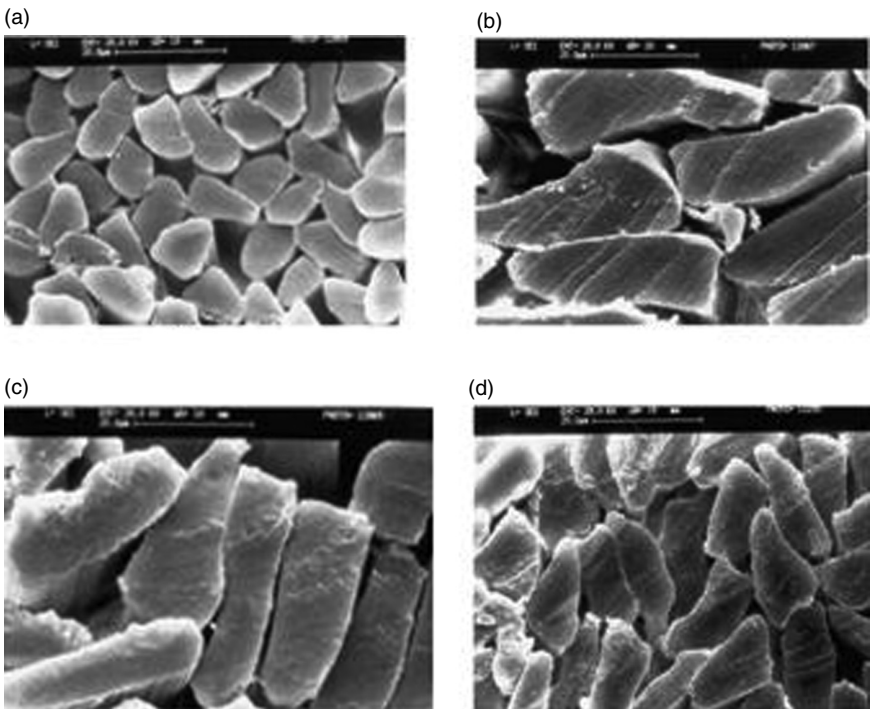
It may be observed that mulberry shows a more or less smooth surface (Fig. 7.6b) whereas the non-mulberry silks such as tasar, muga and eri (Fig. 7.6b (ii–iv)) all have striations on their surface compared to mulberry.

7.3.2 Cross-sectional view

The scanning electron micrographs of cross-section of silk fibres are presented in Fig. 7.7. The cross-section of silk fibre which is made of two types of protein namely, sericin and fibroin, is shown. It can be seen that two strands of fibroin filaments are enveloped by non-fibrous sericin. When a strand of



7.6 Longitudinal view of silk fibres: (a) undegummed, (b) degummed: (i) mulberry, (ii) tasar, (iii) muga and (iv) eri.



7.7 Cross-sectional view of silk fibres (degummed): (a) mulberry, (b) tasar, (c) muga and (d) eri.

fibroin filament is enlarged for its inner structure, it appears like a bundle of fibrils in which a large number of fibrils are accumulated (Minagawa, 2000).

There are variations depending upon the variety of silkworms and also among the individual cocoons. It may be observed that, in this respect, the mulberry and non-mulberry silks exhibit an altogether different cross-sectional morphology. The mulberry silks show a more or less triangular cross-section and a smooth surface (Fig. 7.7a). Among the non-mulberry varieties, tasar and muga exhibit an elongated rectangular or a wedge-shape cross-section and a large cross-sectional area (Fig. 7.7 b and c). The eri silk has a more or less triangular shape (Fig. 7.7d).

Usually in the case of the cocoon fibres of domestic silkworms like mulberry and eri, the cross-section is irregular ranging from triangular shape to circular shape. Moreover, even in the same fibroin filament, there are variations in the cross-section area depending upon the level of the cocoon layer.

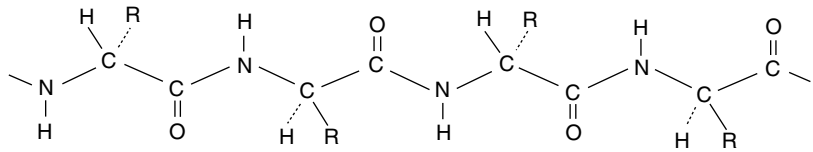
7.3.3 Crystal structure

Most silkworm cocoon and spider dragline silk fibres contain assembled anti-parallel β-pleated sheet crystalline structures (Fraser and MacRae, 1973; Lucas *et al.*, 1960; Marsh *et al.*, 1955). Silks are considered semi-crystalline materials with 30–50% crystallinity in spider silks, 62–65% in cocoon silk fibroin from the silkworm *B. mori*, and 50–63% in wild-type silkworm cocoons. In the β-sheet crystals the polymer chain axis is parallel to the fibre axis. Furthermore, the polyalanine repeats or the glycine-alanine repeats are the major primary structure sequences responsible for β-sheet formation. The β-sheets consisting of the glycine–alanine crystalline repeats in the silkworm fibre are asymmetric, with one surface primarily projecting alanyl methyl groups and the other surface of the same sheet containing hydrogen atoms from the glycine residues (Matsumoto *et al.*, 2006).

X-ray analyses of silk have shown the relatively high orientation of polypeptide chains along the fibre axis. The fibre has a two-phase system consisting of crystalline and amorphous phases. The crystalline part consists of sections of polypeptide chains containing glycine, alanine and serine and has the structure shown in Fig. 7.8.

These sections have simple branching as a result of which the chains may be closely and compactly arranged to the full extent. Sections containing residues of tyrosine, praline, diamine and dicarboxylic acids are characterized by bulky residues which impede regular and close packing of chains and as a result, less oriented (amorphous) regions of the fibre are formed.

The groups found in the amorphous region are more accessible to the action of chemical reagents, for example, full saturation of the basic groups with some acids is possible without any change in the X-ray pattern of the



7.8 Polypeptide chain of fibroin molecule.

fibre. In contrast, hydroxyl groups of serine residues are less accessible to the action of chemical reagents.

7.4 Amino acid composition

The amino acid composition varies in different varieties of silk. Three major amino acids such as serine, glycine and alanine may be found in mulberry and non-mulberry varieties. Among the other major amino acids present are tyrosine and valine. In general in mulberry silks glycine, alanine and serine together constitute about 82%, of which about 10% is serine. Tyrosine and valine may be considered next to these at about 5.5 and 2.5%, respectively. The overall composition of acidic amino groups (i.e., aspartic and glutamic acids) in the mulberry variety is greater than that of the basic amino acids. The other important aspect is the composition of amino acids with bulkier side groups. The presence of bulky side groups can hamper close packing of molecules and hinder the crystallization process. In general, a large portion of the mulberry fibroin is made up of simple amino acids such as glycine and alanine, suggesting a favourable condition for crystallization (Sen and Babu, 2004).

Compared to the mulberry silks, the total amount of glycine, alanine and serine account for about 73% in the non-mulberry variety, less by about 10%. All the non-mulberry silks exhibit a high proportion of alanine compared to that in the mulberry variety. The proportion of alanine is about 34% in tasar, 36% in eri and 35% in muga. This value is consistent but is lower than that particularly for muga (~ 44%). On the other hand, the glycine content in these varieties is about 27–29%, which is lower than that found in the mulberry varieties (~ 43%).

In addition, the non-mulberry varieties have a substantial proportion of amino acids with bulky side groups, especially aspartic acid (4–6%) and arginine (4–5%), which means that not only the acidic but also basic amino acid levels are greater. It is interesting to note the presence of sulphur-containing amino acids (i.e., cystine and methionene) in all the varieties of silk. Methionene content in non-mulberry silks is slightly higher (0.28–0.34%) compared to that found in mulberry varieties (0.11–0.19%), whereas the cystine content is comparable (Sen and Babu, 2003). Amino acid composition of different varieties of silk is presented in Table 7.2.

Table 7.2 Amino acid composition of silk fibres

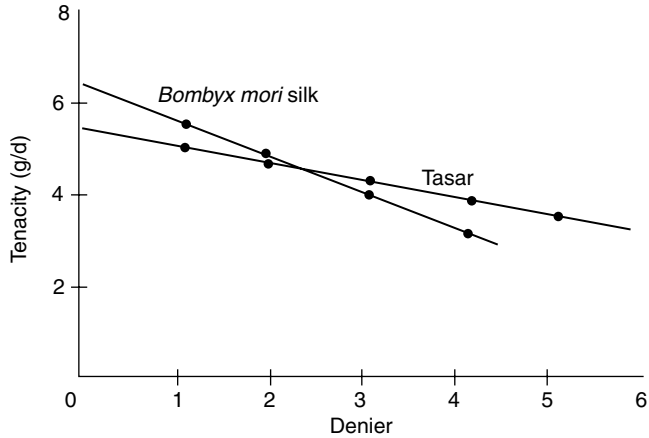
Amino acid	Amino acid composition (mol. %)			
	Bombyx mori (mulberry)	*Antheraea mylitta* (tasar)	*Antheraea assama* (muga)	*Phylisomia ricini* (eri)
Aspartic acid	1.64	6.12	4.97	3.89
Glutamic acid	1.77	1.27	1.36	1.31
Serine	10.38	9.87	6.11	8.89
Glycine	43.45	27.65	28.41	29.35
Hystidine	0.13	0.78	0.72	0.75
Arginine	1.13	4.99	4.72	4.12
Threonine	0.92	0.26	0.21	0.18
Alanine	27.56	34.12	34.72	36.33
Proline	0.79	2.21	2.18	2.07
Tyrosine	5.58	6.82	5.12	5.84
Valine	2.37	1.72	1.5	1.32
Methionine	0.19	0.28	0.32	0.34
Cystine	0.13	0.15	0.12	0.11
Isoleucine	0.75	0.61	0.51	0.45
Leucine	0.73	0.78	0.71	0.69
Phenylalanine	0.14	0.34	0.28	0.23
Tryptophan	0.73	1.26	2.18	1.68
Lysine	0.23	0.17	0.24	0.23

7.5 Properties of silk

Silk fibers are remarkable materials displaying unusual mechanical properties: strong, extensible, and mechanically compressible. They also display interesting thermal and electromagnetic responses, particularly in the ultraviolet (UV) range and form crystalline phases related to processing. The mechanical properties of silk fibers are a direct result of the size and orientation of the crystalline domains, the connectivity of these domains to the less crystalline domains, and the interfaces or transitions between less organized and crystalline domains. Other properties of silk such as good thermal stability, optical responses, dynamic mechanical behavior and time dependent responses have all been used in number of applications in various fields. In this chapter, details of various properties of different varieties of silk have been presented.

7.5.1 Tensile properties

The tensile properties of different varieties of silks in terms of tenacity, elongation-at-break and initial modulus have been determined by a number of workers (Freddi *et al*., 1994; Iizuka, 1994, 1996; Iizuka and Itoh, 1997; Iizuka *et al*., 1993).



7.9 Tenacity (in grams force/denier (g/d)) versus denier of the thread relationship.

Studies conducted on some mulberry and non-mulberry varieties by E. Iizuka *et al.* (1993) reveal that the tenacity, elongation and modulus are all dependent on the linear density of the filament, and the linear density or the mean size in turn depends on the silkworm race. The tenacity is found to be linearly related to the linear density of the filament (Fig. 7.9). The correlation is negative, i.e., as the linear density increases, tenacity decreases. A similar trend has been observed for modulus too. Elongation on the other hand increases with an increase in linear density.

The tenacity ranges between 2.5 and 4.82 gram-force per denier (g/d) for Japanese and Chinese mulberry varieties, 2.4 and 4.32 g/d for Indian mulberry varieties and 3.74 and 4.6 g/d for Indian tasar varieties (Iizuka, 1996). In a study on chemical structure and physical properties of antheraea assama (muga) silk, it has been reported that the tenacity of muga varies between 3.2 and 4.95 g/d (Freddi *et al.*, 1994; Iizuka *et al.*, 1993). Another important non-mulberry variety, eri, showed the lowest tenacity value, ranging between 2.3 and 4.0 g/d (Iizuka and Itoh, 1997).

Elongation-at-break, on the other hand, showed a higher value for all the non-mulberry silks compared to mulberry varieties. The values range between 31% and 35% for tasar, 34% and 35% for muga and 29% and 34% for eri silks, respectively. The elongation values for mulberry varieties ranged between 19% and 24%. Some of the mechanical properties of different varieties of silk are summarized in Table 7.3.

7.5.2 Optical properties

Silk fibroin extracted from silkworm cocoons is a unique biopolymer that combines biocompatibility, implantability and excellent optical properties. Silk may be used as an optical material for applications in biomedical

Table 7.3 Mechanical properties of different varieties of silk

Variety	Sex	Dynamic modulus (10^{10} dyn/cm^2)	Tan δ*	Tenacity (g/d)	Elongation (%)
Shunreix shougetsu	M	1.847	—	5.265	20.36
(mulberry)	F	1.808	—	5.207	21.48
A. mylitta	M	1.132	0.030	3.412	31.36
(tropical tasar)	F	1.087	0.035	3.256	31.12
A. proylei	M	1.305	0.023	4.123	31.45
(temp. tasar)	F	1.087	0.025	4.128	31.48
A. assama (muga)	M	1.205	0.020	3.170	34.83
	F	1.230	0.023	3.823	34.10

*Tan δ is the ratio of the loss modulus to the storage modulus, and is often called the damping or the loss factor.

engineering, photonics and nanophotonics. Silk can be nanopatterned with features smaller than 20 nanometre (nm). This allows manufacturing of structures such as (among others) holographic gratings, phase masks, beam diffusers and photonic crystals out of a pure protein film. The properties of silk allow these devices to be 'biologically activated' offering new opportunities for sensing and biophotonic components.

Many interesting bio-optical devices can be fabricated by doping silk films with fluorescent materials. Some work is being carried out to explore the possibility of enhancing the light emission by patterning the silk film surfaces, as well as making tunable wavelength devices and printing specific patterns on silk film surfaces.

Lustre associated with silk is due partly to the influence on the pattern of light-reflection of its triangular shape. In an attempt to understand the optical properties of silk, many researchers have determined the refractive index and birefringence of fibres. The refractive index of silk generally varies through its cross-section. The birefringence (n) value varies between 0.051 and 0.054 for mulberry silk and between 0.030 and 0.034 for non-mulberry silks (Tsukada *et al.*, 1994).

7.5.3 Visco-elastic behaviour

Silk fibre exhibits visco-elastic behaviour. Time dependent mechanical properties of silk fibres such as stress relaxation, creep, creep recovery, etc., have also been the subject of interest. Creep is a phenomenon associated with time dependent extension under an applied load. The complementary effect is stress relaxation under a constant extension.

Creep and stress relaxation behaviour of silk has been reported (Das, 1996). The instantaneous extension and secondary creep are both higher for tasar silk compared to those for mulberry silk. The stress relaxation was also found to be higher in non-mulberry silks than in mulberry silk.

Silk has also been shown to exhibit inverse stress relaxation phenomenon (Das, 1996). The inverse relaxation could be observed for both mulberry and tasar silks when the level of strain was maintained below a certain value. Inverse relaxation becomes higher with the increase in peak tension. Cyclic loading has been found to reduce the extent of inverse relaxation.

7.6 Applications of silk

Silk is one of the most beautiful fabrics available, with a long and colourful history and changing applications in the world today. Be it for gowns, medical use, home decor and more, the uses of silk constitute a wide and varied topic.

7.6.1 Textile and apparel

Silk's good absorbency makes it comfortable to wear in warm weather and while active. Its low conductivity keeps warm air close to the skin during cold weather. It is often used for clothing such as shirts, blouses, formal dresses, high fashion clothes, negligees, pyjamas, robes, skirt suits, sun dresses and kimonos. Silk's elegant, soft lustre and beautiful drape make it perfect for many furnishing applications. It is used for upholstery, wall coverings, window treatments (if blended with another fibre), rugs, bedding and wall hangings.

Silk has several uses in daily life. It is a common cloth used for high-end garments, including wedding gowns and blouses. It appears frequently in accessories like handbags and headbands and scarves. Because of its lustre and texture, it also is commonly used for home decoration, especially as sheets, bedding, curtains and cushions. It is also used for warmth and skiing garments because of its remarkable ability to retain heat; paradoxically, it is also a popular summer cloth because it keeps the wearer cool in warmer weather.

Silk continues to be used as a material to produce fine dresses. Chinese women continue to have their wedding cheongsam dresses made out of silk. They choose this material because it is one of the finest materials known in both ancient and modern Chinese culture. Delicately woven dragons, flowers and butterflies are sewn into the silk dresses. The material is thick so it allows a woman to look leaner and have cleaner lines. Additionally, the shininess of the material is very flattering and alluring. Women's evening gowns are also made of silk. It also falls well and is a material that is slightly warmer allowing the body to feel a bit of warmth, even if she wears a sleeveless gown in the winter.

Silk shirts remain popular amongst women. Especially for women's blouses, silk is regarded as classic and flattering since it drapes a woman's

body. Additionally, the thinness of the material allows women to easily tuck the shirt into their skirts or wear a jacket on top without having the jacket look bulky. For men, silk shirts are becoming more and more popular. Men have started wearing silk short-sleeve shirts as well as long-sleeve shirts. Men used to only wear silk shirts for the evening, but the lightness of the material has made it a popular alternative to cotton.

Silk is also used very frequently for pyjamas. Silk pyjamas feel soft and smooth and are very comfortable while sleeping. Both men's and women's pyjamas are now made of this fine thread. For women, silk is often used to make lingerie as well. Using this fine thread has become a popular option since it is smooth, tends not to catch on hair, and is very breathable for the body.

Silk scarves have also been around for a long time. The number of gorgeous patterns and designs on the scarves has made these items very expensive. Women tend to tie the scarf around their necks for warmth and for style. Men tend to carry one in their pocket as an alternative to the cotton handkerchief. Be prepared to pay more for a silk scarf with a fancy design.

More and more, manufacturers of bedding have started to make silk sheets and silk pillowcases. The health benefits of silk are starting to be more widely known. Sleeping on them helps prevent coughing and sneezing, especially for those allergic to dust mites since the mites do not like silk. Additionally, sleeping on silk sheets may be beneficial to women's hair, helping it to have fewer tangles and split ends. Beyond the solid colour sheets, many makers of luxurious bedding are now offering their own version of sheets with lots of different patterns and designs in stores.

Silk has come a long way since the Silk Road. It is still a highly sought after material. Having pieces of fine clothing or a scarf or even a silk sheet is often seen as the ultimate extravagance.

7.6.2 Biomedical applications

Silk, and especially *Bombyx mori* silk, has a very long history in biomedical applications. In recent years, the reported exceptional nature of silk led to increased interest in silk for biomedical applications (Hakimi *et al.*, 2007). Silk fibroin has been increasingly studied for new biomedical applications due to the biocompatibility, slow degradability and remarkable mechanical properties of the material. In addition, the ability to now control molecular structure and morphology through versatile processability and surface modification options has expanded the utility of this protein in a range of biomaterial and tissue-engineering applications. Silk fibroin in various formats (films, fibres, nets, meshes, membranes, yarns and sponges) has been shown to support stem cell adhesion, proliferation and differentiation *in vitro* and

promote tissue repair *in vivo*. In particular, stem cell-based tissue engineering using 3D silk fibroin scaffolds has expanded the use of silk-based biomaterials as promising scaffolds for engineering a range of skeletal tissues like bone, ligament, and cartilage, as well as connective tissues like skin (Wang *et al*., 2006). More recent studies with well-defined silkworm silk fibres and films suggest that the core silk fibroin fibres exhibit comparable biocompatibility *in vitro* and *in vivo* with other commonly used biomaterials such as polylactic acid and collagen (Altman *et al*., 2003).

Silk from the silkworm *B. mori* has been used as biomedical suture material for centuries. The unique mechanical properties of these fibres provide important clinical repair options. Silk has been used in native fibre form as sutures for wound ligation and became the most common natural suture, surpassing collagen over the past 100 years (Altman *et al*., 2003). Silk sutures are used in ocular, neural and cardiovascular surgery, as well as a variety of other tissues in the body. Silk's knot strength, handling characteristics and the ability to lie low to the tissue surface make it a popular suture in cardiovascular applications. The above features of silk fibroin have led to the recent emergence of silk-based biomaterials for a wide range of cell and tissue studies such as scaffolds, films, sponges, hydrogels, bone materials, cardiovascular devices, etc.

7.6.3 Fibre-reinforced composites

Silk is a fibre with remarkable mechanical properties. This unique characteristic of silk has led to its use in fibre reinforced composites for various applications. Silk yarn is easily available as the waste product of textile industry, so the composite is cost effective and the perfect utilization of a waste product. Though the silk is extensively used as a valuable material for textile purposes, in recent years it is being widely considered as a reinforcing material for composites made from epoxy and other biodegradable biopolymeric resins. The organization of the silk fibres can contribute significantly to impact resistance by ensuring either or both a sufficient strength of the composite and a good deformability of the composite.

7.6.4 Silk nonwovens

Silk nonwoven fabrics can be developed from silk reeling waste and hard waste generated during twisting and weaving on shuttle-less looms. In the process of conversion of cocoons to fabric, about 4000 metric tons (MT) of silk waste of different forms is being generated annually (in India alone). At present, this waste is used for manufacture of spun silk yarn, noil yarn, throwster yarn and carpet yarn, besides hand-spun yarn. This waste can be more

effectively utilized for development of silk nonwoven fabrics for diversified end uses. In addition, the hard waste generated to the extent of 300 MT in silk twisting and weaving by 100% export oriented units (EOUs) is not being used for any value-added purpose except in manufacture of coarser yarn for carpets. The same may effectively be utilized for development of nonwoven silk fabrics. The other wastes like pierced and cut cocoons may also be tried. The web formation by air laid method and bonding by chemical/needle punching may be attempted for production of nonwoven webs.

Based on the end uses, nonwoven fabric of specific weight can be produced for various applications including technical and medical textiles. The possible end use applications are unlimited and nonwoven silk fabrics could be a potential input in the areas of inner lining for warm garments, headwear, ties, garments and blankets, carpets, furnishing and home apparels, automotive carpeting and insulation, wall coverings, handicrafts like wall paintings, wall hangings, wall coverings, gift tags, purses/wallets, table mats, matting for pictures, journal/book/album covers, greeting/invitation/business cards, lamp shades and many more.

7.7 Future trends

With improved analytical techniques, together with the tools of biotechnology, a new generation of silk-related materials is envisioned that depart from traditional textiles and medical sutures, which are currently the focus for these materials. These proteins are already finding broadening applications in medical fields such as biomaterials. The unique and remarkable mechanical properties of silks have already prompted studies for their application in high performance materials, as well as modes to mimic these features through more traditional synthetic organic polymer chemistry routes. Since the specifics of the silks can be modulated through genetic manipulation, through processing and through choice of a starting protein, a great deal of control of structure, morphology and functional attributes in materials derived from these proteins can be obtained. This control is already exploited in the biomaterials arena and suggests that this family of novel proteins can serve as an important blueprint for understanding structure–function relationships as well as for new and novel materials. A number of consumer products are already promulgated based on silks, including cosmetics, hair replacements and shampoos, among others. Sutures, biomaterials for tissue repairs, wound coatings, artificial tendons, bone repair, and related needs may be possible applications since silks are biocompatible and degrade slowly in the human body. Genetically engineered silks have also been commercialized as cell culture plate coatings to improve cell adhesion. Genetic variants of silks are also actively pursued as controlled release systems to deliver pharmaceuticals in a variety of systems.

New trends in production and applications of silk have to be deployed for maintaining a sustainable market for silk. As a result, researchers in Japan have developed fluorescent silks. The world's first silks exhibiting fluorescence and other pioneering properties have been successfully developed as a result of transgenic silkworm research conducted by Japanese researchers. Fluorescent silk thread is made by pulling threads from cocoons made by silkmoth larvae (silkworms). Researchers have now developed three lines of transgenic silkworms. The first line produces silk threads that emit green, red or orange fluorescent light. These threads were created by introducing into silkworm eggs genes that promote the generation of fluorescent proteins. It has been possible to achieve green fluorescence using genes extracted from jellyfish, a technique developed by Nobel Prize-winning chemist Osamu Shimomura, and red and orange fluorescence with genes extracted from coral, a technique that has already been used in commercial applications. The fluorescent silk threads have great potential for use in the fashion industry, and there is expected to be considerable demand for them from producers of high-end apparel.

Though the traditional practices were used only for textiles, the new approach extends silk's application towards nutritional, cosmetic, pharmaceutical, biomaterial, biomedical and bioengineering, automobile, house building and art/craft applications. This trend suits silk due to faster production rates and increasing global demand for its variable eco-friendly composites and viable contributing impact on value, employment and environmental safety. This move, however, requires more awareness among stakeholders; training and ideas exchange between entrepreneurs; and service accessibility for consumers. Increased applications of natural fibres for varied applications will create awareness about insect-based natural fibres and silk will be subject to continued use and innovative research in the future.

7.8 Conclusions

Although substitution of natural materials has been underway for several decades, natural silks remain commercially important because of their unique properties, as well as their relative environmental stability and biocompatibility, and consumer preferences. The industries should look at the utilization of silk fibre in total for innovative marketable products of modern society's application and appreciation, which facilitate its all-round development. Though traditionally, silk was used for making textiles, the new approaches must be attempted to extend the application of silk in nutritional, cosmetic, pharmaceutical, biomedical and bioengineering, automobile, house building and art/craft products. During the past decade a great deal of progress has been made in understanding silk's genetic and protein structures. On the other hand, materials scientists have long been fascinated

by structure–function relationships in silk proteins. Since the exploration of biomaterial applications for silks, aside from sutures, is only a relatively recent advance, the future for this family of structural proteins to impact clinical needs appears promising.

7.9 Sources of further information and advice

Recommended textbooks are mentioned in the references. Periodicals such as *Indian Silk*, *Indian Journal of Sericulture* and *Sericolgia* can be referred to for information on Indian sericulture, silk technology and other related topics. Occasional articles are found in *Journal of Applied Polymer Science*, *Journal of Textile Institute*, *Textile Research Journal* and *Indian Journal of Fibre and Textile Research.* Additional information on sericultural aspects of silk, silk fabric manufacture, dyeing, printing and processing aspects are available online at www.indiansilk.kar.nic.in and www.fao.org. and in the *Journal of Natural Fibers* by Taylor & Francis, Philadelphia, USA.

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8

Wool fibres

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Abstract: The large-scale and long tradition of the wool industry, as well as the appeal of wool itself as a natural fibre, have long stimulated innovation in other fibre processing industries. This chapter discusses the structure and morphology of wool and its characteristics and mechanical and chemical properties. The end uses of wool fibre are also briefly reviewed and examined in relation to its properties.

Key words: wool industry, structure and morphology of wool, properties of wool, end uses of wool, ecological benefits.

8.1 Introduction

Wool fibre is natural, sustainable and biodegradable, all of which are highly valuable properties in the textile industry. The environmental advantage provided by these properties is an increasingly popular requirement for textile fibre, but wool has many other inherent benefits, which have earned it a reputation for quality from global manufacturers and consumers.

8.1.1 The wool industry

The wool industry produces around 2.1 million tons of greasy wool per year, involving more than 1 billion sheep around the world maintaining high standards of animal welfare. Wool is produced on several million smallholdings and commercial farms worldwide. Retail sales of wool amount to US $80billion of wool products annually. In the European Union (EU) alone, the wool industry is made up of thousands of industrial manufacturing companies working with wool as a raw material. The industry employs millions of people in wool production, harvesting and throughout its many processing stages.

Performance is critical in textiles and wool has a long-standing reputation of versatile wool products. Many generations of change and development have demonstrated the vast potential of wool to meet, adapt and fulfil complex product scenarios.

Wool offers practical attributes that far exceed man-made fibres and as it is grown, not made, it has a complex physical cell structure that gives the

material the natural ability to breathe. A unique characteristic of wool is its ability to absorb and release humidity, providing a garment or product that can adapt to different climates and situations. Its high water and nitrogen content also make it naturally flame retardant and it meets many international regulations without the need for chemical treatments. Wool can also absorb unhealthy carbons in the atmosphere, helping to provide a cleaner environment.

Wool is a globally traded commodity and its market diversity is vast and expanding. It is found in many sectors including fashion, active wear, flooring and interiors, aviation, architecture, manufacturing, medical use and protective apparel. Due to this dynamic versatility, wool can be described as the original 'Smart' fibre. Research and development continually pushes this potential further, opening doors to a future that will safeguard the wool industry, which is a major worldwide employer and brings multiple benefits to people, products and the planet.

Despite all these positive attributes, consumers, industry and governments have been slow to acknowledge the health and safety benefits wool can provide. As a result of subsequent low prices, fewer farmers are likely to produce wool in the future. With this in mind, the wool industry needs support to combat this threat to its existence.

8.2 The effects of the economy on wool

While economic growth forecasts for the major wool consuming countries remain solid, heightened uncertainty is causing purchasers of all commodities (including wool) to be cautious, lowering demand and prices. However, sheep numbers were only slightly lower at the start of the 2011/12 season, which helped wool production to lift moderately, and wool prices were slightly higher at the beginning of 2011/12, and came down by the summer of 2012.

8.2.1 Demand

The current financial uncertainty in Europe is affecting economic and retail conditions around the world, which has an effect on the demand for wool. Before widespread concerns about the Greek government's ability to service its debts emerged in the first quarter of 2010, a steady economic recovery was predicted in the major wool-consuming countries. The recovery was well underway and had been proceeding earlier and at a faster pace than the International Monetary Fund (IMF) and other forecasters had expected in October 2009. This economic recovery was expected to increase the demand for raw wool. However, the problems with the Greek government debt repayment gave rise to similar fears about other European countries. This resulted

in a new bout of uncertainty about the economic recovery, not only in Europe, but in other major wool-consuming countries. The immediate impact of the concerns led to a fall in share markets around the world, a drop in the euro and falling commodity prices. After steady rises from the start of 2009, the price of oil, metals and wool all fell back, starting in April. Since mid-April 2010, the price of metals (US$) have decreased by around 18%, oil prices by 7% and wool prices by 7%. Commodity prices declined as the Greek debt problems fuelled doubt about the ability of governments to service and repay the large debts accumulated during the global financial crisis. This in turn raised concerns about the sustainability of a recovery in the world economy.

As well as these fears, there has been further uncertainty about the resilience of the economic recovery in the US and the sustainability of strong economic growth in China. For example, the IMF, in its latest *World Economic Outlook*, 'forecasts a continuing economic recovery but renewed financial turbulence and Euro-area problems cloud the outlook'. The IMF predicts that the world economy will demonstrate 4.6% growth in 2010 (after a contraction of 0.6% in 2009) and 4.3% growth in 2011. This forecast for 2010 is higher than its forecast in April.

For the main wool-consuming countries, the IMF predicts growth in 2010 of 10.5% in China, 3.3% in the US, 2.4% in Japan, 1.4% in Germany, 0.9% in Italy, 1.2% in the UK, 1.4% in France and 5.7% in South Korea. This demonstrates the rebound in economic growth in the main wool apparel consuming countries (weighted by wool consumption). 2011 was an excellent year for wool, with strong demand for both products, and fibres. The demand weakened in the second half of the year as a result of a slowdown in the European and US economies. The prospects for wool in 2012 are dominated by concerns about the European and US economies (Landmark Operations, Australia: Wool Economic Focus, January 2012).

The uncertainty about financial stability and the economic recovery has caused wool textile mills to be cautious and follow a hand-to-mouth pattern in purchasing. In addition to this, it is not obvious from the annual figures that economic growth in all the major wool-consuming nations in the second half of 2010 is expected to be lower than in the first half of the year. In the northern hemisphere, the majority of wool clothing is purchased in the second half of the year. Taking this factor into account, the outlook for wool demand is still positive, but not as positive as it was before the Greek financial problems emerged.

8.3 Wool production

Wool, the filamentous appendage of sheep skin, is the best known representative of the α-keratin fibres, a large class of mammalian hairs with a common chemistry, structure and morphology. Wool fibre has similar properties whatever the origin of the sheep. Economically the sheep is an ideal fibre factory, producing minimal waste and providing milk and

meat well as wool. Compared to other mammals, the sheep produces the highest amount of wool fibre per unit of pasture area. Due to the fact that sheep eat grass without uprooting it, pasture regenerates quickly after it has been used for sheep. This can be compared with the Cashmere goat, after which at least 2–3 seasons are required for pasture regeneration. One sheep produces approximately 1 kg greasy wool (or some 0.6 kg clean wool) per annum from 1 ha of average pasture (Moeller and Popescu, 2009).

8.3.1 Ecological benefits of wool

Wool is:

- natural,
- renewable,
- sustainable,
- biodegradable,
- low carbon impact,
- energy efficient.

Wool is one of the earth's most sustainable resources; it is an annually shorn and replenished resource. It is also a planet-friendly fibre due to its ability to biodegrade without harm to the environment, and it can be recycled. These inherent advantages continue to underpin wool's heritage as the best natural eco-fibre. The amount of wool available to supply the global textile industry is only limited by the number of sheep farmed across the world, which, in turn, is limited by the available pasture surface, and economic demand for fibre.

Sheep are susceptible to several pests and insecticides and other methods have been developed to combat them. To reduce flystrike, which notably occurs in the climate of Australia, the farmers developed a process called mulesing. Mulesing is a surgical procedure by which the wrinkled skin in the animal's breech area is cut away from the perianal region down to the top of the hind limbs. There is ongoing research to develop an alternative to this operation.

The wool industry is committed to the highest standards of sheep care and well-being and supports scientific research in this area. Wool farmers are dedicated to the job of looking after their animals and keeping them healthy, to ensure the wool maintains the required quality for the textile industry. Farmers are always looking towards the best available animal husbandry practices to ensure good economic management of their business and optimum results in their produce. The routine care of sheep should

Table 8.1 Composition (%) of greasy wool

Wool type	Grease and suint	Sand and dirt	Vegetable matter	Fibre
Merino (< 25 μm)	15–30	5–40	0.5–10	30–60
Crossbred (25–33 μm)	15–30	5–20	1–5	40–65
Long wool (> 33 μm)	5–15	5–10	0–2	60–75

Note: The micron (μm) limits for the three wool types are only orientative.

include shearing, foot pairing and feet care, drenching for worms, dipping for lice, crutching, vaccinations, nutrition (pasture management and supplements) and birthing assistance. This proactive approach provides uncompromising welfare standards for sheep across the world.

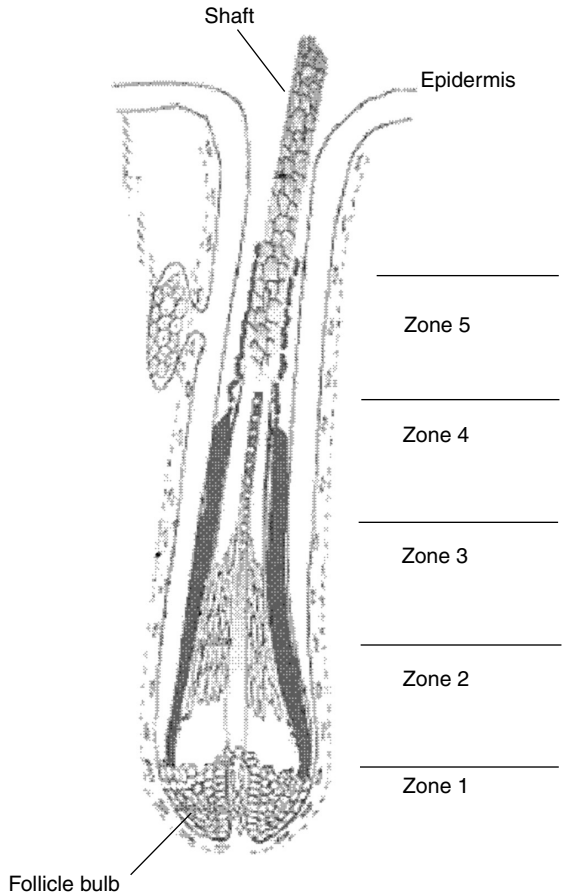
The process of transferring wool fibre from the sheep to the textile industry is labour-intensive. The shearing and collecting of greasy wool is a manual operation, as are skirting (selecting the parts of the fleece to be used) and classing. The collected greasy (raw) wool comprises various amounts of different impurities as detailed in Table 8.1, which have to be removed by scouring and carbonising before the clean fibre is delivered to the textile industry.

Wool fibre is made up of two parts: the follicle, which is located below the surface of the sheep skin and produces the fibre material, and the shaft, which grows from the follicle and is composed mainly of the fibrous protein α-keratin (Fig. 8.1) (Zahn *et al.*, 2003). The fibre can be categorised in terms of zone 1 – bulb zone (proliferation and differentiation); zone 2 – elongation (fibril formation); zone 3 – pre-keratinisation (lateral aggregation); zone 4 – hardening (keratinisation); zone 5 – post-hardening (hard keratin).

While the follicle is a living cell producing a continuous substance, the fibre shaft (wool) is a complex 'cornified' multicellular tissue, which mechanically acts as a whole. The rate of fibre shaft production is around 1 cm per month for each follicle. The number of follicles per sheepskin (the follicle density) varies according to breed and dictates the amount of wool produced per animal. This explains why the annual yield of one sheep can vary from 1 kg (most sheep breeds) to 4 kg (Australian Merino) clean wool fibre. The diameter of the fibre is also genetically determined, although this can also be slightly affected by weather and food. Consequently, wool fibre diameters range from 11 (fine Australian Merino) to 100 (sheep of northern hemisphere) microns.

8.4 Chemistry and morphology

An elemental analysis of wool shows similar percentages of carbon (around 50 wt%), hydrogen (7 wt%), oxygen (22 wt%), nitrogen (16 wt%) and

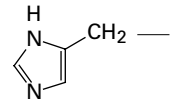
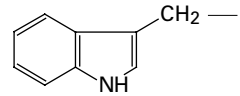


8.1 The follicle and the shaft of a hair. For explanation of zones, see text.

sulphur (5 wt%) wherever the wool comes from (Popescu and Hoecker, 2003). The high sulphur content in wool and other animal hairs comes mainly from the high cystine content of these fibres. Trace elements can also be detected. The total ash content of keratin fibres ranges from 0.3 to 0.9%, and the most frequent trace metals found are Ca, Cd, Cr, Cu, Hg, Zn, Pb, Fe, As and Si, incorporated into keratin from extraneous sources (Sukumar and Subramanian, 2003).

From a chemical point of view wool is a polymer made of amino acids. Total hydrolysis of the peptide bonds which keep the amino acids together yields the 20 common natural α-amino acids (the general structure: H_2N–CHR–COOH) given in Table 8.2 (Popescu and Hoecker, 2007). More than 100 amino acids bind each other to form the protein chains which make up wool fibre.

Table 8.2 The 20 common natural α-amino acids found in α-keratin fibres

Group	Name	Side chain, R
'Acidic' amino acids and their ω-amides		
	Aspartic acid	$-CH_2-COOH$
	Glutamic acid	$-(CH_2)_2-COOH$
	Asparagine	$-CH_2-CONH_2$
	Glutamine	$-(CH_2)_2-CONH_2$
'Basic' amino acids and tryptophan		
	Arginine	$-(CH_2)_3-NH-C(NH_2)=NH$
	Lysine	$-(CH_2)_4-NH_2$
	Histidine	
	Tryptophan	
Amino acids with hydroxyl groups in the side chain		
	Serine	$-CH_2-OH$
	Threonine	$-CH(CH_2)-OH$
	Tyrosine	$-CH_2-C_6H_4-OH$
Sulphur containing amino acids		
	Cysteine	$-CH_2-SH$
	Thiocysteine	$-CH_2-S-SH$
	Cystine	$-CH_2-S-S-CH_2-$
	Methionine	$-(CH_2)_2-S-CH_3$
Amino acids without reactive groups in the side chain		
	Glycine	$-H$
	Alanine	$-CH_3$
	Valine	$-CH(CH_3)_2$
	Proline	
	Leucine	$-CH_2-CH(CH_2)_2$
	Isoleucine	$-CH(CH_2)-CH_2-CH_3$
	Phenylalanine	$-CH_2-C_6H_5$

Because they contain both cationic and anionic groups, the fibres are amphoteric. The cationic aspect is due to the protonated side groups of arginine, lysine and histidine and the free terminal amino groups. Anionic groups are present in the form of dissociated side groups of aspartic and glutamic acid residues and carboxyl end groups.

Research into the peptide arrangement in the protein fibre has been going on since the first half of the twentieth century. Astbury *et al*. (Astbury and Street, 1931; Astbury and Woods, 1932) used X-rays to demonstrate

the presence of a crystalline phase in hair. The X-ray diffraction pattern of animal hairs shows a meridian reflection at 0.51 nanometre (nm) and an equatorial reflection at 0.98 nm. Interpreting these results, Pauling (1951) proposed the α-helix structure to account for the secondary structure of the keratin fibre.

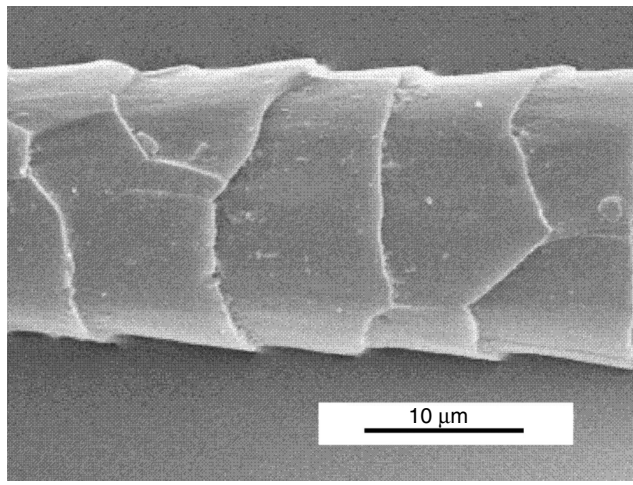
The organisation of the α-helices in proto-filaments, proto-fibrils to micro-fibrils and intermediary filaments follows a pairing rule: two α-helices are joined together by hydrophobic bonds into a double helix structure: a dimmer of 50 nm length; two dimmers twist together into a coiled-coil tetramer; two chains of tetramers self-assemble to become a protofilament; two protofilaments form a protofibril; and four protofibrils arrange into a microfibril or intermediate filament (IF), the crystalline structure of wool fibre.

Morphologically, the fibres are composed of a cortex and a cuticle. Each of the two components is formed of various other morphological components. The cortex contains cortical cells and the cell membrane complex. The cortex may be formed from up to three types of cells, i.e. the ortho-, the para- and the meso-cortex cells (Popescu and Hoecker, 2007). Additionally, in coarse hair there are air-field cavities made up of special types of cells, forming a central medulla. Ortho- and para-cortex cells are found in Merino wool fibres arranged as two strands twisted together, which causes the crimp in the fibre, exposing the ortho-cortex cell strand to the exterior and the para-cortex cell strand to the interior of the curl of the fibre. In other wools (such as the coarse wool of Lincoln sheep) ortho- and para-cortex cells are arranged as concentric cylinders, with the ortho-cells always located in the core. Other hair fibres have only one type of cell, usually ortho-cortex cells. There are also differences in the organisation of the cell components. Ortho-cortex cells exert their components, the microfibrils, in a helical array (whorl structure), making the resulting macrofibrils more distinct and clearly packed than in para-cortex cells, which in turn exert microfibrils in an array parallel to the fibre axis. The cortical cell is further composed of macrofibrils and intermacrofibrillar material. In summary, the cortex is formed of microfibrils (intermediate filament, IF, or keratin proteins, KP) and keratin associated proteins (IFAP or KAP) which compose the intermicrofibrillar matrix containing cytoplasmatic and nuclear remnants. This ensemble is wrapped up in the cuticle, which acts as an external sheath with its own architecture. The cuticle is formed of four layers: the epicuticle, the a-layer, the exocuticle and the endocuticle.

The epicuticle has a peculiar structure, as the layer responsible for the paradox of the keratin fibres (Popescu and Hoecker, 2007): a hydrophobic surface wrapping a hydrophilic core. The reason for this is the presence of 18-methyl eicosanoic acid (18 MEA), which is anchored by an ester bond to a protein matrix. As a result of this structure the wool fibre has a surface

Table 8.3 Animal fibres structure

Composite	Type	Component 1	Component 2
α-Keratin fibre	Ring/core	Cuticle	Cortex
Cortex	Filament in matrix	Cortex cells (spindle shape)	Cell membrane complex
Cortex cell	Filament in matrix	5–8 macrofibrils	Intermacrofibrillar matrix
Macrofibril	Filament in matrix	500–800 microfibrils (IFs)	Intermicrofibrillar matrix



8.2 Wool fibre surface showing the scales.

tension of about 30 Newton/metre (N/m), below the value of water and most oils, which makes the fibre naturally water- and soil-repellent. This unique property of wool, which does not appear in other textile fibres, is very useful in producing low-maintenance carpets.

Summing up, the α-keratin fibre is an example of a natural composite system, with a complex dual structure at all levels (Table 8.3). Wool fibres have a slightly elliptic cross-section and are protected by the scales arranged on their surface like tiles on a roof (see Fig. 8.2). The number of amino acids differs between α-keratin fibres and silk, as shown in Table 8.4 (Popescu and Wortmann, 2010).

8.5 Properties of wool

The complex structure of wool is schematically represented by a three-phase model. The model describes the main mechanical and thermal properties

Table 8.4 Amino acid composition of wool, cashmere and yak fibres

Amino acid (mol %)	Wool	Cashmere	Yak
Glycine	8.1	9.9	9.8
Alanine	5.0	5.8	5.6
Serine	10.2	12.2	10.0
Glutamine + glutamic acid	12.1	12.4	12.5
Cystine	11.2	6.0	6.4
Proline	7.5	6.7	6.6
Arginine	7.2	7.0	7.1
Leucine	6.9	7.5	8.3
Threonine	6.5	6.6	6.6
Asparagine + aspartic acid	6.0	6.2	6.7
Valine	5.1	5.5	5.9
Tyrosine	4.2	3.5	3.4
Isoleucine	2.8	3.2	3.5
Phenylalanine	2.5	2.8	3.0
Lysine	2.3	2.8	3.0
Tryptophan	1.2	—	—
Histidine	0.7	1.2	1.0
Methionine	0.5	0.5	0.4

by representing wool as formed of α-helical intermediate filaments (IFs), a crystalline phase accounting for 25–30% of the dry fibre, largely with axial orientation and embedded in an amorphous matrix (Feughelman, 1959; Wortmann, 1992). The region between the intermediate filaments and the amorphous matrix is the interface, considered as the third phase of the 3-phase model (Hearle, 2000; Istrate *et al*., 2009). The contribution of the cuticle, the outer protective sheet, to the mechanical properties of wool is considered to be largely negligible (Feughelman and Hally, 1960; Bendit and Feughelman, 1968). A comparison of fibre properties is presented in Table 8.5.

This characteristic structure leads to the marked anisotropic properties of wool fibre, and explains the differences between axial and lateral swelling (Onions, 1962).

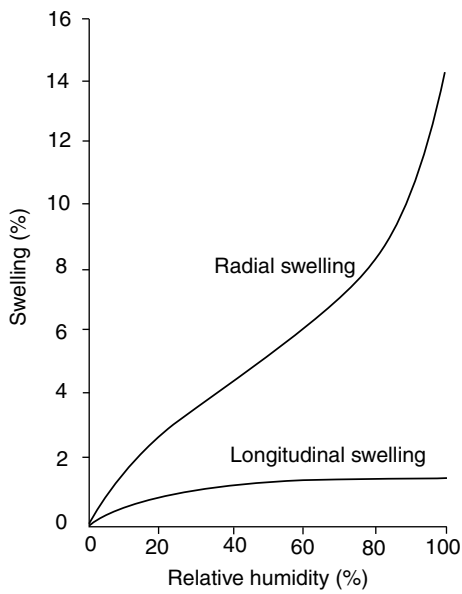
The curves in Figure 8.3 show the changes in wool fibre dimensions as the fabric goes from dry to wet, with increasing regain. The swelling is a property of the amorphous matrix only. The effect is more pronounced (16% between dry and wet) on radial than on axial (1–2%) direction, and reflects the strong tendency of the matrix to absorb water.

The stress–strain curve recorded for wool fibre is illustrated in Figure 8.4, representing the fibre's behaviour at different levels of humidity. The curve can be broken down into three regions that are affected differently by humidity. After decrimping (not shown in Fig. 8.4), the first region shows the tension in the fibre increasing fairly linearly up to a strain of 1–2%. After this point, elongation increases rapidly compared to small increases

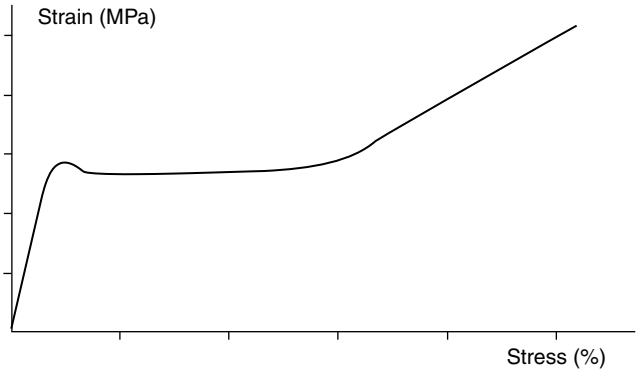
Table 8.5 Comparison of fibre properties

	Wool	Polyester	Nylon	Acrylic
Appearance				
Drape	1	3	3	2
Texture	1–2	3	3	2
Colour	1–2	3	2	1
Crease retention	2	1	2	3
Wrinkle recovery	2	1–2	2	3
Comfort				
Moisture absorption	1	5	4	5
Elasticity	1–2	2	2	3
Permeability	1–2	4	4	4
Insulation	1	4	4	4
Performance				
Water repellency	1–2	2	2	3
Abrasion resistance	2–3	1	1–2	2–3
Laundering	1	1	1	2
Drycleaning	1	3	2	4
Ultraviolet (UV) stability	2	1	3	1
Safety				
Fire resistance	1–2	3	3	5
Anti-static	1	5	4	5
Acid resistance	1–2	1	4	1

Source: IWTO.
Note: Grades on scale from 1 = excellent, 2 = very good, 3 = good, 4 = moderate, 5 = poor.



8.3 The anisotropic swelling of wool fibre.



8.4 Stress–strain curve of wool fibre recorded at 20°C under standard relative humidity (RH 65%).

in stress. This section of the curve is known as the yield region and ends at around 25–30% elongation. The third region of the stress–strain curve is called the post-yield region, which terminates on rupture of the fibre. The three slopes of the initial, yield and post-yield regions are in the approximate ratio of 100:1:10, respectively.

Of particular interest is the recovery of the fibre after a strain of up to 30%, which is one of the reasons for the typically long life of wool. Wool lasts for hundreds of years without losing its appearance. This is exemplified by the 2500-year-old Pazyryk carpet found in a Siberian tomb, still on display in the Hermitage Museum in St Petersburg. Several models have been proposed to describe the stress–strain curve of wool and its hysteresis behaviour, the best known being Feughelman's model (1994) and the Chapman–Hearle model (1968). In both cases the mechanical effort is seen as being distributed between the a-helix of the crystalline region of the fibre (IFs) and the amorphous matrix. Their contributions depend strongly on the moisture content of the fibre, with the matrix effect almost vanishing at 100% relative humidity. The difference between the two models is mainly due to the way in which the crystalline and amorphous phases are connected: while Feughelman's model does not consider any link between the two phases, the Chapman–Hearle model assumes that they are bridged by disulphide bonds.

The elastic and visco-elastic properties of wool fibres, as determined by the mechanical properties of the IFs and the matrix, largely determine the crease resistance, dimensional stability, drape and handling of wool fabrics. The moisture content plays a very important role in the mechanical behaviour of the fibre.

Wool fibres are hygroscopic, and the amount of water taken up depends on the relative humidity of the air, temperature and the history of the fibre. The sorption and desorption curves exhibit a hysteresis, reflecting the different behaviour of the fibre on uptake of water molecules compared to

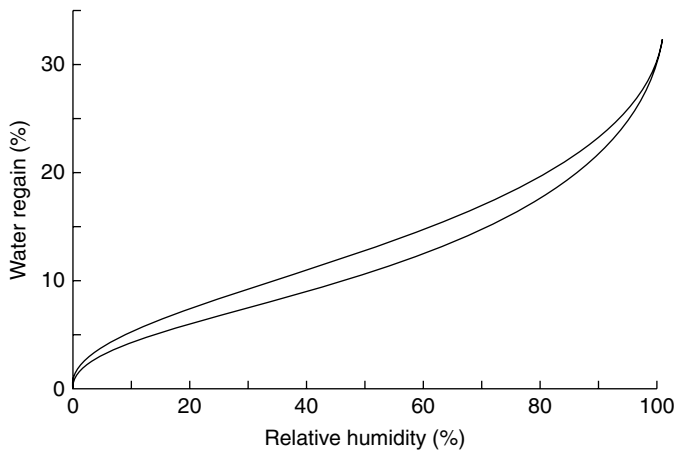
its behaviour on release (Fig. 8.5). At average relative humidity, the difference between the two curves is about 2%. The moisture uptake is accompanied by a dissipation of heat, which is around 110 Joules per gram (Jg^{-1}) for adsorption of 18% moisture (Speakman and Cooper, 1936).

The fact that wool can absorb up to 35% water before feeling 'wet' is one of its fundamental properties, responsible for the breathability of the fibre and having major consequences for the use of wool in fibre and textile processing.

Wool is also unique among textile fibres in its thermal behaviour, which makes it a safe fibre. When wool is heated in air, the loss of water is recorded up to 105°C, followed, after 200°C, by two processes accompanied by major mass losses. The first process is endothermic, occurring from 200°C to 400°C and the second process is exothermic and occurs within the range 450–600°C. As a result, a general scheme for the thermal degradation of wool may be proposed (Popescu *et al.*, 1995):

$$\text{wool} \rightarrow \text{dry wool} \rightarrow \text{pyrolysis products} \rightarrow \text{oxidation products}$$

During the endothermic step the chains of proteins decompose to lighter products and volatile compounds such as CO, NH_3 and H_2S are evolved (Ingham, 1971). Some of the pyrolysis products then react with oxygen through several gas phase reactions, leading to oxidation products. The large amount of hard-to-oxidise products formed during the pyrolytic step hinders the oxidative phase and makes almost impossible for wool to keep burning in a normal atmosphere once the ignition flame is removed. The ability to burn is measured by the limiting oxygen index (LOI) (Beck *et al.*, 1975). Wool records 25 for LOI, one of the highest values among the textile fibres. This means that the atmosphere, which is made up of about 21%



8.5 Moisture sorption–desorption hysteresis.

oxygen, needs to contain at least 25% of oxygen to maintain the oxidative reactions in wool. Therefore, lack of sufficient oxygen means that wool self-extinguishes once the ignition is removed.

A list of the most significant and useful properties of wool is given below:

Wool is healthy, because it:

- is breathable,
- can control humidity,
- is less allergic,
- can adsorb toxic chemicals,
- can reduce sound.

Wool is safe, because it:

- is flame resistant,
- provides UV protection,
- reduces static electricity,
- has high thermal resistance.

8.5.1 Benefits

Non-flammable

As mentioned before, wool is non-flammable, unlike almost all alternatives. It simply requires more oxygen to burn than is available in the air, making it a superior fibre for fire safety. Furthermore, it does not melt, drip or stick to the skin when it burns and a comparison with other fibres of the carpet industry demonstrates its advantages (Table 8.6).

The flame-resistance of wool fibre may be further improved by a quick and simple treatment with zirconium or titanium salts, known as Zirpro treatment, developed in the early 1970s by the International Wool Secretariat (Benisek, 1984).

The treatment is based on the ability of negatively charged titanium or zirconium acid complexes to exhaust on positively charged wool under strongly acidic conditions. Over the years studies have shown that titanium complexes are more effective than those of zirconium in improving the flame-resistant property, but they also cause wool fibres to turn yellow (Benisek, 1974).

The zirconium and titanium complexes that provide the flame-resistant effect are those made with alpha-hydroxy carboxyl acids (tartaric, citric, malic acids) or with fluorides. While the complexes with hydroxyl-carboxyl acids will only exhaust at or above 100°C (Benisek, 1971), fluoride complexes can exhaust at below 100°C, making the latter treatment more protective and less

Table 8.6 Flammability index of carpets

Fibre	Ignition index (0–40)	Flame spread index (0–20)	Heat evolved index (0–20)	Smoke evolved index (0–20)	Flammability index (0–100)
Wool	26	0	0	10	36
Polypropylene	28	12	14	14	68
Nylon	30	14	16	16	76
Acrylic	28	16	20	14	78

Note: The lower the value, the better the index.
Source: IWTO.

energy consuming. Consequently Zirpro treatment is performed, in most cases, by exhausting potassium hexafluorozirconate (K_2ZrF_6) on wool, from a solution of 6–8% (o.w.f.) at a temperature of 60°C and a pH of 2–3 (adjusted with hydrochloric acid), for up to 30 min. Zirpro treatment imparts a flame-proof property to wool, which is resistant to washing, light and compatible with any other finishing process. This makes it a popular choice for fibres designated for interior textiles, as well as for apparel fabrics, for example uniforms.

The standard Zirpro treatment means that wool fibre meets most flame-proofing requirements and standards. There are also variants of the treatment which are designed to meet certain applications and standards such as 'Low Smoke Zirpro', a treatment for wool furnishings requiring flame-resistance, low smoke and toxic gas emission (e.g., for aircraft furnishing), or 'Zirpro/tetrabromophthalic acid', a treatment designed to meet flammability standards requiring very short after-flaming times.

Low allergy

Wool does not harbour dangerous chemicals, dust or mould that can lead to allergic or other reactions.

Fewer volatile organic compounds (VOCs)

VOCs are organic chemicals which vaporise and enter the atmosphere as an air pollutant. Wool is a natural product and emits no VOCs. Wool can also absorb a number of VOCs from surrounding materials and lock them permanently within the fibre core (Hoecker and Wortmann, 2003).

A long safety record in insulation

The unbeatable safety profile of wool has been proven. Insulation from man-made fibres is more recent and is made from extremely fine, brittle strands, which can become airborne on handling. These strands can damage the respiratory tract when inhaled and cause severe irritation to the skin.

This necessitates safety clothing and masks during installation; these precautions are not necessary with wool insulation.

UV protection

Wool helps protect against serious risk to skin and associated long-term health issues caused by the potentially damaging rays of the sun. Testing of various textiles in clothing shows that wool has a natural UV protection factor of 30+ in more than 70% of cases – this is much higher than most synthetics and cotton.

A better material for sensitive locations

The safety profile of wool makes it an ideal product for every location but particularly for those where health and safety is paramount. Hospitals, churches, schools, nursing homes and public transport can all benefit from its long-lasting natural abilities and high performance ensuring sustained risk prevention (Hoecker and Wortmann, 2003).

Less static electricity

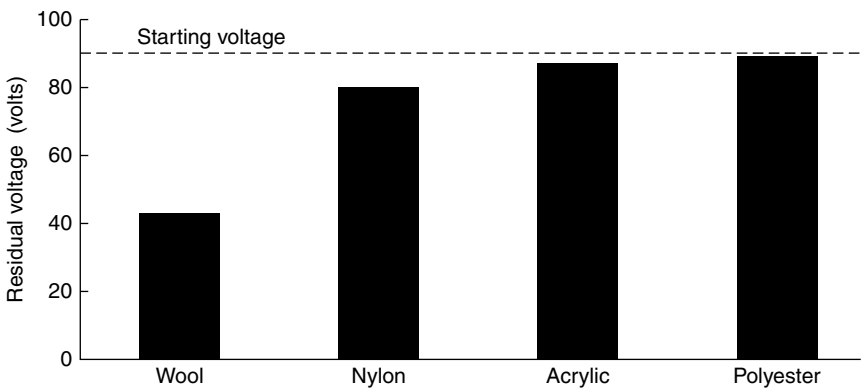
Due to its moisture absorption capabilities, wool is far less likely to cling to the body when worn (Fig. 8.6).

More sound absorption

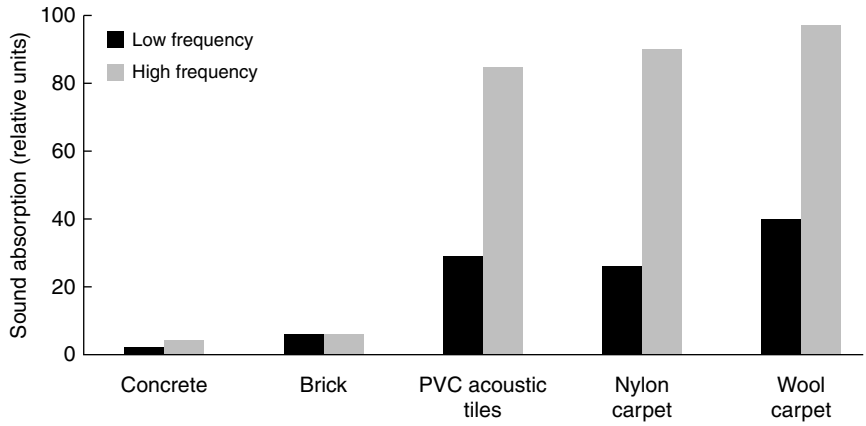
Wool is a perfect sound-insulating material, with the capacity to dampen or absorb both high and low frequency sound (Fig. 8.7).

Clothes that breathe

Wool has the natural ability to breathe and can absorb up to 35% of its own weight in moisture due to its hydrophilic core (see also Fig. 8.5). This



8.6 Static charge leakage from various fabrics. (Source: IWTO.)



8.7 Typical airborne sound absorption values. (Source: IWTO.)

is then naturally released as vapour into the air and provides comfort from perspiration throughout the day. No other fabric offers this amazing wearer-sensitive comfort.

Multi-climate

Wool is active and synthetics are passive. The active response of wool to fluctuations in body temperature means that it offers warmth when it is cold, but will self-adjust when temperatures increase by releasing heat and moisture to maintain a comfortable clothing climate.

Health

Wool has a number of health benefits including improved sleep for the general population, reduced risk of SIDS (sudden infant death syndrome) for babies and lower incidence of microbial infection for hospital patients.

Active perspiration control

Wool is more effective than synthetics at absorbing perspiration caused by exertion. The natural absorption of wool moves moisture away from the skin so that it can evaporate, ensuring coolness and dryness.

Easy care

Wool has had a reputation for being difficult to machine wash in the past, but modern wool fabrics are treated to be truly easy-care.

The surface of wool fibres is covered with scales which all point towards the tip of the fibre. When two fibres rub against each other, the particular surface topography results in what is known as the differential friction

effect (DFE) whereby the fibres experience greater friction when they slide along the scales than when sliding against them. When wool is agitated or put under pressure in water the DFE causes the fibres to move together and become entangled, a process known as felting. Felting causes the fabrics to shrink and become denser (Makison, 1979).

When it is controlled, the felting is used for finishing purposes and allows producing items with a specific wool look, which cannot be achieved by other fibres (Shetland knitwear, loden or melton fabrics, felted hats, etc). When it cannot be controlled, for example in household fabric care, felting becomes undesirable because it leads to shrinking of the material.

Since the shrinkage is due to the presence of scales on the surface of the fibres, there are two main types of shrink-proofing processes: degradative and additive. In other words, the scales can either be damaged or covered. The most effective processes are those which combine the benefits of both, by partly removing the scales and covering the fibres with resins to render the surface smooth. The first and still most widely used process for shrink-proofing is the Kroy-Hercosett process, in which wool scales are destroyed using chlorine and the resulting fibre is wrapped in an epichlorhidrin-based resin, Hercosett.

Generally the scales are degraded by the attack of oxidative reagents. The effects of chlorine attack on wool scales have been studied for almost 100 years, being observed firstly by Allwörden (1916, p. 120). During the last decades several other reagents (most often ozone and permono-sulphuric acid) as well as enzymes have been proposed as replacements for chlorine, in order to address the environmental issue of reducing AOX (halo-organic pollutants) in water (El-Sayed *et al.*, 2001).

The resins applied to the fibres may either (a) wrap the fibres, smoothing their surfaces and lowering the differential friction effect, or (b) build bridges between fibres, arresting their relative movement.

A non-conventional alternative, which does not affect the scales but activates their surfaces for further resin application, is offered by plasma treatment (Kan *et al.*, 1998).

General speaking, the shrinking of wool fabrics is now fully controllable and wool items with machine wash and tumble-dry care instructions (total easy care – TEC) have become standard on the market.

Durable

Wool fibres can be bent 20 000 times without breaking, which explains why wool garments are so long-lasting.

Odour reduction

Wool will absorb moisture, reducing sweat on the body; this in turn reduces the amount of resulting body odour caused by sweat and its contact with any bacteria on the skin.

Quick drying

It was formerly thought that synthetics dry at a faster rate than wool; however, the latest technology enables wool to be as quick-drying.

Stain resistant

Wool fibre has a protective layer that prevents stains from being absorbed. As it is also anti-static, it picks up less dust and lint from the air.

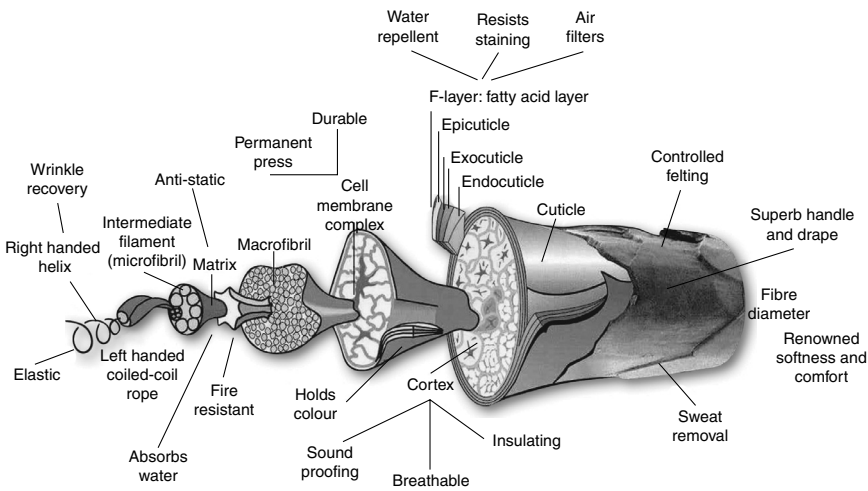
8.6 Industrial usage of wool

Wool is a long-life fibre, maintaining its moisture retention properties and flexibility for many years. When disposed of in soil, it biodegrades without harming the environment, closing an optimum life-cycle and fertilising the land (Hoecker and Wortmann, 2003).

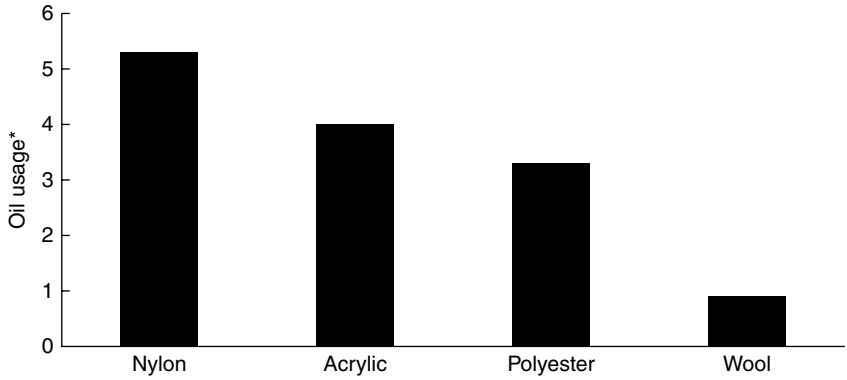
Figure 8.8 summarises the discussion of wool structure and indicates the practical use of each of the structural elements.

A fundamental requirement of sustainable products is the capacity to limit energy use in production. Significantly less energy is required in the production of wool products compared to man-made fibre products, ensuring that carbon dioxide emissions are kept very low (see Fig. 8.9). Therefore the increased use of wool, which has a *low carbon impact*, can positively reduce the level of greenhouse gases in the atmosphere.

Wool is used in every field of textiles (see Fig. 8.10): in apparel (suits, jackets, pullovers, coats, sportswear, skiwear, scarves, gloves, socks, hats,



8.8 Wool structure and application summary. (Source: CSIRO.)



8.9 The energy required for producing textiles from various textile fibres. (*kg of oil used to produce 1 kg of final fabric.) (Source: IWTO.)

underwear, active wear, children's wear, thermal underwear, waterproof fabrics, casual wear, shoes, formal wear, hand knitting wool, uniforms, kilts), interior textiles (carpets, duvets, mattresses, pillows, blankets, upholstery, tapestries, lamps, chairs, rugs, futons, bedspreads, tablecloths, wall coverings, sofas, felts) and technical textiles (aircraft interiors, thermal insulation, sound insulation, sound vibration control, roof insulation, filtration, fire proof wear, police uniforms, military uniforms, air conditioning, bandages, second skin injury prevention, piano felts, wool filters for dust chemical odours, billiard cloths, automotive composites, tennis balls).

The most important fields of application and preferred usage of wool are underlined below, illustrating its advantages.

- Clothe our children in wool and…

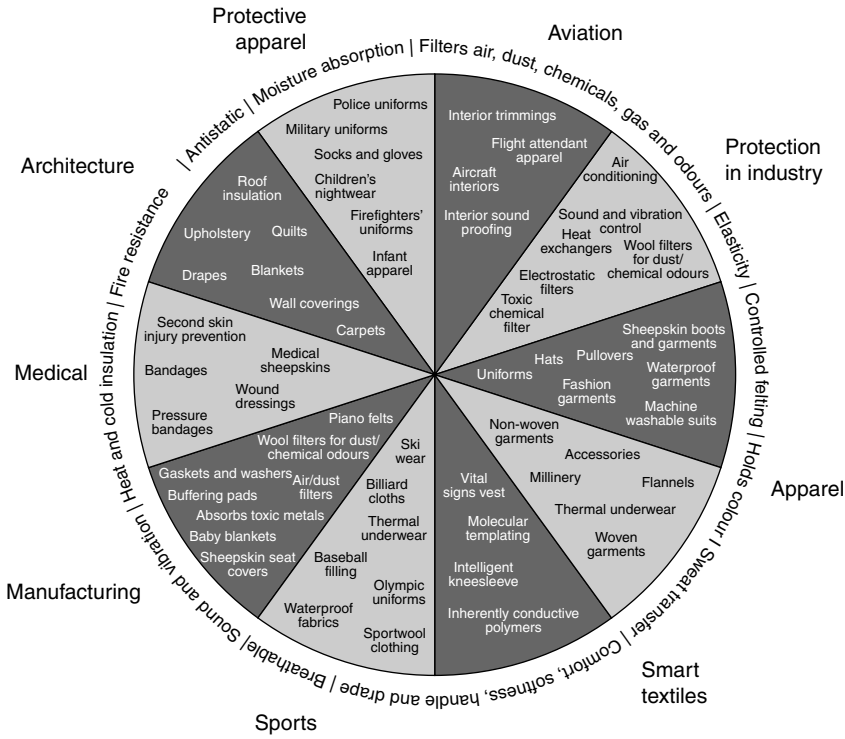
allow their skin to breathe more easily, keeping them warm in winter and cool in summer whilst protecting them from harmful UV rays and the risk of fire.

- Use wool in active-wear, sports-wear and work-wear and…

discover superior comfort and a healthy wearer-sensitive climate, which provides perspiration management and essential odour control. Wool's natural elasticity – up to 30% flexible extension without damage hashnum; coupled with its resilience, moisture absorption and unique flame retardant attributes make it a healthy, safe fibre for sport and work wear.

- Fit more wool-rich carpets in our houses and public buildings and…

limit airborne dust, stimulate internal humidity control, reduce heat loss and cushion noise. In the home, approximately 10–20% of heat is lost through



8.10 Wool usage in the textile world. (Source: CSIRO.)

the floor. Wool carpets insulate the home and can save an average of 11.3% on domestic energy consumption.

- Equip buildings with wool interiors and…

provide improved fire safety and air quality. Wool is naturally flame resistant, does not melt or drip and emits less smoke and toxic gases than any other commonly used fibre. In addition, its complex cell structure provides a natural chemistry that will clean the air and keep it purified for up to 30 years. Following the installation of wool in five buildings that had formaldehyde concentrations above the World Health Organization's recommended level of 0.05 parts per million (ppm), German researchers recorded that the level in all five buildings had dropped below that level within 24 h.

- Use more wool in bedding and…

enjoy 25% deeper regenerative sleep and improved fire safety properties. Recent studies show that when sleeping under a wool quilt, the heart rate

Table 8.7 World sheep population by country

	2007
China	171 961 000
Australia	85 711 000
CIS	72 421 000
India	64 269 000
Iran	52 220 000
Sudan	49 000 000
New Zealand	39 122 000
United Kingdom	33 946 000
Pakistan	26 500 000
Turkey	25 400 000
Nigeria	23 994 000
Ethiopia	23 700 000
Spain	21 847 000
South Africa	21 275 000
Syria	21 000 000
Algeria	19 500 000
Morocco	17 250 000
Argentina	15 880 000
Brazil	15 600 000
Peru	15 000 000
Mongolia	14 845 000
Somalia	13 100 000
Uruguay	11 000 000
Afghanistan	10 000 000
Greece	8 803 000
France	8 499 000
Italy	8 227 000
Romania	7 678 000
Others	199 398 000
Total	1 097 108 000

Note: Includes woolled and non-woolled sheep. CIS, the Commonwealth of Independent States.
Source: FAO/The Woolmark Company.

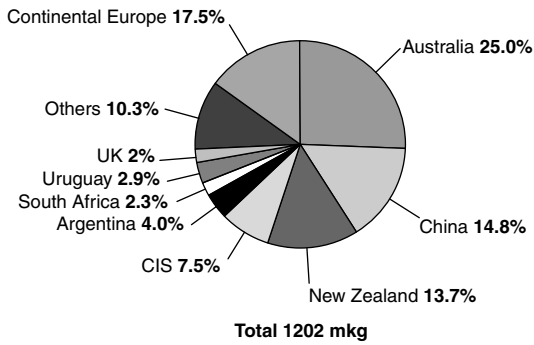
will be statistically lower for the whole sleep period, providing a more rested condition.

- Ensure wool is used in curtains and upholstery to…

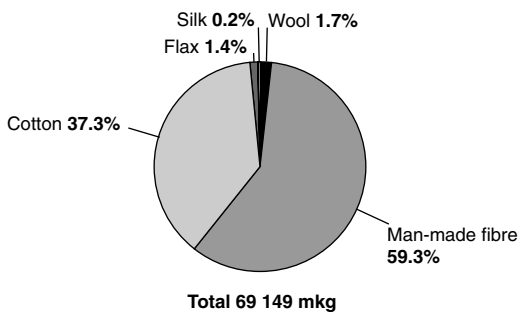
reduce excessive sound transfer, which causes noise pollution; provide additional protection against the hazard of fire posed by man-made fabrics in soft furnishings; and further improve internal humidity control.

- Support the wool industry in protecting the word 'wool' from being abused and…

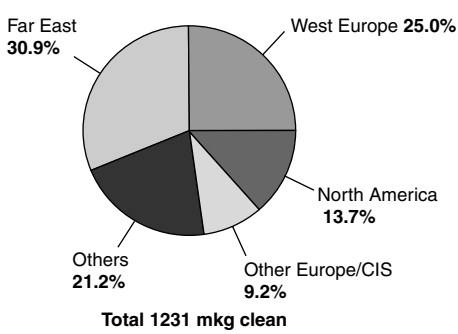
ensure that the rights of every consumer choosing to buy wool are protected, because only real wool must be allowed to be marketed with this valuable description.



8.11 World wool production: clean 2007 (% share). (Source: IWTO.)



8.12 World fibre production: 2007 (% share). (Source: IWTO.)



8.13 Virgin wool consumption at retail stage 2007. (Source: IWTO.)

8.7 Branding and consumer friendliness

The wool industry supports accurate and safe consumer rights to ensure protection against false and misleading statements and branding. A detailed, consumer-friendly system of identification and traceability of wool products helps to build consumer confidence. However, support from governments and the respective regulatory bodies is necessary to protect this important benefit. Wool has numerous natural attributes that make it one of the safest and most comfortable fibres to wear.

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9

Mohair, cashmere and other animal hair fibres

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Abstract: Although luxury animal fibres, excluding silk, represent far less than 0.1% of global fibre production, they play a very significant role in the luxury, high-value-added end of the market, notably the apparel market, being renowned for their special and mostly unique features, such as comfort and softness. This chapter covers the production, properties, processing and end-uses of the various luxury animal fibres, with the exclusion of silk, with the main focus on the down (undercoat) fibres of those animals with two fibre coats.

Key words: luxury animal fibres, mohair, camel, yak, musk-ox, cashmere, Angora rabbit hair, alpaca, guanaco, vicuña, llama.

9.1 Introduction

This chapter covers the various animal hair fibres that are generally referred to as luxury fibres, excluding the wool from sheep and also silk (which are described in Chapter 7, volume 1: Silk fibres, and Chapter 8, volume 1: Wool fibres). These luxury fibres have been discussed in two previous books[1,2] to which the reader is referred for more information and a more detailed list of references. The fibres considered here represent considerably less than 0.1% of global fibre production. Therefore, in terms of production volumes, they are insignificant. Nevertheless, they are highly sought after and play a significant role in the luxury, high value-added end of the market, notably the apparel market. Most of these fibres are produced by animals which inhabit inhospitable mountainous regions, covering a range of altitudes and extreme climates, where a highly insulating and protective fibrous coat (mostly a double coat) is essential for survival. The hair and fibres tend to be medullated, which combines good insulation with lightness. Due to the extremes of temperature they encounter, most of these animals have developed two distinctly different coats: (i) an outer coat consisting of coarse medullated guard hairs, produced by the primary follicles, which offers protection from the sun, rain and dust, and (ii) a finer and shorter down hair or fibre (undercoat or inner coat), produced by the secondary follicles, which provides outstanding insulation against extremes in temperature. The only exceptions are the Angora goat, alpaca and vicuña which, like sheep, are essentially single coated,

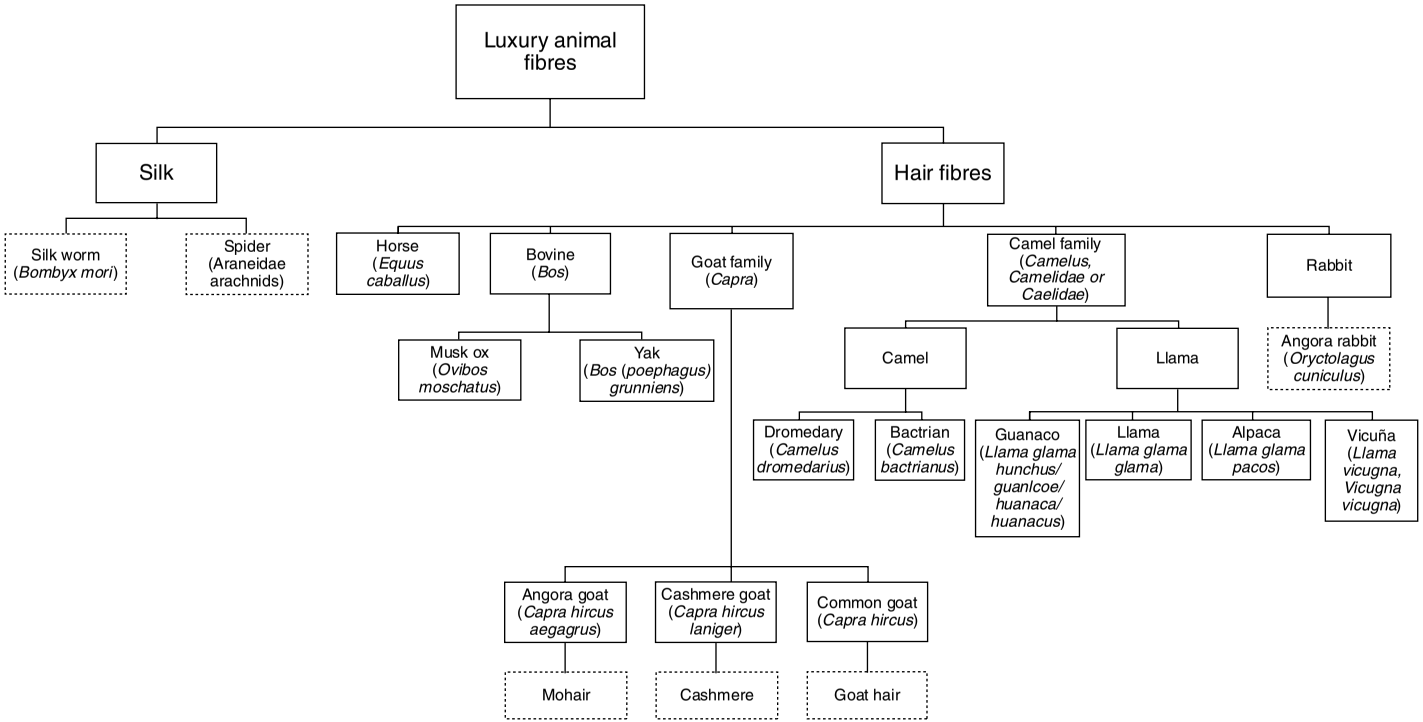
although still containing a combination of secondary and primary follicle fibres. In some cases, e.g. llama, there is also a group of intermediate fibres. For a given species, the younger the animal, the higher the altitude and the sparser the food the finer the fibre generally. The nature of their environment also largely determines the natural colour of animal fur,[3] for camouflage and other purposes; for example, the white winter fur of arctic animals, the grey-brown fur of forest and marshland animals and the yellowish fur of animals living in sandy areas.[3,4]

The fine undercoat (down) fibres, of two-coated animals, which are generally shed during spring, are the most valuable from a textile perspective, because of their combination of fineness, softness, lightness and good thermal insulation, and need to be separated from the undesirable coarse guard hair, either by hand or mechanically, a process called de-hairing. The finer the fibre and the lower the percentage of coarse fibres (guard hair) remaining in the fine component after de-hairing, the better the textile quality and value of the fibre. In this respect, successful de-hairing is largely dependent upon the differences between the two components in terms of fibre diameter/rigidity/linear density, friction and length and inter-fibre cohesion. Townend *et al.*[5] and Algaa and Mägel[6] have dealt with the de-hairing of luxury animal hair fibres.

This chapter focuses on the down fibres (fine undercoat) of those animals with two coats, the coarser guard hair (outer coat/hair) not being dealt with, except in a few isolated cases, and then only briefly. The ASTM D 2816 defines coarse hair as hair or fibres coarser than 30 μm, the criterion often used to differentiate between guard hair (outer coat) and down (undercoat).

Essentially, the main speciality animal hair fibres covered can be grouped into the following three main families or groups (see Fig. 9.1):

- Goat (*Capra hircus*)
  - Cashmere (*Capra hircus laniger*)
  - Angora (*Capra hircus aegagrus*)
  - Cashgora
- Camel (*Camelid/Camelus*)
  - Alpaca (*Lama pacos*)
  - Bactrian camel (*Camelus bactrianus*)
  - Guanaco (*Lama hunchus* or *Lama guanicoe*)
  - Llama (*Lama glama*)
  - Vicuña (*Vicugna vicugna*)
- Bovine (*Bos*)
  - Musk-ox (*Ovibos moschatus*)
  - Yak (*Bos (poephagus) grunniens*)



9.1 Main luxury animal fibre groups.

Angora rabbits (*Oryctolagus cuniculus*) do not belong to any of the above family groupings.

The South American Camelid (Camelidae) family consists of four species, three, namely llama (*Lama glama*), guanaco (*Lama hunchus* or *Lama guanicoe*) and alpaca (*Lama pacos*), representing the genus *Lama* and the fourth, vicuña (*Vicugna vicugna*), being a separate species.[7] They are now largely found in the Southern Andes, centred mainly on Peru's Altiplano region, known as the 'puna', a flat and rather barren terrain some 4600 m above sea level. There are also hybrids of the llama and alpaca. According to Greaves and Rainsford[7] the origins of the Camelid family (alpaca, guanaco, llama and vicuña) are not very clear, it being generally agreed that the two domesticated breeds, namely alpaca and llama, descended from the guanaco. It is also fairly widely believed that the alpaca originated as a hybrid between the llama and guanaco,[7] occurring after the Spanish Conquest of Peru in the early 1530s. According to Peruvian Andes archaeological sites, evidence suggests that the domestication of Camelids occurred between 7500 and 12 000 years ago.[7]

Table 9.1 compares the fibre characteristics and production figures for the different fibre types. It should be noted, however, that figures quoted in

Table 9.1 Approximate commercial fibre properties and production*

Fibre	Mean fibre diameter range (μm)	Typical (average) mean fibre diameter (μm)	Processed mean fibre length range (mm)	Typical processed mean fibre length (mm)	Raw fibre production per animal (kg/year)	Global raw fibre production (tonnes/year)
Alpaca	20–35	27	70–80	75	2– 4	6500
Angora rabbit	10–18	13	30–60	45	0.8–1.2	4000
Camel	15–25	18	30–70	50	2–5	1000
Cashgora	18–23	20	30–60	55	1– 2	
Cashmere	13–19	15.5	20–50	35	0.1–0.4	15 000†
Guanaco	13–18	15	20–50	35	0.3–0.9	10
Llama	20–32	28	50–80	65	1.5–3.5	1000
Mohair	22–42	30	50–110	80	2–6	5300
Musk-ox	12–20	16	40–80	60	2– 3	3
Vicuña	11–14	12.5	20–40	30	0.2	5.5
Yak	15–25	20	25–50	35	0.3–1.3	7000

* Compiled from various sources, with inputs also from:
Alpha Tops SA, Incalpaca TPX (Textiles Peruanos de Exportación), Seal International, Dr B. McGregor: Deakin University.
† ≈ 5 million kg dehaired.

Table 9.2 The composition of raw whole fleeces[8]

Fibre	Moisture (%)	Grease (%)	Water solubles (%)
Wool	11.0–11.7	9.5–27.0	3.9–7.1
Mohair	12.0–14.4	1.2–8.0	1.8–4.2
Australian cashmere	10.7–13.9	0.7–2.5	1.2–3.5
Chinese cashmere	11.1–12.9	5.0–7.2	2.3–3.0
Cashgora	13.2	1.2–2.8	0.6
Llama	12.0	2.8	–
Alpaca	10.9–14.4	2.8–3.9	0.6–2.4
Camel	9.9	0.5–1.1	–
Yak	10.4	12.3	–

the literature often do not specify whether they refer to greasy, raw, whole fleece or down fibres, or to staple or fibre length, etc. Tucker *et al.*[8] presented the composition of the whole greasy (raw) fleeces of various speciality fibres (see Table 9.2).

Most luxury or speciality animal fibres tend to be finer, less crimped and smoother than wool, their cuticular scales also being less pronounced (flatter or thinner), typically 0.4 μm thicker or even thinner, compared to wool which is generally 0.6 μm and thicker (typically about 0.8 μm).[9] The scales are also more widely spaced. Where crimp is present, it is generally not as pronounced or of as high a frequency as that of fine wools and in some cases is better described as a curling or waviness. Fibre cohesion and friction are consequently lower than for wool, requiring special conditions, or blending with other fibres, such as wool, for acceptable mechanical processing performance and yarn quality. After de-hairing, the down fibres are generally processed into yarn following either the worsted route for the longer fibres or the woollen route for the shorter fibres. Mechanical processing, although mostly done on machinery similar to that used for wool, needs to be adapted and optimised to suit the specific requirements of each of these fibres. Most of the expert knowledge to do so is held as a closely guarded secret by the various companies which process these speciality fibres. This also applies to the de-hairing process, which depends, amongst other things, on the differences in length and diameter of the respective fibre populations, as well as the percentage of down fibres present.

Chemically, these fibres belong to the same protein (keratin) family of fibres as wool, although their fine structures (morphological) and surface structures do differ, they also often being medullated. Table 9.3[10] gives the chemical (amino acid) compositions of some of these fibres. Because of their similarities in chemical composition, it is not easy to differentiate between them chemically. Ways in which they can be differentiated are covered in Chapter 11.

Table 9.3 Diameter and amino acid composition (mol.%) of speciality animal fibres[10]

Amino acid	Aust. cashmere*	Chinese cashmere	Aust. cashgora†	Mongolian yak	Camel	Wool	Guanaco	White alpaca	Black alpaca	Llama	Vicuña
Diameter (μm)	12.7–17.9	17.1±2.4	16.2±3.3	18.4±1.9	18.7±2.6	17.1	13.9±1.7	26.4±6.0	40.7±9.8	19.5±2.6	12.3±1.7
Amino acid											
Cysteic acid	0.1–0.2	0.1	0.1	0.4	0.3	–	0.5	0.2	0.6	0.4	0.5
Aspartic acid	6.6–7.1	6.7	7.1	6.6	8.1	6.9	7.2	7.3	6.9	7.2	7.1
Threonine	6.6–7.3	7.0	6.9	6.5	6.6	6.8	6.5	6.3	6.2	7.0	6.4
Serine	10.7–12.7	10.9	11.5	10.3	10.4	12.0	11.1	9.6	10.3	11.3	10.6
Glutamic acid	11.2–13.0	13.0	13.5	12.5	13.6	12.8	13.7	14.6	14.0	16.0	14.3
Proline	8.1–9.0	7.7	7.5	7.5	7.2	8.1	7.9	7.6	7.8	8.4	7.9
Glycine	9.0–10.2	8.8	8.4	9.3	7.8	9.5	8.1	7.9	7.9	5.9	8.1
Alanine	5.8–6.2	5.5	5.7	5.8	5.9	5.8	5.5	5.6	5.4	6.8	5.5
Cystine	4.2–5.6	5.5	4.8	5.4	4.6	4.6	6.0	6.0	7.6	6.3	5.9
Valine	5.0–5.7	5.7	6.0	5.9	5.9	5.2	5.8	6.0	5.9	6.3	6.1
Methionine	0.3–0.5	0.4	0.4	0.5	0.7	0.5	0.5	0.4	0.5	0.5	0.4
Isoleucine	2.6–3.0	3.1	3.2	3.4	3.3	2.8	3.0	3.2	3.0	3.3	3.0
Leucine	7.4–8.4	7.4	7.7	8.0	7.7	7.9	7.2	7.8	7.2	8.3	7.5
Tyrosine	3.4–4.1	4.1	3.5	3.5	3.3	4.0	2.9	2.8	2.6	2.8	2.6
Phenylalanine	2.6–3.0	2.9	2.8	3.2	3.0	2.7	3.1	3.0	2.5	3.2	2.6
Lysine	2.5–3.0	2.8	2.8	3.0	2.7	2.9	2.5	2.8	2.6	2.9	2.7
Histidine	0.6–0.8	0.8	0.6	1.0	0.8	0.9	0.8	0.9	1.0	1.0	1.0
Arginine	6.4–7.2	7.5	7.4	7.5	8.0	6.7	7.7	7.9	8.2	8.7	7.7

* 10 samples from individual goats.
† Sample from one goat.

These hair fibres are mostly processed, dyed and finished using similar machinery to that for wool, but the settings and the conditions (e.g. dyeing and finishing recipes, etc.) are adjusted to suit the specific characteristics, notably length and fineness, of each fibre. Because of the smooth and medullated nature of many of these speciality fibres, which differs from that of most apparel wools, dyeing recipes need to be adjusted from those used for wool, if a specific dye shade is to be achieved. Great care is also taken in practice to select the dyeing and finishing conditions, such as treatment time, temperature and pH, so that the desirable characteristics of the fibres, for example lustre and softness, are not deleteriously affected.

9.2 Alpaca

9.2.1 Fibre production, harvesting and properties

The ancient Incas already treasured the alpacas for their fine and soft fleeces which were reserved for royalty. After the Spanish Conquest of the Incas in the sixteenth century, alpacas were banished from lowland pastures to the hostile highland terraces of Peru,[11] their numbers decreasing greatly. Alpaca (*Lama pacos* or *Lama glama pacos*) from the genus *Lama*, descended from the Guanaca, is a member of the Camelidae family and mainly inhabits the 'Altiplano', a vast high, arid plateau of southern Peru, Bolivia, Chile and Argentina (South American Andes) at altitudes around 4000–5000 m,[12] most (close on 90%) being found in Peru. Alpacas (Fig. 9.2) are also found in significant numbers in other countries, such as the USA, Australia and Europe.

Alpacas are ruminants (or pseudo-ruminants), the only member of the Camelid family bred specifically for their fibres, being smaller than llamas but larger than vicuñas. They thrive on ichu (ychu) grass and at high altitudes. Originally shearing (mainly hand shearing), took place biennially, but now annually during the summer rainy season, from November to April, which is also their breeding period. In the case of alpaca, there is a less distinct difference between the fibre diameter of the outer coat and the undercoat (down). There are, however, essentially two types of alpaca, namely the relatively rare Suri which has long (some 140–170 mm^2),[13] straight (or slightly wavy) and silky (lustrous) hair which tends to be finer, with fewer coarse fibres (coarser than 35 or 30 μm),[14] and more valuable than that of the second type, namely the bigger and heavier Huacaya (Wakaya/Wayko), which has some 90–110 hair/mm^2.[13] The coat of the latter consists of compact and highly crimped fibres, the Huacaya accounting for close to 90% of the alpaca population and fibre production.

The average length of alpaca fibre ranges broadly from about 125 to over 200 mm (it grows about 100–125 mm per year), and the individual fibre

9.2 Group of alpacas. (Source: Inca Tops, SA, Arequipa, Peru.)

diameters range from about 10–75 μm. Alpaca fibres tend to be lustrous and are mostly medullated, elliptical in cross-section and pigmented. The alpaca fleece average diameter varies from about 17 to 35 μm (more typically 25–35 μm), with an overall average of around 26/27 μm[7] (CV from 25 to 35%). Alpaca fibres have become coarser over time, recently less than 10% being around 22.5 μm (20%, 20–22.5 μm),[5] 40% around 26.5 μm (35%, 24.5–26 μm),[15] 20% about 31 μm and more than 30% around 34 μm and coarser.[16] 45–50% coarser than 31 μm.[15] Some findings[7] on mummified remains indicate that 1000 years ago alpaca fibres may have been some 10 μm finer, and more even and less hairy than today, ranging from 14 to 21 μm, and mostly around 18 μm.[16] Nevertheless, according to Greaves and Rainsford[7] the fineness and related quality of alpaca fibres have not changed significantly from 1970 to 2005.

Alpaca has a variety of natural colours, including white, cream, light and dark fawns, piebald, greys and blacks, the lighter the colour the more valuable the fibre, grey being particularly popular. Most alpaca is either pure white or flecked with a few brown or black hairs,[17] the balance being sorted into more than 20 shades, ranging from light fawn to black,[17] sorting being by hand, also according to fineness.

Today alpaca fibre is mainly produced in Peru and also in significant quantities in Bolivia, Chile and Europe. It was estimated that in 2007 there were some 4.5 million alpacas in Peru, mainly being farmed by alpaca farmers (Alpagueros) in the Altiplano region bordering the city of Puno, 3871 m

above sea level in the south of Peru,[11] Arequipa, being the centre of Peru's alpaca trade and processing centre.[11] Covered shelters built in Peru for alpacas significantly reduced the mortality rate of baby alpacas, which can be around 40% due to respiratory and digestive illnesses under normal cold conditions, increasing to 80 or even 100% under severe cold conditions.[18] Alpacas have a productive life of up to 20 years, adults weighing between about 55 and 70 kg, and measuring about 1–1.1 m in height (up to the shoulder).[19] Peru, in around 2007, embarked on an important alpaca genetic improvement project (including DNA markers, artificial insemination and embryo transplants), recognising that 95% of alpaca farmers are small breeders, without an awareness of, or access to, modern technology and genetic improvement technologies.[16]

Huarizo, which resembles llama and produces coarser hair than the alpaca, is the term used in Peru for the hybrid produced by crossing a male llama with a female alpaca, the reverse crossing (i.e. female llama with a male alpaca) being referred to as Misti,[7] whose fleeces are not of the same quality as those from alpaca. When referring to the fibre *per se*, the term Huarizo is applied to alpaca fibre ranging from 29.1 to 31.5 μm, this quality tending to have a relatively broad medulla.[7] Crossing an alpaca male with a vicuña female produces the paco-vicuña, and the reverse cross the vicuña-paco.

Indications are that alpaca grown at high altitudes suffer considerable weathering damage, particularly to their tips, partly due to higher UV irradiation at such levels,[20] and the damage is more severe when the animals are only shorn every second year. The UV range from 290 to 310 nm causes the most damage by far to the wool fibre, the intensity of UV radiation increasing with increasing altitude.

Alpacas are now shorn, usually by hand, rarely by machine, annually (less often only every 18–24 months for combing, producing a staple length of over 200 mm), producing some 3 kg of fibre. Australian alpacas reportedly produce 6–8 kg of greasy fleece annually.[21] The lack of grease (lanolin) on the fleece necessitates oiling of the shears. The fibre yield of a fleece is around 85–90%, the raw 'white' alpaca fleece tending to be greyish in colour due to contaminants, such as dust, suint and grease;[22] some typical values are given in Table 9.4.[20,22]

According to Valbonesi[14] (quoting various references), both alpaca and llama fibre diameter (fineness) is affected by the age, sex, fleece colour and position on the body of the animal, nutritional conditions, period of the year, herd and/or origin. The diameter of alpaca fibres increases with age (up to an age of about 4 years[23]), for example from 21 μm at 10 months of age to 25 μm at 4 years of age,[24] the CV being around 28%. Finer fleeces have a lower CV (e.g. 18% for 22 μm) than coarser fleeces (e.g. 26% for 36 μm), the fibres from the upper part of the body tending to be finer

Table 9.4 Typical values for greasy alpaca fleeces[20–22]

Fibre	Grease (%)	Dust/vegetable matter (%)	Suint (%)	Yield (%)
Alpaca (Huacayo)	1–3	3–10	≈ 1	87–94
Wool (Med. Cross-bred)	15–30	5–20	4–7	43–57
Mohair (Cape)	1–8	5–10	2–4	78–92

than those from the lower part of the body,[20] with those from the rear part of the animal (britch area) invariably containing highly medullated, coarse and stiff fibres, similar to kemp fibres in cross-bred wool. Fibre length variation within a fleece is considerable (CV >40%). McGregor and Butler[25] found that, for Australian alpacas, the Suri produced fleeces (midside saddle fleece samples) 2 μm coarser than the Huacayas, with mean fibre diameter increasing with both the age (up to 7 years) and live weight of the animals, the CV of diameter ranging from 15% to 37% (average 24%), dropping from around 30% at 1 year of age to around 22% at 4 years of age.

Alpaca tends to have relatively more coarse fibres, e.g. coarser than 33 μm, than wool of the same mean diameter.[22] Alpaca fibres are generally medullated, the degree of medullation and non-circulatory (ellipticity ≈ 1.2) increasing with increasing fibre diameter,[22] the fibres becoming increasingly ovoid or kidney shaped as they become coarser. Fine alpaca has virtually no kemp, extra fine fibres (≈ 15–20 μm) being largely un-medullated, or have a fragmented medulla (≈ 20–30 μm), fine fibres (≈ 30 μm) having an interrupted medulla, medium fibres (≈ 40 μm) a continuous medulla and coarse fibres (≥ 60 μm) a broad medulla. Coarse alpaca, with a mean fibre diameter around 34 μm,[7] is strongly medullated, the medulla being slightly more than 50% of the fibre diameter.[7] Because of their medullae, the airflow method of fibre diameter measurement will underestimate the fibre diameter, the degree to which this happens depending upon the degree of medullation. More accurate results can be obtained by projection microscope, Laserscan and optical fibre diameter analyser (OFDA) based methods.

The ASTM standard specification (D2252-85, approved in 1991) for the fineness of different types of alpaca is given in Table 9.5.

Baby alpaca hair (≈ 22.5 μm) represents the finest hair from the first shearing, superfine alpaca (25.5/26 μm) forming a larger proportion of the production, also spanning the full range of more than 24 natural colours, coming from adult animals,[26] almost half of Peru's production being > 31 μm. The mean fibre diameter can range from as fine as 21 μm for Bolivian llama thampuls[27] to over 37 μm for Huacaya alpaca from Australia,[28] extra-fine (< 23.5 μm), fine (23.1–26.5 μm), coarse (> 31.5 μm) and inferior (> 30 μm) being some categories used.

Table 9.5 Fineness specifications for types of alpaca

Type	Description	Average diameter (μm)
T Extra	–	< 22.0
T	Tui, 12 months of age	22.00–24.99
X	Extra fine adult	22.00–24.99
AA	Medium adult	25.00–29.99
A	Coarse	30.00–35.99
SK	Skirtings	> 30.00
LP	Locks and pieces	> 30.00

Source: Standard specification for fineness of Types of Alpaca (ASTM-D2252–85, approved 1991).

In Peru, the Peruvian Technical Norm NTP 231:301 (Classified Alpaca Fibre), published in 2004,[7] defines the qualities of alpaca fibre as follows:

Baby alpaca: ≈ 23 μm
Superfine alpaca: 23.1 ≈ 26.5 μm
Huarizo alpaca: 29.1 ≈ 31 μm
Coarse alpaca: ≈ 31.5 μm

In Australia, alpaca fibre is classed into five diameter categories, five length categories and six colour categories (see Table 9.6[21]).

Pepper[29] has given the alpaca fibre classification shown in Table 9.7.

Other alpaca grades encountered include:

Inferior: > 39 μm
Super Fine: ≈ 26 μm
Baby: ≈ 22 μm

and

Coarse: > 31.5 μm
Fine: 23.1 – 26.5 μm
Extra Fine: ≤ 23.5 μm

Alpaca tops and yarn are classified as follows:

Baby (22.5 μm)
Fine Spinning
Adult (34 μm)
Suri (26/27 μm)
Superfine (26 μm)
Coarse alpaca (31–34 μm)

Table 9.6 Australian alpaca diameter, length and colour classing lines[21]

Fibre diameter	Length	Colour
Superfine (SF) < 20 μm	A: 120–150 mm	White (W)
Fine (F) 20–23 μm	B: 80–120 mm	Fawn (F)
Medium (M) 23–26 μm	C: 60–80 mm	Brown [light brown (BR), dark brown (DKBR)]
Strong (S) 26–30 μm	D: < 60 mm	Black (BLK)
Extra Strong (XS) >30 μm	O: (overgrown) > 150 mm	Rose grey/roan (RG), Grey (G)

Table 9.7 Alpaca fibre classification[29]

Type	Name	Average fineness (μm)	Minimum average length (mm)
Michell alpaca fibre classification			
BL	Baby	22.5	55
SU	Suri	26.5	68
FS	Fleece	26.5	66
AG	Coarse	32.0	68
LL	Llama	28.0	60
Inca Tops' classification			
BA	Baby	22–23	55
SU	Suri	25.5–26	68
SF	Super Fine	25.5–28	66
AD	Adults	28–30	68–60

Alpaca fibres are smoother and have a lower resistance to compression[30] than wool. The mean scale height of alpaca fibres coarser than 19 μm is about 0.4 μm compared to that of 0.8 μm for wool fibres of similar diameter.[31] It is therefore hardly surprising that the directional frictional effect (DFE) is much lower[20,32] for alpaca (around 0.20 for Huacaya alpaca and 0.16 for Suri alpaca[20]) than for wool which is around 0.40.[32] The with-scale and against-scale coefficients of friction (μ) of Suri fibres were found to be 0.29 and 0.40, respectively, the corresponding values for Huacaya being 0.28 and 0.42, respectively,[20] and wool 0.15 and 0.32, respectively. The differential frictional effect (DFE) is 0.16, 0.20 and 0.36 for the fibres in the above order. The initial modulus of alpaca is higher than that of wool.[33] According to Liu and Wang[34] Alpaca shrinks more in the Aachen felting test than wool of similar diameter, possibly due to the higher cuticular scale frequencies of alpaca, even though the higher scale thickness and DFE of wool should be expected to cause a higher felting shrinkage.

Cuticular scale frequency for alpaca varies from around 10 to 11 per 100 μm,[30,34–38] which is similar to that for llama. The difference between the scale lengths of wool and alpaca fibres decreases as the fibres becomes finer.[20] Because of the similarities of the cuticular and other surface structures of llama and alpaca (Camelid) fibres, it was considered well nigh impossible to differentiate between them, particularly between Suri, Huacaya and llama fibres.[30,39] Nevertheless, Valbonesi *et al.*[14] found that cuticular scale frequency, together with fibre diameter, enabled Suri fleeces (fibres) to be accurately and reliably differentiated from Huacaya and llama fleeces, and also to differentiate between llama and Huacaya fleeces, but less reliably so. Medium to coarse alpaca fibres (23–35 μm) have no distinct ortho-para-cortex differentiation.[20]

The breaking strength of Suri alpaca is lower than that of Huacaya alpaca, both wet and dry,[20] the dry tenacity of Suri being similar to that of wool but significantly lower than that of Huacaya, while its wet tenacity is lower than those of Huacaya and wool which are similar. The dry elastic moduli of the three types of fibre (Suri, Huacaya and wool) are similar. In one study,[40] the wet breaking tenacity and stress at 30% extension of Huacaya fibres were found to be double that of Suri fibres, the extension at break being similar for the two fibres. The greater strength of the Huacaya fibres was attributed to its more ordered structure (higher crystallinity) in the alpaca helices making up the micro-fibrils as revealed by X-ray diffraction. Australian alpaca was found to have much lower fibre curvatures (crimp frequencies) than wool (or cashmere[21]) of similar diameter, curvature decreasing with increasing fibre diameter[41] from about 50°/mm at 15 μm to about 15°/mm at 40 μm, the corresponding values for wool being 125°/mm and 58°/mm, respectively.[41] Fibre crimp (curvature) decreases gradually from the scoured state to the top.[41]

The complete amino acid composition of alpaca fibres has been determined. It varies widely between animals, due to factors such as nutrition, climate-induced stress and genetics.[22] Cystine plays an important role in the molecular architecture of a fibrous protein, the level affecting fibre and yarn properties.[22] Table 9.8 compares the levels of cystine for wool and alpaca.[22]

Table 9.8 A comparison of the cystine levels for wool and alpaca[22]

Fibre	Cystine content (mmol/kg)
Huacaya[20]	950
Suri[20]	1250
Wool[42]	900

The lipid component of the cell membrane complex (CMC) of different animal species differed, for example the CMC lipids of alpaca and llama fibres do not contain any sterols (e.g. cholesterol).[22,43] The amount of extractable CMC-lipid in alpaca fibre was found to be 2.6% by weight,[43] which was significantly higher than that of the other animal fibres examined.

9.2.2 Fibre processing

Alpaca hair is hand-sorted according to fineness and colour and then scoured on a scouring line similar to that used for wool (e.g. 5 bowls), the clean yield being around 85–90%.[2] Alpaca fleece contains up to 10% coarse fibres which need to be removed by de-hairing,[22] which takes place after scouring. Little information is available, i.e. public knowledge, concerning the processing, including de-hairing, of alpaca, since this is kept secret by the companies processing the fibre. The less distinct difference between the diameter of the outer coat and the undercoat makes effective de-hairing more difficult.[44] Wang *et al.*[21] investigated the technical feasibility of de-hairing alpaca using a prototype cashmere de-hairing machine, the de-hairing efficiency being assessed by the OFDA measured fibre diameter distribution, with changes in fibre length used to assess fibre damage during de-hairing. De-hairing of Alpaca is not as effective as that of cashmere, because of the smaller difference between the fine and coarse components in terms of diameter, stiffness and crimp.[21]

After de-hairing, mechanical processing, generally on the worsted system (rectilinear combing), sometimes on the woollen system, is similar to that for wool. Due to its smoothness and low fibre crimp, alpaca is difficult to process, as is the case with other speciality animal fibres, such as mohair. Blending with wool is common practice which improves cohesion and processing performance on modern wool processing machinery.[45] Wang *et al.*[45] found that blending alpaca with low crimp wool produced knitted fabrics which shrunk less than those produced from alpaca blended with high crimp wool, but the latter was softer. They concluded that high crimp wool may be preferable for blending with alpaca, particularly in terms of processing performance. Knitting yarns from 100% alpaca are typically around 16 Nm (≈ 60 tex) and weaving yarns around 28 Nm (≈ 35 tex),[46] or even 2/16 Nm (i.e. ≈ R120/2 tex resultant).

9.2.3 End-uses

Alpaca fabrics, particularly those containing the fine fibres, are generally considered to be soft, warm, light and luxurious. The medullated nature of the fibres suggests that these fibres will, on a weight for weight basis, provide

better thermal insulation than unmedullated wool, although the relative amount of air trapped within the fabric structure will also play a significant role. Alpaca is popularly used in knitted and woven apparel, primarily in knitwear, often brushed or raised, finding application in products such as ladies coats and skirts, suits, sports jackets, upholstery, pile fabrics for rugs and simulated furs, hand-knitting yarn, blankets, pullovers, cardigans, stoles and scarves.[12] White alpaca tends to be utilised in the knitting sector, dyeing to fashion shades, while the natural colours tend to be used in woven fabrics, particularly coating fabrics, which are often raised (teaselled). Baby alpaca is particularly used in ladies' accessories, such as scarves, stoles, throws and capes.[47] Alpaca is frequently blended with other natural fibres, notably wool and silk, even cotton, since price is an important factor. In blends with wool and mohair it is often used in overcoats. Alpaca is considered an ideal fibre for fancy yarns, such as bouclé and frisé variations,[26] bouclé, in pure alpaca, or alpaca blended with silk, mohair or Angora rabbit hair, being popular for knitted garments. Poorer quality alpaca is also often used as filling in quilts, duvets and pillows,[21] often in blends with wool, polyester or cotton, since it is desirable to remove coarse fibres and contaminants to avoid discomfort.[21] Alpaca and camel hair reportedly felt at a slower rate than cashmere.[48]

The International Alpaca Association (IAA), based in Peru, runs Alpaca Marks,[49] such as gold, silver and white alpaca which relate to fibre fineness and blend percentages. The Gold Mark applies to 100% alpaca, not coarser than 28 μm.[49] There is a Huarizo Mark for alpaca coarser than 28 μm, 31–34 μm representing coarse qualities. Peru's Ministry of Agriculture's National Council for South American Camelids the Consejo Nacional de Camélidos Sudamericanos (CONACS) launched a registered 'Alpaca Peru' Mark for all alpaca products produced by artisan knitters and weavers.[50]

Chemical bleaching of pigmented alpaca typically involves treatment with an iron salt (mordant) under acidic conditions, followed by the removal of the mordant not bound to the pigment and then bleaching the pigment with hydrogen peroxide, now that improvements in the second step have been researched.[51] Alpaca is used either in its natural shade (colour), or dyed to the required shade. Alpaca generally requires more dye than wool in order to achieve the same depth of shade, probably due to the medullated nature of the fibre. For more information the reader is referred to the references for more information.[2,21,22,25]

9.3 Angora rabbit hair

9.3.1 Fibre production, harvesting and properties

Angora fibre, from the Angora rabbit (*Oryctolagus cuniculus)* (see Fig. 9.3), is one of the lightest natural fibres due to its highly medullated (largely

9.3 Angora rabbit. (Reproduced from Pier Giuseppe Alvigini, *The Fibres Nearest the Sky*, Mondadori Editore, Verona, by kind permission of Mr Pier Alvigini at Alvigini S.A.S., 13900 Biella Via Dante, 12 Casella Postale 430, Italy.)

hollow) nature, making it ideal for thermal insulation and for maintaining a steady skin temperature, while still allowing the skin to breath.[52]

The good qualities of the hair of the Angora rabbit have been appreciated for many centuries, and it is the only rabbit bred solely for its hair. The Angora rabbit essentially has two coats of hair, namely guard hair which is coarse (up to 60 μm), long (20–100 mm) and spike like (spiky), offering protection against the rain and elements, and a shorter (10–40 mm), fine (12–14 μm) down, offering thermal insulation. Angora rabbit hair occurs in various colours, mainly white, but also grey, black-and-white and camel brown, although the main strain (albino) bred for its fibres produces white fibres only.[2] Raising Angora rabbits successfully is very labour intensive and highly skilled work. They are fairly resistant to most illnesses, provided their metabolism is not disturbed and is 100% functional,[53] in which case their resistance to diseases is reduced, and it is important to keep them under highly hygienic conditions.

The Angora rabbit reportedly originated from Ankara in Turkey, having been raised for over 200 years in Europe, reportedly being sighted in England as far back as 1708.[54] In France the numbers increased to such an extent that it was estimated that around 1860 some 10 000 kg of 'Angora Silk' was produced annually.[53] The rabbits were referred to as 'Silk Rabbits' or sometimes 'Combing Rabbits'.[53] Intensive farming with Angora rabbits took place in Western and Central France, and in 1935 some 90 000 kg of rabbit hair was being produced.[53] French Angora rabbits were introduced in Germany during the second half of the twentieth century where breeding expanded rapidly, as well as selective breeding of improved animals.[53] The French bred larger animals with more hair (better suited for fashion articles), while the Germans bred for finer and softer hair, the German rabbit mostly being shorn and the French rabbit 'plucked' (epilated).

There are essentially four types (strains) of Angora rabbits, namely 'French', 'English', 'German' and 'Chinese'. The 'French' type generally contains more guard hair (up to 14%) and is 'spikier', longer and more difficult to dye than the 'German' type (maximum of 3% of guard hair), and is more suitable for the production of a brushed appearance and fashion type garments. The 'German' type produces a softer product, being very suitable for, and popularly used in, various types of underwear and medical wear. German breeders wanted as fine and low a level of coarse hair (spike hair) as possible because it facilitates the spinning of fine yarn used in underwear. The English Angora rabbit is smaller than the German and French types and has hair of different colours which are very popular in home industries, where the natural colours are desirable. English Angora rabbits are normally grey, with black heads. In the early 1900s, China imported Angora rabbits, breeding rabbits for both their hair and meat, and in the mid-1960s became a major producer of Angora rabbit hair, mainly obtained from young animals (8–12 weeks old). The Chinese strain, developed in the early 1950s, accounts for the bulk of the Chinese and South American production of hair, the fibres produced generally being shorter and finer than those of the French and German Angora rabbits,[53] the Tanghang representing an improved strain.[53] Over time, many other countries introduced Angora rabbit farming. Today, most Angora rabbit hair is produced in China; China produced 70% of the global production of some 8 million kg towards the end of the 1980s.[53] In China, the rabbits are farmed on a highly intensive small-scale factory farm system by individual farmers. Significant quantities of hair are also produced in South America (e.g. Chile and Argentina), Europe (e.g. France) and in India.

The hair from Angora rabbits can be harvested by pulling/plucking, or combing by hand, or by shearing using either hand shears or electrical clippers. The animal is generally brushed prior to this so as to facilitate fibre harvesting and classing and the removal of any contaminants. When shearing in winter, it is necessary to leave a short length of fibre to protect the rabbit from severe cold. Animals are shorn (clipped) for the first time around 6–8 weeks of age to remove their 'nest' hair, and plucked for the first time around 100 days and then every 100 days thereafter. The fibre from the first shearing is not as valuable as that from the subsequent shearing,[53] but shearing at an early age encourages subsequent hair growth. In China the hair is mostly removed by hand plucking; the ripe or mature fibres (after 3 months of growth) which easily come out are mainly removed by this method. The French mainly use a method called 'epilation' (plucking or 'combing'), which involves the use of a type of 'comb' with a serrated edge to pluck the fibres four times annually (every 90 or so days). This produces a greater variation in fibre length than the hand plucking method. Plucking generally takes place when the hair is most easily removed (i.e. is 'ripe' or 'mature'), and tends to produce more 'spiky' hair than shearing. Both methods of

plucking encourage the growth of coarse hair, but slowly reduce the total hair production.[53] The Angora rabbit has around 180–200 hairs per mm^2 of skin, which can drop to between 100 and 120 after 4–5 pluckings. Shearing is popular in Germany. This method tends to produce 'double cut' waste and bits of cut skin which can cause problems during spinning and dyeing, but has advantages in that it subjects the rabbits to less pain and stress, only taking 10–20 min when using an electric clipper. This method produces hair with a greater proportion of fine hair, with very little seasonal difference, and only a difference of less than 10% between the summer and winter clip, compared to 20% with the combing method. The rabbits are generally shorn every 3 months, before the hair starts falling (shedding) which can cause felting (matting). Hand plucked hair, which contains only the 'ripe mature fibres', is considered a better quality than shorn hair, it also leaves the animal with a coat 25–35 mm long as a protection against the cold. The different methods of harvesting Angora rabbit hair and the comparative qualities have been described in detail.[53]

A healthy, good quality Angora rabbit produces about 1 kg of first grade hair per year. Shorn hair production can be as high as 1.5 kg per year (i.e. ≈ 380 g four times per year). Commercial yields of down hair vary between about 420 and 820 g a year in China and up to 1000 g in France and 1200 g in Germany,[2] the yield being affected by nutrition and the quality of the rabbit.

The Angora rabbit can be considered to produce essentially three kinds of hair,[2] the lengths referring to staple length:

Guide hairs: 100–110 mm long; they guide and cover the growth of the other hair.
Guard hairs: 80 mm long; these have rough points that lock together, lie over the down and seal it off.
Down: 60 mm long, with a diameter of about 14 μm, very smooth, with few cuticle scales.

Hair classification schemes

Angora rabbit hair is essentially graded according to its staple length and cleanliness, its appearance being of paramount importance. In certain cases the quality of the hair is specified quite independent of the source of the hair (e.g. whether from German or French Angora rabbits).

In Germany, the official classification and trading scheme, based upon DIN-60407, is as follows:[53]

1st Grade: Pure white, very clean, at least 6 cm long and not entangled (matted), without any double cuts.

2nd Grade: As for 1st Grade, but shorter than 6 cm, and longer than 3 cm.
3rd Grade: As for the above two grades but shorter than 3 cm.
Felted 1: Pure white, very clean but felted and matted.
Felted 2: White, felted and matted and dirty or with plant residues.

In France, the third grade is omitted, all hair with a staple length greater than 4 cm being included in the first grade. This allows for shorter intervals between harvesting.[53]

The following grades, into which Angora rabbit hair is sorted after grooming and where fleeces or part fleeces with lower quality fibre have been removed, have been given:[2]

Grade 1: Clean, free of felting, over 6 cm long (70% of the coat).
Grade 2: Clean, free of felting, under 6 cm but over 3 cm (15% of the coat).
Grade 3: Clean, felted, second cut.
Grade 4: All dirty, discoloured fibres.

The guard hair ranges from between about 20 and 100 μm, while the mean fibre diameter of the fine down ranges from about 9 to 14 μm, typically 12–14 μm, with the diameter of single fibres ranging from about 5–30 μm, 60% being in the range of 10–15 μm.[55] The fibre diameter distribution of French Angora rabbit hair is stated to be better described by a log-normal rather than a normal distribution,[56] the geometric mean, as opposed to the arithmetic mean, therefore being a more satisfactory representation of the 'true' mean fibre diameter, as determined by the evaluation of fibre cross-sections.[56] Stephanie and Wortmann[57] concluded that nutrition and climatic conditions, rather than differences in origin and quality, determined differences between the specimens they tested.

Mean fibre length of good grade Angora rabbit hair is around 45 mm, with a CV of around 50% and a mean fibre diameter of around 13 μm (CV = 25%). Number based mean fibre length, based upon the Zweigle Staple Sorting instrument, varies from around 16 mm for the poorer quality fibres to almost 53 mm for the top quality (super, pure white, unfelted 6 cm), the CV ranging from about 65%–90%, depending upon the source and quality of the hair. The number based short fibre content (≤ 10 mm) ranged from about 15%–45%, and for fibres ≤ 20 mm from about 25%–80%, the corresponding weight based values being 2–15% and 6–48%, respectively.[58] In some countries, such as South Africa, the percentage of hair with an average staple length shorter than 30 mm cannot exceed 10% in the better grades.

The cross-section of Angora rabbit hair is generally not circular,[59] occurring mostly as elliptical (ellipticity ≈ 4)[56], rectangular, square or bilobal.

Only a small percentage of the hair has a broken medulla or no medulla, the vast majority having one, or some even two or more medullae (channels).[59] The density of Angora rabbit hair can vary fairly widely, from about 0.95 to 1.3 (average between about 1.15 and 1.25 g/cm^3), depending upon the type and degree of medullation.[60] Blankenburg and Philippen[59] reported that the density of the hair they studied varied from about 1.1 to 1.2 g/cm^3, with an overall average density of 1.2 g/cm^3, with between 5 and 17% of the fibres they studied having no medulla, 83–89% having one medulla and 1–7% having two or more medullae (kemp), the corresponding values for fibre diameter being 9–11 μm, 12–14 μm, 13–37 μm, respectively, when measured on a projection microscope. In general, the hair from the female rabbit (doe) is stronger (30 kgf/mm^2) than that from the ram (buck), with that from the young rabbit being weaker than that from the adult,[53] winter hair tending to be weaker than summer hair. The extension at break of the hair lies between about 30 and 40%.[53] Angora rabbit hair scale height is of the order of 0.4 μm on average,[61] as is the case for other luxury animal fibres, such as cashmere and mohair. Angora hair felts less than wool in the felt ball density test.

Stephanie and Wortmann[62] gave values for the three different breeds of Angora rabbit hair which they tested (Tables 9.9 and 9.10).

Table 9.9 The percentage of medullated fibres in Angora rabbit hair from Germany, France and China[62]

	Percentage of fibres		
Fibre type	Germany	France	China
Unmedullated	33.1	22.3	12.2
Single medulla	66	74.7	85.1
Multiple medullae	0.9	2.9	2.9

Table 9.10 Average diameter (μm) of different types of Angora rabbit hair from Germany, France and China[62]

	Average fibre diameter (μm)		
Fibre type	Germany	France	China
All fibre types	13.4	18.7	12.2
Single medulla	14.1	19.0	12.2
Multiple medullae	31.3	42.5	31.5
Unmedullated	11.4	14.5	8.8

Table 9.11 Quality parameters for Angora rabbit hair from various origins[58]

Sample no.	Quality/grade	Source/origin	Fat/grease content (%)*	Vegetable matter content (%)†
1.	First grade, pure white, unfelted 6 cm	Czechoslovakia	0.8–1.0	< 0.1
2.	Second grade pure white, unfelted 3–6 cm	Czechoslovakia	0.8–1.0	< 0.1
3.	Third Grade, pure white, unfelted, 3 cm	Czechoslovakia	0.8–1.0	< 0.1
4.	Third grade, white, slightly felted and contaminated	Czechoslovakia	0.9–1.2	0.1–0.2
5.	Fourth grade, white, heavily felted and contaminated	Czechoslovakia	1.2–1.5	0.3–0.5
6.	Super choice	France	0.9–1.0	< 0.1
7.	Premium choice	France	0.9–1.0	< 0.1
8.	'tout Venant'	France	1.2–1.5	0.2–0.3
9.	Quality I	Argentina	0.6–0.7	< 0.1
10.	Quality II	Argentina	0.8–1.0	< 0.1
11.	Quality AB	Japan	1.1–1.2	< 0.1
12.	Quality B	Japan	1.1–1.2	< 0.1
13.	Quality 95%	China	1.2–1.3	0.4–0.6
14.	Quality I	Germany	0.6–0.7	< 0.1

* Methylene chloride extractable matter (IWTO-8–61).
† Boiled in 0.2N NaOH – solution for 15 min.

Fröhlich[58] listed the quality parameters for Angora rabbit hair (Table 9.11). He also gave average values for the chemical properties of the above fibres (Table 9.12).[58]

Gupta *et al.*[63] gave the properties of three Angora rabbit genotypes, German, British and Russian, as well as their crosses (Table 9.13). They also gave a comparative table for Angora rabbit hair and other fibres (Table 9.14).[63] Gupta *et al.*[64] gave the average values for the six genetic groups of rabbit hair which they tested (Table 9.15).

Table 9.16 compares the properties of Angora rabbit fibre (hair) with those of wool.[65] The comparative values (Table 9.17) for Angora rabbit hair and wool[66] have also been given for a selection of physical properties.

9.3.2 Fibre processing

Angora rabbit hair need not be scoured prior to processing, generally containing less than 1% of natural grease (generally 0.7–1.0%, sometimes even exceeding 1.2%, of fatty matter).[65] Dust and vegetable matter are often

Table 9.12 Average values for chemical and physical properties of Angora rabbit hair from various countries[58]

Property	Average value	CV (%)
Grease content (%)	1.0	–
Vegetable matter content (%)	<0.1	–
Cystine content (%)	13.6	1.5
Alkali solubility (%)	9.0	16
Acid solubility (%)	7.5	25
Urea bisulphite solubility (%)	65.3	4.2
Regain (65%RH)	13.7	1.2
Water retention (%)	52.9	8.4
Mean fibre diameter (μm)	12.8	5.7
Tenacity (kgf/mm²)		
Dry	19.9	12.4
Wet	17.8	10.8
Extension at break (%)		
Dry	33.1	5.0
Wet	45.9	2.0

RH, relative humidity; CV, coefficient of variation.

removed from the fleeces prior to sorting, by a process called grooming.[2] The fibre yield is some 97%, the hair containing very little contamination – only around 1–2% of hair scales and skin flakes. When hair is obtained from skin, blowing of the hair is the traditional way of cleaning it, i.e. removing skin pieces, etc. Due to its smooth fibre surface, relatively short length, lack of crimp and cohesion and greater propensity to generate static electricity, Angora rabbit hair presents more difficulty and waste during mechanical processing than wool; an antistatic lubricant (e.g. 0.3%), applied prior to carding, is essential if it is to be processed successfully.[65]

The properties and processing of Angora rabbit hair have been discussed,[65] it being stated that it was possible to process the hair on the worsted and woollen systems, economically and to the desired quality, provided special precautions and care were taken in terms of machinery fittings and adjustments. Spinning problems can arise due to the fibre surface characteristics, fineness, length and length distribution.[65] Because of the difficulties often experienced in spinning pure Angora hair, it is generally not spun in 100% form, being blended with wool (e.g. 50–90% of wool), and also nylon, with which it is highly compatible, a blend level of 15% rabbit hair, 75% wool and 10% nylon being popular. It is processed on either the woollen or worsted systems, depending on fibre length and blend. Blending with wool has a highly beneficial effect in terms of processing performance. Nevertheless, even then, special precautions, care and skill are required to successfully process the blends. Before mechanical processing, the fibre length variation (CV %) of Angora rabbit hair can vary from about 60 to 90%,[65] accompanied by a high short fibre content. The moisture content tends to be lower

Table 9.13 Properties of Angora rabbit hair[63]*

Genotype	Fibre fineness (μm)	Fibre length (mm)	Grease content (%)	Suint content (%)	UB† solubility (%)	Tenacity at break (gf/tex)	Elongation at break (%)
German Angora	13.5 ± 0.38 (50.6)	41.3 ± 0.45 (18.8)	0.58	2.65	59.0	14.8	39.7
British Angora	11.8 ± 0.33 (48.4)	45.5 ± 0.60 (25.2)	1.30	2.36	77.7	13.1	40.1
Russian Angora	11.8 ± 0.31 (45.3)	48.9 ± 0.74 (23.3)	0.79	2.57	64.1	14.9	39.2
German x British	11.6 ± 0.30 (44.9)	40.2 ± 0.51 (21.9)	0.62	2.18	66.0	14.7	40.6
German x Russian	12.5 ± 0.39 (54.1)	52.5 ± 0.85 (28.0)	0.61	–	61.1	13.2	38.8
German x (Russian x British)	11.6 ± 0.29	55.7 ± 0.82	0.79	2.15	68.5	14.0	36.2

* The CV values are given in brackets.
† UB - urea bisulphite.

Table 9.14 Physical and mechanical properties of various fibres[63]

Fibre	Fibre diameter (μm)	Linear density (tex)	Fibre length (mm)	Dry			Wet		
				Breaking load (gf)	Ext. (%)	Tenacity (gf/tex)	Breaking load (gf)	Ext. (%)	Tenacity (gf/tex)
Rabbit									
Fine hair	10.4	0.16	32.3	2.0	34.5	12.5	1.4	65.0	8.7
Guard hair	44.0	0.92	43.8	10.7	34.2	11.2	7.2	54.0	7.8
Cashmere									
Fine hair	10.9	0.26	30.5	3.2	32.5	12.3	2.7	59.6	10.4
Guard hair	48.4	1.67	50.7	19.4	34.0	11.6	13.6	58.0	8.1
Mohair	30.4	1.35	–	19.5	39.5	14.4	16.1	87.8	11.9
Rambouillet sheep wool	16.8	0.50	25.0	6.0	35.0	12.0	3.2	68.0	6.4
Avivastra sheep wool	21.5	0.87	45.1	9.0	29.7	10.3	5.2	54.0	6.0
Cotton (Coimbatore H-4)	12.0	0.17	28.0	5.0	5.2	29.0	6.6	6.3	37.0
Polyester	–	0.17	51.0	8.1	35.5	48.0	–	–	–
Acrylic	–	0.17	51.0	4.7	25.0	28.0	–	–	–

Table 9.15 Average values for six genetic groups of rabbit hair[64]

Property	Average value	Range
Fineness (tex)	0.18	0.14–0.23
Tenacity (gf/tex)	12.1	13.1–14.9
Extension at break (%)	39	36–40

Table 9.16 Mechanical and physical properties of Angora fibre and sheep's wool[65]

	Angora fibre	Sheep's wool
Fibre length (mm)	Approx. 15–60	20–250
Fibre fineness (μm)	12–17	16–40
Fibre cross-section (shape)	Rectangular	Circular
Fibre surface structure	Relatively smooth scales Very low crimp	Distinct scale sheath Good crimp
Relative density (g/cm^3)	1.1	1.32
Fibre tensile strength (kg/mm^2)	17–26	15–25
Wet fibre strength as % of dry	70–90	70–90
Breaking extension (%)	30–35	25–60
Wet fibre extension as % of dry	120–140	110–130
Regain at 21°C/6% RH (%)	13–15	15–17
Moisture retention (%)	45–55	40–45

Table 9.17 Comparative physical characteristics of rabbit hair and wool[66]

Property	First grade rabbit hair	Wool
Crimp/cm	2.7*	5.5
Breaking strength (gf)	2.8	10
Breaking elongation (%)	37	40
Coefficient of friction (μ)	*With scale*	*Against scale*
Rabbit hair on rabbit hair	0.21	0.49
Rabbit hair on wool	0.21	0.62
Wool on wool	0.5	0.80

(≤12%) and more variable than that of wool. A blend of Angora rabbit hair and wool generally produces more hairy yarns than pure wool.

On the woollen system, it is advisable for the fibre to have a moisture content (regain) of at least 14–15%, and a fatty matter content of around 0.6–0.8%[65] prior to carding, adequate time for conditioning being important. It is advisable to use, for blending, relatively fine wool (≈ 21 μm) with good crimp and not too long when processing on the woollen system.[65] Nm12 woollen spun yarn is fairly common, and it is advised[65] that the yarn should preferably have between 140 and 160 fibres in their cross-section. The use

of carbonised wool in the blend has some advantages because of the lack of contaminants in such fibres. The relatively high Angora fibre waste produced necessitates that at least 10% more Angora fibre be incorporated in the blend than that which has been specified. Excessive drafts during ring spinning need to be avoided, with a relatively high twist factor ($\alpha > 80$) being advisable. For the worsted system, it is advisable to use Angora with a staple length of at least 45–50 mm, with fibre fineness and length even more critical than for the woollen system. Fine pinning during gilling or drafting, precise drafting zone (nip) settings, low drafts and delivery speeds are important.[65] Mixing (blending) with wool generally takes place in sliver form. It is maintained[65] that provided all the necessary precautions are taken and processing settings and conditions optimised, it is possible to produce Angora/wool blend yarns on both the woollen and worsted systems which are of the same quality as the corresponding pure wool yarns. It is important to inspect the Angora blend yarns using an inspection (wrap) board, since this gives an indication of the visual appearance of the ultimate knitted garment. Onal and Korkmaz[67] have reported on the effect of fibre blend, yarn twist and fabric relaxation on the abrasion and pilling resistance of Angora rabbit hair blended knitted fabrics.

Until the early 1990s it was not possible to spin Angora rabbit hair on the conventional cotton spinning system,[55] but in more recent years it has also been processed on the short staple (i.e. cotton) system, mostly in blends with cotton, and then either ring-spun or rotor-spun (OE). Because of its length, processing on the cotton system has its benefits. Krishnan *et al*.[55] describe the conditions required to process Angora rabbit hair (38 mm) in blends with other fibres, such as cotton, viscose, wool, silk and acrylic, on the cotton system, ending in rotor (OE) spinning of hosiery yarn destined for knitting.

The same dyes used for wool can be used for Angora rabbit hair. Acid milling and 1:2 metal complex dyes are popularly used and the Chinese varieties are found[68] to have a greater dye affinity than other varieties. Wortmann *et al*.[69] found that blank dyeing of Angora rabbit hair yarns caused changes in the morphological structure of the medullated fibres, a significant proportion of the medulla cells starting to collapse at temperatures around 70ºC, where the extent of the collapse depends upon the stress on the fibre in the yarn that is released due to a softening process in the keratin. Fibres with a 'ladder' type medulla passed through an intermediate stage where their medullae appear to be continuous before collapsing completely.

9.3.3 End-uses

Angora rabbit hair is very popular in knitwear and is used in both knitted and woven outerwear, ladies underwear, hosiery, gloves and knitted millinery and felt hats, although fibre shedding can sometimes present problems.

It is very popular in medical and thermal underwear, under-blankets in hospitals, nightwear and blankets, etc., particularly the hair from the German Angora rabbit. The hair from the French type is very popular in fashion wear (e.g. shawls).

The German textile industry was the greatest user of Angora rabbit hair during the latter part of the twentieth century, with popular end-uses there being medical and other types of underwear. In medical underwear,[53] it is considered to reduce muscular and other pains, attributed to its electrostatic, thermal and moisture absorption properties, the medullated and fine structures of the fibre resulting in it having excellent thermal insulation properties.

Angora rabbit hair knitted products can present serious fibre shedding problems, due to their low fibre friction, length and strength, together with the fact that low yarn twists are generally employed to maximise the softness of the garments. Zhaogeng and Bo[66] concluded that Angora rabbit hair shedding during wear was largely due to the fibres breaking rather than slipping, resulting from the low breaking strength of the hair. The low strength also results in serious fibre damage and breaking during carding, which can reduce the average fibre length by as much as 10 mm.

Additional information in relation to Angora fibre can be found in the references.[2, 53,65,70 and 71]

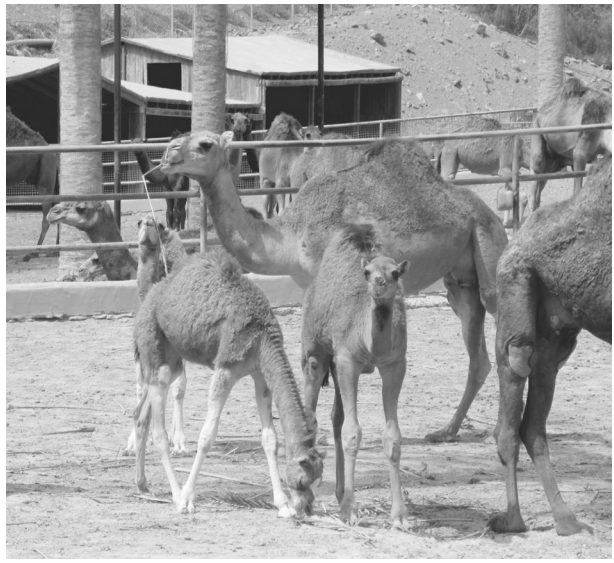
9.4 Camel

9.4.1 Fibre production, harvesting and properties

General

Camels belong to the Camelidae family, which has two genera,[72] *Camelus* and *Lama*. The genus *Camelus*, which is part of the Camelidae grey family,[2] is made up of two species: the one-humped Dromedary camel (*Camelus dromedarius*) (see Fig. 9.4), also referred to as the Syrian, European or 'Arabian camel', mainly from Arabia, Northern India and the Mediterranean, and the two-humped Bactrian camel (*Camelus bactrianus*) (see Fig. 9.5), also referred to as the Asiatic or Central Asian camel, mainly found in Northern China and Mongolia, in areas bordering the Gobi desert, and to some extent in other parts of Asia.[72] The latter produces the best and softest fibres. The finest quality comes from China, from young animals. It is thought to have descended from the cross-breeding of the ancient pure Bactrian (Bactriana) camel and the Dromedary camel, which produced the Bokhara type of camel, also known as the Boghdi.[70]

According to Harizi *et al.*,[72] there were about 14 million Dromedaries and 4 million Bactrian camels world-wide in 2002, with the greasy hair



9.4 One-humped Dromedary camel.

9.5 Bactrian camel. (Reproduced from Pier Giuseppe Alvigini, *The Fibres Nearest the Sky* (2nd ed. 1984), Mondadori Editore, Verona, by kind permission of Mr Pier Alvigini at Alvigini S.A.S., 13900 Biella Via Dante, 12 Casella Postale 430, Italy.)

production at the time being estimated[2] at 3–3.5 million kg, the bulk being from China and Mongolia. In the 1980s India reportedly[73] had 1.5 million camels (half Bikaneri bred), representing some 10% of the world camel population.

Camel hair used in textile applications almost solely comes from the Central Asian or Bactrian camel, the hair of the one-humped Dromedary

generally being not important for textile applications because of its relative coarseness and inadequate length and strength.[74] The Bactrian camel yields a light fawn (tan) lustrous fleece mixed with coarser brown hairs. It has a double coat of hair, with the coarse outer hair or guard hair, 30–120 μm in diameter and from 60 to 375 mm in length, and the fine down or undercoat, 10–30 μm, or even 40 μm (average about 18 μm)[75] in diameter (CV from about 25% to 35%[76]) and about 25–125 mm (typically 50 mm, CV ≈ 24%), in length,[74] and characteristically reddish brown (tan) in colour. Most camel hair is produced in China (including Tibet) and Mongolia, some also being produced in Afghanistan, Iran and Russia.

Bactrian camel

As already stated, the Bactrian camel is the main source of camel hair used in textiles. When the camel moults, it sheds its hair (fleece) in large tufts over a period of time (≈ 6–8 weeks). Starting during late spring, the neck hair falls off first, then the mane, and finally the body and belly covering,[77] the hair on the humps remaining. When the camels moult, the hair can obtained by combing, shearing (done more commonly today) and collecting of shed hair, resulting in a combination of coarse guard hair and fine down, referred to as raw (i.e. un-dehaired) camel hair. Raw camel hair contains approximately 75–85% fibre, 4–5% fat (grease) and 15–25% sand and dust.[78]

Today the camels are increasingly being shorn (clipped)[78] once a year, the hair on the back generally not being shorn, serving as a cushion for the pack or saddle. The hair is also not shorn too short, so as to leave sufficient fibre to protect the animal from excessive heat and cold.[78] The whole fleece weighs typically between 2.5 and 3.5 kg, while in cold regions, the hair yield can be as high as 5.5 kg, with the average annual yield being about 4 kg.[78] The Bikaneri camels (in India) produce some 700 g of hair per annum, with an average fibre diameter around 25 μm, some 15% of the fibres being completely medullated and 45% partially medullated.

Msahli *et al.*[79] (quoting Franck *et al.*) stated that the characteristics which determine the fibre quality of South American domestic camelids are mean fibre diameter, colour, type of fleece, fibre length and uniformity of diameter and length. The finest quality camel hair is a light, bright fawn while the lower qualities are deeper in colour.[80] There is a relationship between fibre diameter and fibre length.[81] Algaa and Mägel[81] review the properties of camel hair.

The surface of camel fibre is covered by a layer of fine and irregularly shaped scales, having diagonal edges which are not very prominent.[82] The fibres are circular to oval, the cortical layer exhibiting regular striations due to strings of pigment granules[82] which give the fibre its characteristic pale red-brown colour, and some fibres have a medulla which is often fragmented.[82]

Camel hair has a tensile strength of about 16 cN/tex and a moisture regain of 13%.[74] The finer fibres do not have a medulla, while the coarser fibres have a medulla which varies in character and size.[74] Table 9.18[82] compares the properties of camel hair with those of certain other fibres. Weng *et al*.[83] gave a table comparing camel hair fibre properties with those of 70s Australian wool (Table 9.19).

Table 9.18 Comparison of properties of camel hair and other fibres[82]

Properties	Camel hair	Silk	Wool	Cotton
Tenacity (gf/den)	2–2.5	1–1.5	1.5–2.0	2–5.5
Elongation (%)	39–40	25–40	25–45	6–10
Density (g/cm^3)	1.32	1.34–1.38	1.33	1.50–1.54
Moisture regain (%)	13.0	11.0	14–16	9
Acid resistance	Excellent	Excellent	Excellent	Bad
Alkali resistance	Bad	Good	Bad	Excellent
Resistance to moth/ fungus	Resistance to fungus but not to moth	Resistance to fungus but not to moth	Resistance to fungus but not to moth	Resistance to moth but not to fungus
UV resistance	Bad	Bad	Bad	Good

Table 9.19 The properties of camel hair and 70s Australian wool[83]*

Property	Camel hair	70s Australian wool
Average length (mm)	43	55
Average fineness (μm)	17.8	19.1
Fineness dispersion (%)	28.6	21.2
Absolute strength (cN)	4.9	5.7
Relative strength (cN/dtex)	1.47	1.53
Elongation at break	39.0	38.5
Initial modulus (cN/dtex)	20.4	15.3
Work of rupture (cN·mm)	13.8	16.2
Crimp factor (times/cm)	3–4	4–7
Crimp ratio (%)	5.7	7.8
Residual crimp ratio (%)	3.6	5.3
Crimp-elastic recovery percentage (%)	64.0	68.5
Compression elasticity (%)	60.0	57.4
Fast-elasticity (%)	33	33
Slow-elasticity (%)	27	23
Elasticity (%)	60	56
Milling ball density (g/cm^3)	0.13	0.18
Mass resistivity (Ω)	2.3×10^{13}	3.3×10^{13}
Moisture content (%)	13.9	13.3
Oil (grease) content (%)	1.3	13.8
Handle	Good	Fine
Lustre	Good	Good

*Values rounded off. 70s refers to the wool count, a measure of quality.

Dromedary camel

In Tunisia there were some 100 000 one-humped Dromedaries in 2003[72] (quoting Sghair) producing more than 100 000 kg of hair annually, only a little of which is harvested and used by Bedouin people to make traditional clothing, such as 'bernous' and 'wazra' worn by men.

The Dromedary camel also has a two-layer coat of hair, a coarse outer coat or guard hair (outer hair) and a fine undercoat or down.[79] The raw fibre obtained from the camel has to be de-haired in order to separate and remove the more valuable undercoat from the coarse guard hair. In general, the efficiency with which the two components can be separated during the de-hairing process depends upon how much the two components differ, particularly in terms of fibre length, fineness (diameter) and rigidity, as well as in their surface characteristics and inter-fibre cohesion:[79] the greater the differences, the greater the de-hairing efficiency generally.

Harizi *et al.*[72] evaluated and reported on the physical and mechanical properties of the under-hair (down) of Tunisian dromedaries. They found the scale frequencies for the under-hair and guard hair to be between 6 and 8 and 13 and 15 per 100 μm, respectively, compared to 6 and 7 for cashmere[72] (quoting Phan *et al.*). The scales of the Dromedary fibres are, on average, extremely long and quite visible, but do not protrude much from the fibre surface, appearing almost convex.[72] The colour of the hair varies from beige to brown to almost black.[72] The average fibre diameter is around 17.7 μm (CV = 27%) for the undercoat and 90 μm (CV = 28.5%) for the guard hair,[72] with an average regain of 15%,[72] which is slightly less than that for wool.[72] The average Almeter (AL100) fibre length (weight based) of the de-haired under-hair of a one-year-old Dromedary was found to be about 53 mm (CV = 41%), with 7.7% short fibres (< 15 mm), the hair being shorter than that of Mongolian camel (> 70 mm),[72] but longer than cashmere (≈ 44 mm).[72] Harizi *et al.* obtained a tensile tenacity of 212 MPa, an elongation at break of 37% and an initial modulus of 3.87 GPa for the under-hair (≈ 20 μm), the corresponding values for the guard hair (≈ 105 μm) being 121 MPa, 49.7% and 2.02 GPa, respectively. The average values for the under-hair were as follows:

Mean fibre diameter: 21.2 μm (35.9% CV)
Mean fibre length (Hauteur): 18.1 mm (CV = 53.3%)
Tenacity: 11 cN/tex (CV = 12%)
Strain: 37% (CV = 21.6%)

The average stress and strain were higher, and the modulus lower, than those of Merino wool, camel hair and cashmere.

Harizi *et al.*[72] found that de-haired fibre taken from the neck, chest, shoulders, nape of the neck, throat, tail and back (area 1) had, on average, similar

mechanical properties to those of fibre taken from the belly, side and kidney areas (area 2), but generally showed different trends with the age of the camel in terms of diameter and length. The average diameter of the fibres from area 1 was greater, also increasing more with age, than that of fibres from area 2. Fibre length increased with age for area 1 and decreased with age for area 2. Fibre bundle tenacity first decreased, and then increased with age, showing a minimum at about 8 years of age.[72]

9.4.2 Fibre processing

Bactrian camel hair

The processing of camel hair essentially consists of sorting, willowing (to remove dirt, dust and plant material), washing (scouring), de-hairing, spinning, fabric formation (knitting and weaving), dyeing and finishing. Sorting involves grouping the hair according to the fibre colour and the age of the animal, and visually separating the coarse hair from the fine, soft hair, after which the fibres can be willowed and then washed to remove any dirt or debris, which is followed by the de-hairing process.[82]

Raw (i.e. un-dehaired) camel hair from Mongolia contains between 20 and 35% guard hair and that from Iran between 50 and 65%,[81] and as much of this guard hair as possible has to be removed prior to textile processing. Although camel hair was originally de-haired fairly satisfactorily by carding and combing operations (even by hand in the nineteenth century) a mechanical de-hairing process, involving specially developed machinery, is more commonly applied today,[75] which is more efficient, slower, gentler and more sophisticated. Details of the de-hairing process are generally a closely guarded secret, with relatively few firms able to do it. De-hairing removes not only the coarse hair but also extraneous matter (contamination), such as vegetable matter and bits of skin (dandruff).[82] When combing is involved, the fine short fibre (i.e. noil) removed can often be of a higher commercial value than the tops.

De-haired down fibre diameter ranges from 16 to 20 μm, intermediate hair from 20 to 29 μm and guard hair from 30 to 120 μm.[2] Baby camel hair, which is the softest, has a diameter of about 16–17 μm. The mean fibre length of the down typically ranges from about 30 to 40 mm, with the guard hair being up to 375 mm long,[2] while the length of baby camel hair is similar to that of the adults.[2] Fine camel hair tops typically have a length of 50 mm and a CV ≈ 25%.

After de-hairing, camel hair is processed on both the woollen and worsted systems, often in blends with wool, sometimes also with nylon for hosiery and knitted products.[83] Weng *et al.*[83] concluded that the low cohesion of camel hair relative to that of 70s Australian wool was mainly responsible

for its relatively poor spinning performance, its low cohesion being mainly attributed to its relatively low crimp and friction and relatively high initial modulus. Its shorter fibre length and greater fibre diameter variability may also have contributed to its inferior spinning performance *vis-à-vis* that of the 70s Australian wool. The long and coarse guard or outer hair, such as from camel manes (Mongolia) and third grade Chinese camel, is carded, gilled and Noble combed (sometimes twice), the very long fibres often being reduced in length (broken) prior to combing.

Since, like most speciality animal fibres, camel hair occurs in nature in various shades of grey or brown, due to the presence of a natural pigment (melanin), a bleaching process is often employed to remove its natural colour. The camel hair undercoat is characteristically reddish brown, reacting to chemicals very much like mohair and cashmere.[75] For white or pastel shades, such fibres need to be bleached, ideally with minimal damage to the fibre. Bereck,[4] Khishigsuren *et al*.[84] and Kang and Park[85] have reviewed and reported on the bleaching of naturally melanin pigmented animal fibres. In practice, pigmented fibres are bleached using either oxidative or reductive processes or their combination, and commonly involves a treatment with ferrous salts (mordanting) followed by rinsing and bleaching with hydrogen peroxide.[84] Khishigsuren *et al*.[84,86] described an improved ferrous mordanting process for bleaching camel hair, which resolved serious problems of discoloration and excessive damage of bleached fibres associated with iron deposition during mordant bleaching, and there was no need for an after-treatment with a reducing agent. The process used a mixture of thiourea and hydrogen peroxide to produce thiourea dioxide. This is an effective reducing agent when applied during mordanting,[87] and lower mordanting temperatures are possible.

Dromedary camel hair

Msahli *et al*.[79] reported on the effect of various parameters on the laboratory de-hairing of Dromedary camel hair using a Shirley Analyser. They found that four passages through the Shirley Analyser were optimum for de-hairing, the hair being scoured prior to de-hairing, with an opening process prior to the Shirley Analyser being of benefit, particularly in terms of fibre length. After the fourth Shirley Analyser passage, the down still contained about 10% of coarse hairs (> 30 μm).[79] If an opening process was first carried out, the Shirley Analyser component had a mean fibre length of 35 mm (CV = 47%), that of the original raw fibre having been 48.3 mm (CV = 60.4%), and the diameter 22 μm (CV = 39%) compared to that of the raw hair which was 26.5 μm (CV = 64.4%). The Stelometer bundle tenacity for the down was 15 cN/tex and that of the raw hair 10 cN/tex.

9.4.3 End-uses

Camel hair (down) fabrics, both woven and knitted, are well known for their warmth, comfort and hard wearing properties. They also drape well[74,75] and are used in both ladies' and men's high-quality clothing (apparel). Good quality pure camel hair products are highly sought after, but expensive, resulting in the fibre often being blended with wool. It is used, particularly in blends with wool, for men's and women's coats, jackets and blazers, skirts, hosiery, sweaters, gloves, scarves, mufflers and caps, dressing gowns and robes,[82] as well as in high-quality blankets.[75] As an example, camel hair and noils are blended with fine wool and spun on the woollen system for use in fabrics for overcoats, knitwear and rugs.[80] Camel hair is also blended with nylon to produce hosiery and other knitted products.[82] Products containing Camel hair should be dry cleaned or hand washed.[82]

The long and coarse guard hair, after being combed into tops, is spun into yarn on the worsted system and used in good quality interlinings, ropes, industrial belting,[74] tent fabrics,[80] in carpets and warm waterproof coats[82] and even paint brushes.[75] The hair from the North African camel is used in carpets, being harsh and unsuitable for apparel.[75]

Further information on camel hair can be found in the references.[2 and 81]

9.5 Cashgora

9.5.1 Fibre production, harvesting, properties, processing and end-uses

Although Cashgora initially attracted much interest, this was not maintained and the production of de-haired fibre dropped from about 200 000 kg in 1990 to about 60 000 kg in 2000.[2] Today little Cashgora fibre appears to be produced commercially, particularly in New Zealand and Australia.

The name Cashgora was coined in Victoria, Australia, in the late 1970s.[88] It comes from the Cashgora goat (Fig. 9.6), and it has been labelled the first new natural textile fibre of the last 100 years. The name 'Cashgora' was accepted as a generic term by the International Wool Textile Organisation (IWTO) in 1988.

The double coated Cashgora goat is the progeny of a cross between a male (ram or buck) Angora goat (*Capra hircus aegagrus*) and a female (doe) down-bearing (cashmere-bearing) feral or cashmere goat (*Capra hircus laniger*), the first crossing producing the finest fibres. These goats were originally predominantly reared in New Zealand[89] and Australia, and shorn twice a year, as is the case for Angora goats. It has been stated that Cashgora is normally produced in the first and second cross and can be regarded as fine mohair[90] or coarse cashmere.[91] In Australia, the first cross between a female

9.6 Cashgora goat. (Source: http://flyinggoatranch.blogspot.com.)

feral goat and a male Angora goat is called a Cashgora, the fibre having some of the characteristics of both cashmere and mohair. Nevertheless, crosses of the Angora goat with cashmere goats, the *Anglo-Nubian* and dairy goats have also been recorded[92] – goats similar to the Cashgora goat, and involving cross-breeding native Kirghis from the former USSR and Angora goats from Turkey were already being produced in 1820.[93] It has been stated[91] that Cashgora is a classification for coarse cashmere: the Cashmere and Camel Hair Manufacturers Institute classifies only fibre not exceeding 19 μm as cashmere and everything exceeding this as Cashgora and not entitled to be classified as cashmere.[91] Friedlin[93,94] reported on the production and characteristics of New Zealand Cashgora which was defined[95] as the down component from a two-coated fleece (down and guard hair) having a mean fibre diameter between 17.5 (sometimes it can be as fine as 17 μm) and 22 or 23 μm, a standard deviation below 6 μm, a CV of fibre diameter below 28% and less than 6% of fibres coarser than 30 μm. Three types of Cashgora have been defined, ranging from the top end (18.5 μm), marketed as 'Ligne Or', the medium range (20 μm) marketed as 'Ligne Emerande' and the lower range (just below 22 μm) marketed as 'Ligne Saphir'.[96] At René Friedlin the de-haired Cashgora was classified in three classes according to diameter, namely:[93] 17–18.5 μm, 19.5–21 μm and 22–23 μm.

Cashgora fibre (or hair) can therefore be taken as the 'down' (fine de-haired secondary fibre) component of the fleece of the two-coated Cashgora

goat, under 22 μm (in some cases as 18–22 or 23 μm) in mean fibre diameter, with a length generally between about 30 and 90 mm (usually between 40 and 60 mm, individual fibres even exceeding 100 mm). The mean fibre diameter of Cashgora can in fact range from about 19 to 24 μm and the CV from about 25 to 35%. Finer fibres are down to 12 μm, with the coarser end running up to 45 μm. The fibres are medullated in some cases, and the fleece has almost the same lustrous appearance of mohair, none of the fibres being crimped. It has to be de-haired (i.e. the down fibre has to be mechanically separated from the coarse, rather kempy hair or primary fibres). The down has a low to medium (gentle) lustre and is generally white, soft and delicate to the touch. Cashgora is de-haired using the same criteria as for cashmere, namely, fibres coarser than 30 μm are classified as guard hair, the fine inner down representing approximately 50% of the mass of the fleece.

Phan *et al*.[97] discussed the morphological features of Cashgora, showing that they differed sufficiently from those of cashmere to allow the two types of fibres to be distinguished. Phan *et al*.[98] stated that the scale structure of Cashgora is more similar to that of mohair than to that of cashmere. Nevertheless, Cashgora fibres are considered to possess either cashmere-like features (i.e. cylindrical and semi-cylindrical scales) or the characteristics of mohair, with 'splits', lance-shaped scales and subscales.[31] In the kid and young goat stage (up to 2 years of age) the fleece of the Cashgora contains fibres which are similar to cashmere and also fibres which are of the mohair type, both with lustre.[99,100] As the animal ages, the fine cashmere type fibres disappear and the fleece reverts to super-fine mohair in characteristics. A guard hair is always present.

Cashgora fibres range from a bilateral to non-bilateral structure, some resembling the bilateral structure of the cashmere, others resembling the non-bilateral structure of mohair, with the majority being intermediate.[10,101] It contains both ortho-cortex and para-cortex, with fewer fibres exhibiting a bilateral structure than is the case for cashmere. After de-hairing, Cashgora generally follows a similar processing route as wool and mohair. Cashgora can generally be woollen-spun and used in most articles of clothing (e.g. jackets, coats, scarves and stoles, with the exception of underwear) as well as in blankets. It is considered more suitable for the weaving trade for high grade lightweight suiting fabrics.[93] Albertin *et al*.[102] have compared the behaviour and properties of Cashgora during finishing operations.

Knitted Cashgora garments do felt and shrink during machine washing,[103] although they can safely be hand-washed. The pilling of the knitted garments was low (good). A chlorination shrink-resist treatment, involving sodium hypochlorite, reduced felting shrinkage during machine washing to acceptable levels, but caused some yellowing and scale modification, the latter assessed by means of the methylene blue test.[103]

9.6 Cashmere

9.6.1 Fibre production, harvesting and properties

Cashmere goats (*Capra hircus lasinger*) (see Fig. 9.7), also known as 'Shawl goats' or 'goats of Tibet',[104,105] have two fibrous coats (usually white): a coarse outer coat (guard hair) and fine down (undercoat).

The thick 'guard hair' provides physical protection while the fine undercoat provides thermal insulation,[106] an ideal combination for extreme climatic conditions, particularly extremely cold and wet conditions. Generally, the fine down hair grows during summer, preparing the animal to withstand the winter, and is then shed in spring when it can be harvested, the animal not requiring this highly insulating layer under warm, even hot, summer conditions. The outer hair of the ordinary goat is mostly too coarse for textile use, although raw goat hair (i.e. a mixture of the down and guard hair) was used in earlier times in fabric (e.g. tenting) and carpets.[106] Goat hair is still used to make brushes and interlinings, with the down removed to make felts.[106] Cashmere goats, found in extreme climatic mountainous regions, around the Himalayas and Central Asia, particularly in the regions around the Gobi desert,[52] are domesticated in various countries, such as China, Inner Mongolia, Iran (from the Passang or Iranian goat), and Afghanistan, and also bred in countries such as Australia and New Zealand. There is, however, no specific or distinct breed of cashmere goat,[107] although cashmere goats are generally white with spiral horns,[106] ranging between about 40 and 70 kg in weight and 0.6 and 0.8 m in height, with an average life span of about 7 years.[2]

9.7 Cashmere goat. (Reproduced from Pier Giuseppe Alvigini, *The Fibres Nearest the Sky*, Mondadore Editore, Verona, by kind permission of Mr Pier Alvigini at Alvigini S.A.S., 13900 Biella Via Dante, 12 Casella Postale 430, Italy.)

The fine down (undercoat) grown by the two-coated 'cashmere' goats is referred to as cashmere, being finest in the first year of the goat's life. The term cashmere, which originated from the Kashmir region (where in the 1700s Europeans first saw the fibre woven into shawls) and goats of Tibetan origin, is sometimes used interchangeably with the words Kashmir, pash and Pashmina[108] (the latter is the Persian term for cashmere,[109] and the local term for the cashmere or Pashmina goat and cashmere from the southern Himalayan region in India and Nepal; see also Section 9.13.8). Cashmere, also referred to as Tibet hair,[110] 'soft gold' and 'fibrous diamond',[111] can be defined,[112] according to the US Wool Products Labelling Act, as the 'fine (de-haired) undercoat fibres produced by a cashmere goat, with an average fibre diameter not exceeding 19 μm, and with no more than 3% of the fibres (by weight) having an average diameter that exceeds 30 μm, and a CV of diameter not exceeding 24%'. According to this definition, therefore, the coarse guard hair from the cashmere goat is not regarded as cashmere. Cashmere is generally taken as the fine (de-haired) undercoat (down) fibre from a two-coated goat.

The diameter of the down from cashmere goats is similar to that (≈14 μm) from 'ordinary' goats, although the former normally produces more down fibre than the latter. The AATCC definition of cashmere was coarsened in 2001,[113] with the ASTM not regarding fibres coarser than 30 μm as being cashmere.

Phan and Wortmann[114] proposed the following definition of cashmere:

- The de-haired, fine undercoat fibres produced by a double-coated species of goat.
- An upper limit of 19 μm for mean fibre diameter and 24% for the CV of 'diameter'; with the level of coarse hairs (> 25 μm) less than 3% by weight.
- Goat fibre samples with a mean fibre diameter (MFD) between 19 and 23 μm are classified as Cashgora.
- First class cashmere should have a MFD below 15.5 μm and a soft handle.
- Cashmere with an MFD higher than 15.5 μm is second class.

The bulk of the global cashmere production comes from China and Mongolia (combined over 90%), with the rest coming from Iran, Afghanistan, Russia, India, Pakistan, Turkey, Australia, New Zealand, Britain and the USA.[104] China's production of raw cashmere fibre (i.e. guard hair plus cashmere) was estimated[115] at some 15 million kg, in 2005, some 6.5 million kg being produced in Inner Mongolia.[116]

The guard hair (outer coat), of the cashmere goat is long (around 15 cm), coarse (from 30 to 150 μm, average about 60 μm) and medullated, and is

produced by the primary follicles. The fine, short, unmedullated down or undercoat (with a prominent surface scale structure), termed cashmere, ranging in single fibre diameter from 4 to 30 μm,[117] is produced by the secondary follicles. Between 100 and 300 g of fine down is produced per goat, with an average diameter ranging from about 12 to 19 μm, and average length from about 20 to 50 mm. The finest cashmere generally comes from China and Mongolia, although the average diameter of Mongolian cashmere increased from about 16 μm to between 17 and 19 μm after the mandatory culling of older male goats was revoked, the fineness of cashmere from male goats increasing by up to 1.5 μm at the age of 2 years. Nevertheless, the introduction of the Liaoning breed in Inner Mongolia is bringing the diameter down to about 15.5 μm. The Arbas breed of cashmere goat in Inner Mongolia is considered to produce excellent quality cashmere, with a high yield of up to 400 g of fine down.

The cashmere goats moult over several weeks during spring, when the cashmere is harvested either by combing (e.g. in China), or shearing (e.g. Iran, Afghanistan and Australia),[118] the long, outer or guard hair often being shortened (clipped) prior to combing. The method of harvesting affects the fleece characteristics, such as the ratio of down fibre (cashmere) to guard hair, and the level of contaminants, these also being influenced by nutrition and goat quality.[118] It also affects de-hairing efficiencies, for example it is more difficult to de-hair shorn hair than combed hair.

The total hair removed from a cashmere goat can be about 800–900 g. Of this some 500 g is clipped guard hair. The balance of 300 g (raw cashmere) is hand sorted which removes ± 20% of residual guard hair. Willowing removes about 10% of sand and dirt and the remaining 200 g is then scoured, leading to a further 20% loss. De-hairing then decreases the yield by another 30% (due to guard hair removal, known as cashmere waste), leaving 50% fine de-haired cashmere down (i.e. ≈ 100 g).[119] The fleece of the cashmere goat typically contains between 50 and 75% of fine down (i.e. undercoat).

Australia's cashmere industry, based on their feral goats,[120] was launched in 1972.[104] Australian feral goats have a fine, 15 μm or less[121] (even down to 13 μm), and ≈ 60 mm long undercoat (down) and ≈ 60 μm hair fibres (≈ 55 mm). They are probably the progeny of the original domestic goats from various European and Asiatic areas, during the latter 1800s.[121] Only about one in ten of these feral goats produce cashmere type fibres, which are often pigmented, the average down production per feral goat being between 75 and 125 g per year in 1981.[122] In order to produce white Australian cashmere, white top quality Angora goat males (bucks) are crossed with female feral goats carrying coloured down fibres, producing mostly white soft down fibres, > 18 μm, which is called Cashgora in New Zealand.[121] Cashgora, 18–23 μm, has a CV 25–30%, whereas well de-haired cashmere usually has a CV less than 20%[121] (see Section 9.4). Australian cashmere, which is produced

Table 9.20 Cashmere produced in different countries[104]

Country	Average scouring yield (%)	Average dehairing yield (%)	Average diameter (μm)
China*	75	51	14–16
Mongolia*	75	51	16–17
Iran†	80	35	17–19
Australia‡	92	30	15.5–18.5

* Blackburn 1990.
† Ekhtiyari 2000.
‡ Australian Cashmere Marketing Corporation.

in both Australia and New Zealand, is shorn, is predominantly white in colour, and considered to be longer, stronger, cleaner, softer, less crimped (i.e. lower curvature) and more lustrous than that produced in other countries.[104] It is therefore considered suitable for processing on the worsted system. The average fine fibre yield is some 200 g per animal, with an objective of 500 g per animal ultimately. Singh *et al.*[104] gave a table containing comparative data of cashmere production in various regions.

Cashmere is not as lustrous as mohair, and ranges in colour from white to brown, even black. According to the surface structure as well as handle, cashmere can be differentiated into 'cross-bred cashmere' and 'classical Asian cashmere'.

Various 'cashmere fibre types', classified according to average fibre diameter, presence of guard hair and colour are produced commercially (white is the most valuable; even 5 fibres/g of coloured fibres in white cashmere impact negatively on quality). International markets generally evaluate (grade) raw cashmere quality according to fineness (fibre diameter), colour, length and level of contamination (e.g. bits of skin, vegetable matter, etc). Fibre diameter greatly influences the market price of cashmere: the finer the better the perceived quality and price. The colour of the fibre also has a great impact on its price, with white being the most expensive. Black cashmere generally has to be bleached, particularly if it is to be dyed to pastel shades. Price is also affected by the fibre length, level of guard (coarse) hair and level of contamination (e.g. bits of skin, vegetable matter, etc.).

Fibre diameter can be measured by projection microscope (IWTO-8-97), Airflow (IWTO-6-98), Sirolan-Laserscan (IWTO-12-98) and OFDA (IWTO-47-95), as well as by scanning electron microscopes (SEM) and cross-sectional (CS) methods.[123] Some differences between the different techniques having been observed, however.[117] Fibre length, for example, can be determined by the time consuming single fibre length test (IWTO-16-67), or using an Almeter (e.g. AL-100) according to IWTO-17, values of 40 mm being obtained on Mongolian cashmere, when using the latter method.[117]

Cashmere fibres generally have very little crimp. Work by McGregor[124] and McGregor and Postle[125] shows that cashmere from newer producing regions, such as Australia, New Zealand and the USA has an even lower fibre curvature (i.e. crimp) than cashmere from the traditional sources, such as China, Mongolia, Iran and Afghanistan, with cashmere fleeces not exhibiting the well-defined staple crimp structure (formation) found in Merino fleeces. Fibre curvature was, as in the case of wool, negatively related to fibre diameter and nutrition affected the curvature of Australian cashmere. For the Chinese cashmere, the age and sex of the goats affected the fibre curvature, as follows; bucks 52°/mm, does 65°/mm and kid bucks 78°/mm.[124] For Mongolian goats, crimp curvature varied from 55 to 66°/mm, decreasing with mean fibre diameter, and also varying between certain regions.[117] McGregor and Postle[125] gave two tables representing results obtained on commercial de-haired cashmere and cashmere tops from various origins (Tables 9.21 and 9.22).

Chinese cashmere is considered to be the best quality, with an MFD between about 13.0 and 16.5 μm. Iranian/Afghan cashmere is 1–2 μm coarser (around 17–19.5 μm) and that from Australia and New Zealand is around 16–18 μm. According to tests done in Germany on commercial cashmere samples from different origins, the MFD ranges between about 18 and 19 μm,[121] with less than 1% (even 0.2% for knitwear and 1% for wovens[126]) of coarse fibres (i.e. coarser than 30 μm). For good quality Chinese cashmere,

Table 9.21 Median, standard deviation (SD) and range of pooled data for attributes of de-haired cashmere[125]

Top attribute	Median	SD	Maximum	Minimum
Mean fibre diameter (MFD) (μm)	16.4	1.4	19.3	13.5
CV of MFD (%)	21.7	1.6	29.0	19.8
% Fibres >30 μm	0.5	0.5	2.1	0.1
Fibre curvature degree/mm	60.0	9.3	79.7	40.1
Resistance to compression (kPa)	5.6	0.6	7.7	4.5
Incidence of medullated fibre (%w/w)	0.43	2.4	8.8	0.0
Mean medullated fibre diameter (μm)	34.8	12.3	62.9	23.6
LAC* (mm)	22.8	7.8	36	15
CV (LAC)* (%)	67.0	5.7	75.6	52.1
LAC fibres <25 mm (%)	63.1	11.3	86.7	38.0
LAC longest 5% (mm)	53.6	10.6	77	32
Inferred 'Barbe' (mm)	33.6	7.1	50	19
Ratio LAC: MFD (mm/μm)	1.41	0.30	2.21	0.93
Bundle tenacity (cN/tex)	9.9	0.9	12.0	8.2
Bundle extension (%)	40.8	4.5	50.0	31.3
Lightness	61.1	3.6	65.4	44.8
Yellowness	–0.3	1.8	3.7	–4.3

*LAC: length after carding.

Table 9.22 Median, SD and range of pooled data for attributes of cashmere tops[125]

Top attribute	Median	SD	Maximum	Minimum
Mean fibre diameter (MFD) (μm)	17.5	1.2	19.3	15.2
CV of MFD (%)	21.2	1.2	23.8	19.8
Fibres >30 μm (%)	0.6	0.4	1.6	0.1
Fibre curvature (degree/mm)	58.0	5.5	68.6	48.9
Resistance to compression (kPa)	5.9	0.4	8.3	3.6
Incidence of medullated fibre* (% w/w)	0.3	0.4	1.5	0.1
Mean medullated fibre diameter* (μm)	29.8	9.2	51.7	23.6
Hauteur (mm)	41.1	4.9	50	28
CV of hauteur (%)	41.2	6.9	57.4	31.8
Hauteur fibres < 25 mm (%)	16.8	11.6	51.1	6.9 59
Hauteur longest 5% (mm)	69.7	6.6	82	37
Barbe (mm)	46.7	4.9	57	31.2
CV of Barbe (%)	36.7	5.0	47.8	1.52
Ratio hauteur: MFD (mm/μm)	2.37	0.36	2.91	8.3
Bundle tenacity (cN/tex)	10.4	1.0	12.0	19.5
Bundle extension (%)	39.1	6.6	50.0	

* Adapted from McGregor and Postle, 2004.

the fineness of the individual fibres ranges from about 8 to 24/25 μm,[123] with fibres coarser than 22 or 23 μm rare in well de-haired cashmere; it is stated that fibres between 25 and 30 μm are morphologically conspicuous and in appearance more like the coarse guard hair.[121] The upper threshold of the mean fibre diameter of commercial cashmere is around 19 μm, being set at 16 (+ 0.5 μm) by the Chinese National Standard, at 18.5 to ±0.5μm by the CCMI (Cashmere and Camel Hair Manufacturers Institute) in the USA, and at 19 μm by the ASTM.[123]

Well de-haired cashmere samples typically have a CV of diameter of 20%[114] (even up to 25%),[76] compared to that of 25–30% for Cashgora. The mean fibre length of the fine undercoat fibre of Chinese raw cashmere typically varies from about 21 to 40 mm (super grade), the lengths of the individual fibres ranging from about 5 to 80/90 mm,[123] depending upon the quality of the sample. Cashmere from China and Mongolia, as well as from Iran and Afghanistan, has shades of white, light grey, dark grey and brown; that from Iran and Afghanistan is typically cream, fawn and dark brown. Chinese cashmere (de-haired) average fibre length ranges from about 24 mm for the poorer qualities (e.g. Brown, Second Grade) to 36 mm for the top qualities (e.g. Super Grade).[110,114,121] That of Asian and Australian cashmere is measured at between 21 and 40 mm,[110,114] the individual fibre lengths ranging from 10 to 90 mm. Super Class cashmere has a fibre length of 38 mm (CV ≈ 33%) and First Class 36 mm. On the basis of the IWTO 5-66 test method, values for mean fibre length between 21 and 40 mm were found for Asian as

well as Australian cashmere.[121] Coarse fibre (>30 μm) contents for Chinese Super Grade, First Grade and Second Grade are less than 0.1%, 0.2% and 0.5%, respectively (National Criteria of China for Cashmere Analysis, practised by CCIB offices in China).

Mongolian cashmere mean fibre diameter tends to increase with goat age, on average by roughly 1 μm from the age of 1 year to the age of 5 years.[117,127] It was found that the mean fibre diameters of cashmere from different provinces of Mongolia ranged from about 15.5 to 18 μm (CV ≈ 20%), increasing with age,[117] and mean fibre curvature from about 62 to 70º/mm[127] (55 to 66[117]), with a mean of 68.4º/mm. The mean bundle tenacity was ≈ 12.5 cN/tex and the bundle extension 45%.[117] In all, 94% of the samples had a down yield of over 60%, the average down yield being 80.4%, ranging from about 65% to about 85%. Mean fibre length of the two best provinces exceeded 38 mm in most cases, with 50% exceeding 40 mm. Yak and camel hair are about 1–3 μm coarser than cashmere, yak having some 2 scales per 100 μm more than Mongolian cashmere.[127]

Scale height of speciality fibres, such as cashmere, is about 0.4 μm and that of wool about 0.8 μm.[114] Different strains of classical Asian cashmere exhibit similar fibre surface characteristics,[123] with typically regular cylindrical and semi-cylindrical scale shapes, i.e. each cuticle cell envelopes the entire or half of the fibre shaft, with a mean scale frequency of 6–8 per 100 μm fibre length, e.g. 6–7 per 100 μm and a scale height < 0.5 μm for Mongolian cashmere.[117] Cashmere from the relatively new sources exhibits quite different scale shapes than the Asian types, usually having a higher scale frequency (> 8 per 100 μm). It is visually more complex and less regular, with many scales arrowhead-shaped like mohair,[114,123] and tends to be more lustrous and smooth (slippery), with a harsher handle than its Asian counterparts. The cortex of cashmere consists mainly of ortho-cortical and meso-cortical cells.[128]

9.6.2 Fibre processing

Raw cashmere can have between 25% and 30% of sand and dust, 4–5% (8–10%) of grease and 65–70% of fibre (clean fibre yield). Typically, cashmere processing commences with sorting, willowing (to remove dirt, grass, etc.) and aqueous scouring, to remove natural oils (grease), etc. Scouring yield is about 70%,[126] followed by de-hairing.[118] The most important step in cashmere processing is that of de-hairing, the effectiveness of which has a major impact on the quality, price (value) and processing of the fibre. The residual grease after scouring should be around 0.5%. De-haired cashmere (down yield), based upon the raw fibre weight, can vary from around 30% to 70%, this being largely dependent upon the method of fibre harvesting, i.e. combing or shearing, and the type and quality of the goat. Originally

de-hairing was done by hand, when it took some two hours to de-hair less than 60 g of fibre.[106] A cashmere de-hairing process was developed by Joseph Dawson in the 1870s, with the first successful commercial de-hairing machine being invented by Joseph Dawson and Sons in the UK some 100 years ago[129] with many different patents and designs of de-hairing machines appearing subsequently. Mechanical de-hairing essentially consists of three phases: opening up (individualising) of the fibres, separation and removal of the guard hair and fibre mixing.[130] Commercial de-hairing production rates are around 3 kg/h/m width.

The mechanical de-hairing of various animal fibres, such as cashmere, camel and yak, have been discussed.[130] Fibre rigidity/stiffness (which is largely determined by the fibre diameter) as well as fibre length play an important role in efficient mechanical de-hairing. A diameter threshold of around 30 μm is often used to distinguish and separate the fine down from the coarse guard hair. The separation criterion used for de-hairing by the combing principle is fibre length;[130] it merely removes relatively short fibres, hence, it is only effective if the fibre lengths of the fine down and the coarse guard hair do not overlap. A roller carding principle of de-hairing has also been developed.[130] The carding stage is particularly important in producing good quality cashmere yarns, requiring appropriately fine card wire and sharp points, as well as a very level surface. The world standard for Super A grade cashmere is 38 mm, modified cotton combing machines providing a length of 40 mm.[131]

It is possible to use both a modified Shirley Analyser[132–134] and the Shirley Trash Separator[134] for the laboratory de-hairing of cashmere, and for determining the yield, although some caution is necessary in the interpretation and application of the results, particularly those pertaining to fibre length. Aspects relating to the objective measurement of cashmere have been dealt with by Stubbs and Marler.[135] The Australian Wool Testing Authority Ltd. (AWTA Ltd.) developed[135] (see also www.awta.com.au) standard procedures for cashmere fleece testing, as well as for yield and mean fibre diameter measurement of sale-lots. Glasbey *et al*.[136] in 1994 reported on the use of digital image processing of microscopically measured diameters of raw cashmere (i.e. un-dehaired) to determine the mean fibre diameter of the down and guard hair, respectively, as well as the cashmere yield, without the need for first de-hairing. Lupton *et al*.[137] reported on a method they developed for estimating cashmere down yield and average fibre diameter of raw (i.e. un-dehaired cashmere) using an optical Fibre Diameter Analyser (OFDA). The fibre diameter results, but not the yield, correlated well with Shirley Analyser de-haired sample results. The density of guard hair decreased with increasing fibre diameter, due to concomitant increases in fibre medullation[137] (Phan and Wortmann[138] have dealt with morphological aspects of cashmere). Singh[21] and co-workers[139] have reported on a

prototype cashmere de-hairing machine purpose built for 'Australian' shorn cashmere, which contains 60–70% coarse hair.

McGregor and Butler[118] found that the following factors were associated with more efficient de-hairing and/or longer de-haired cashmere: white colour, longer raw cashmere (staple length), greater fibre curvature, lower vegetable matter, normal length guard hair, and the absence of visible cotting, with coarser diameters also de-hairing more efficiently. Generally, commercial cashmere samples have a coarse hair content below 1%.[114] The level of coarse (guard) hair (usually taken as the fibres > 30 μm) present in good quality de-haired cashmere should be below 1%, or less than 0.5% by weight, according to Wang *et al*.[21]

Because cashmere is so expensive, and for improved processing, performance and yarn and fabric quality, it is often blended with silk and fine wool, such as lambswool, sometimes with a small percentage of nylon to provide added stability and strength. Nevertheless, unless it is blended with an extremely fine fibre, its handle and softness can suffer.

Due to its length and other factors, de-haired cashmere has been mainly processed on the woollen system, often mule spun. If it is long enough, it is processed on the worsted system, but only very rarely on the semi-worsted system. Similar machinery to that used for fine wool is employed, but with settings, conditions, etc., which are most suitable for cashmere and generally kept secret. Until the mid-1970s most cashmere was processed in Europe, mainly Scotland and Italy, but since the late 1970s China has increasingly processed cashmere locally,[104] this also applying to Mongolia. It has been stated[140] that in Scotland, a down (i.e. de-haired) yield of some 120 g of fibre can reduce to about 66 g at the spinning (i.e. yarn) stage. Some work has even been done on processing cashmere (35 mm, 18.7 μm) on the cotton system, followed by rotor (open-end) spinning.[141] A good quality cashmere top typically has a mean fibre length of about 38 mm and a CV of 33%.

Cashmere is generally fairly sensitive to dyeing and finishing conditions and the severity of the conditions (e.g. temperature, time, pH, agitation, etc.) must be minimised. Soft water is recommended for producing the lather which is so important in the optimum washing (scouring) processing of cashmere knitwear. It is also more sensitive to alkali than wool.

Pigmented keratin fibres contain black melanin pigment granules[100] (0.9 μm × 0.3) which are inert to all solvents. Oxidative and reducing agents, except hydrogen peroxide,[142] are used in what is essentially a two-stage bleaching process, involving an initial fast solubilisation of the melanin, followed by a slow decoloration. Bleaching is often associated with severe fibre damage: iron mordanting improves whiteness but with the penalty of increased keratin damage.[142] The binding of the ferrous ions to melanin was far stronger than that to the keratin. Khishigsuren *et al*.[143] showed that an after-bleaching rinsing process, involving sodium bisulphite on cashmere,

produced a better whiteness and less fibre damage than the conventional bleaching process. Pigmented fibre bleaching generally involves three stages: mordanting with ferrous salts, rinsing and treatment with hydrogen peroxide in an alkaline medium.[143] 18-MEA is the major lipid bound to the surface of a cashmere fibre,[142] which is removed by the hydrogen peroxide bleaching.[142] After-Chrome dyeing of cashmere with SCA-Cr mordant decreased the level of hexavalent Cr (VI) in the residual dye-bath and damage to the fibre[144] was notably reduced in terms of surface oxidation damage to the fibres.

9.6.3 End-uses

The quality of cashmere, notably fineness and length, plays an important role in determining its specific end-use (i.e. product), with the higher quality fine cashmere preferred for knitwear, where softness is of primary importance, while the coarser (lower) qualities often go into weaving. It is considered desirable for very high quality knitting and weaving yarns that the cashmere should be 40 mm or even longer. Good quality cashmere, notably the finer qualities, is popularly used in knitwear (particularly fully fashioned), scarves, shawls, ladies' underwear, etc., Scottish manufacturers set an 'unofficial' standard for the upper limit of cashmere to be used in such knitwear as 15.5 μm.[114] Fully fashioned knitting of cashmere has always been popular since it reduces the amount of costly fibre waste, and produces superbly shaped quality garments. Because pure cashmere knitting yarns tend to be weak, optimum lubrication is important to ensure relatively trouble-free knitting; so too are proper machine settings, low yarn tensions, straight as possible yarn paths, low take-down tensions and relatively slow knitting speeds.

Because cashmere is such a sought after fibre and so expensive, together with the rather limited supplies, it is in some cases even reused (recycled or re-processed). Reused (recycled) cashmere should, however, be clearly labelled as 'recycled cashmere'. Fisher's discriminant analyses have been used to distinguish between new and recycled cashmere.[145]

Although machine washable cashmere garments have been produced, they are generally cleaned by dry-cleaning or gentle hand-washing, using water which is not very hot and a pure soap powder or liquid or detergent specifically recommended for cashmere. The garment must not be rubbed or wrung during washing and should be rinsed thoroughly in cool water without softener and dried flat, out of direct sunlight. Cashmere is said to become more quickly saturated with water than wool.[107] The measurement of pilling in cashmere knitted garments has been investigated.[146] For further information on cashmere see the references[2 and 100] and www.cashmere.org.

9.7 Guanaco

9.7.1 Fibre production, harvesting and properties

Some 2 million years ago, the guanaco (genus: *Lama hunchus* or more commonly *Lama guanicoe*), a wild (now semi-captive) member of the South American Camelid family, first appeared in the palaeontological record, evolving in South America from *Hemiauchenia*.[147] The guanaco (see Fig. 9.8) is larger than the alpaca and vicuña, but smaller than the llama, and is a very timid animal and was originally killed for its meat and fleece,[12] although today it is farmed in semi-captivity and shorn for its fleece.

The guanaco represented an important source of meat and fibre, the latter used in textiles for thousands of years by pre-Columbian cultures,[147] and reserved for the clothing of ruling families during the Inca Empire.[147] According to Rainsford,[147] there were an estimated 30–35 million guanacos roaming the South American continent prior to the Spanish conquest of eastern South America in the early 1530s, but their numbers decreased drastically due to excessive hunting, competition for pasture from European livestock and the introduction of fencing.[147] It is indigenous to Southern Patagonia, both in Argentina and Chile. It is estimated[147,148] that, in more recent times, there were well over 600 000 animals in the world, (including some 500 000 in Argentina, 90 000 in Peru and 70 000 in Chile), mainly in small herds in Patagonia and the island Tierra del Fuego,[148] about 1% being

9.8 A guanaco. (Source: Inca Tops SA, Arequipa, Peru.)

bred in semi-captivity. Guanaco, also known as Huanaco in Peru, can survive at almost any altitude, from sea level at Tierra del Fuego to 4500 m above sea level along the Andes of Northern Chile, Peru and Argentina (also referred to as luan), and almost every climate from semi-tropical to extremely cold snow-capped glaciers.[147,149] They are both grazers and browsers, with areas of lush bog, known as 'bofadels', ideal for breeding males,[147] adult males weigh between 100 and 150 kg[147] and adult females between 100 and 120 kg and their height ranges from about 1 to 1.2 m.[147] Very young guanacos (chulengos) are hunted for their meat and valuable skins, and fibre is also shorn from their skins.[150]

In 1975 the guanaco was listed on the Convention on Trade in Endangered Species of Wild Flora and Fauna (CITES) list (regulations in Appendix II), meaning that, legally, guanaco fibre can only be shorn and processed into luxury goods in accordance with CITES regulations covering protected animal species.[147] Guanacos are now bred in semi-captivity, while wild adults are captured, shorn and released.[151] A challenge to guanaco fibre breeders is to achieve the critical fibre production of between 4000 and 4500 kg of raw fibre required for industrial processing.[151] In Argentina, growers were allowed (in 2000) to capture a maximum of 10% of their total wild guanaco population with a view to adapting the young 'chulengos' to a new life which requires high fencing, special baby feeding and separation of families to avoid adult male fights.[152] To raise guanacos in semi-captivity involves rounding-up pregnant wild females and keeping then in corrals until they give birth,[147] after which the mothers are kept with their young for three months to familiarise them with human contact, and then they are released back into the wild, making them easier to round up and recapture for shearing.[147] Attempts have also been made to raise the guanaco entirely in captivity, by capturing the very young, preferably week-old 'chulengos', bottle feeding them for 120 days, taking great care to avoid mortality,[148] and then releasing them into specially prepared paddocks with fences up to 2.1 m to graze on nutritional pastures, supplemented during the first winter.[148]

The guanaco fleece is shorter, coarser (≈ 18–24 μm) and lighter in colour than that of the vicuña,[12] weighing some 750 g[147] to 1 kg, and consists of two coats: a protective relatively coarse outer coat (≥ 50%) and a much finer (≈ 16.5 μm) undercoat (≤ 50%). The animal is honey (fawn) coloured with a white underbelly and legs, and greyish-colour head and ears and the colour of the fleece ranges from golden/beige for the younger animals (chulengos) to dark beige and reddish brown for the adult animals.[150] The guard hair is up to 45 μm (usually 23–35 μm) in diameter and up to 140 mm in length. The diameter of the undercoat ranges from about 13 to 19 μm, the fibre diameter varying according to feeding conditions. Good animals achieve a mean fibre diameter of 14 μm[154] while a young guanaco fleece can even be as fine as 13.3 μm, and 34 mm long,[153] with a weight of 450 g.[148] Commercial

de-haired guanaco fibre ranges in diameter from about 13 to 17 μm,[7] with the bulk being 15–16 μm.

In an attempt to standardise the quality of fibre offered by the farmers (estancias), the Argentinian government has implemented regulations requiring its representatives to consult with the farmers concerning their fibre-harvesting systems (both from wild or semi-captive animals).[147] The price paid for guanaco fibres depends mainly upon fineness, length and clip preparation.[153]

9.7.2 Fibre processing

Hair harvested (shorn) from the guanaco needs to be de-haired to separate the coarse guard hair from the fine undercoat fibres. Guanaco fibre is mainly processed on the worsted system, although some fibre is also processed on the woollen system, scouring yields varying from about 85 to 95%,[150] with greasy to clean yield quoted[2] as 65–70%. In one case, some 400 high quality guanaco fleeces were processed into 14.97 μm tops, having a CV of 20.22%, Hauteur of 33 mm, a Barbé of 39.1 mm and a relatively low coefficient of variation of Hauteur (CVH) of 43%, having a fawn shade similar to camel.[153]

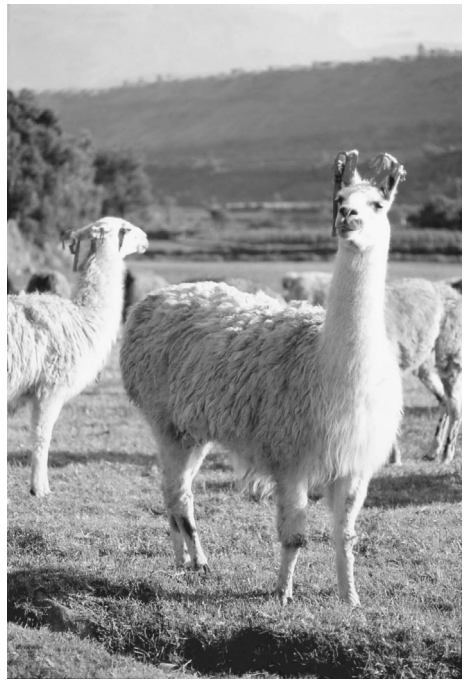
9.7.3 End-uses

Guanaco fibre end-uses are similar to those of vicuña, being mainly used in luxury woven fabrics, supplied to exclusive tailoring companies, the suits of which can retail for US$10 000.[147] It is less commonly used in knitwear, although up to 20% guanaco fibre blended with Merino wool is used in fine fashionable sweaters and other knitted garments.[148] Fine-count worsted yarns, in various blends with Merino wool and/or cashmere, are also supplied to exclusive tailoring businesses in London, Milan and Tokyo, where blazer cloth is popular, and where dress overcoats are made from a fabric containing guanaco, silk and cashmere,[147] each garment being individually certified. Woollen-spun guanaco yarns are used in suiting and over-coating fabrics for exclusive top-quality fashion brands.[147] Guanaco is also making inroads into markets for scarves and shawls. Pure (100%) guanaco is more common in accessories.

9.8 Llama

9.8.1 Fibre production, harvesting, properties, processing and end-uses

The llama (*Lama glama glama*), a ruminant or pseudo-ruminant living in the high altitudes of the Andes, has traditionally, and mainly, been a beast



9.9 Llamas. (Source: Inca Tops SA, Arequipa, Peru.)

of burden of the Andes,[12] from the time of the Incas. It is the largest of the South American branch of the camelids. It is smaller than the camel (see Fig. 9.9) measuring about 1.2 m at the shoulder, and weighs around 100–120 kg.[155] It has been domesticated since time immemorial.

The guanaco or huanaco (*Llama glama hunaca*), a ruminant (or pseudo-ruminant), is the common ancestor of the domesticated llama and alpaca.[156] The natural habitat of the llama is the 'Antiplano', the vast, high and arid plateau of Peru, Bolivia, Chile and Argentina, at altitudes between 4000 and 5000 m,[12] feeding mainly on ichu grass. It has been estimated[155] that in 2006 there were some 3.4 million domesticated llamas in the world, with some 2.0 million in Bolivia. Peru has some 1.1 million and there are also significant numbers in the USA.

The llama fleece has a similar mean fibre diameter as the alpaca but is a double coat. There are, however, essentially two main types of llama. One type, the Kara (K'cara, qara/carguera or light fleece), is mostly used as a beast of burden and is the typical double-coated animal (single fibre diameter of the fleece ranging from about 10 to 150 μm), which has predominantly coarse guard hair and relatively fewer fine undercoat fibres or down.[14,35] The second type, 'C'haku/chachu' (woolly or heavy fleece), which is the main source of llama hair used for textiles, has essentially a single coat of

relatively fine and soft fibres, but with a relatively high level of medullated (kemp) fibres amongst the secondary unmedullated fibres.[35] Interbreeding between llamas and alpacas has resulted in many and varied types (hybrids) of animals and fleeces, the cross of llama male with an alpaca female producing the Huarizo, known in India as Huaro, the reverse cross producing the Paco-llama or Misti.

The fleece of the llama (typically 15–60 μm) is not as attractive as that of the alpaca. It contains both coarse, long, kempy hairs and fine undercoat hairs and it is not easy to separate[12] the coarse guard hair from the fine down, due also to the presence of 30–40 μm intermediate fibres. The coarse guard hair is typically 30–40 μm in diameter (these are also referred to as intermediate fibres), but can even be up to 150 μm, and ranges from about 250 to 300 mm in length (typically over 100 mm) and is kempy. The fine undercoat or down ranges from about 10 to 35 μm in diameter (average around 22 μm; and CV from 25% to 35%[76]) and 65 mm in length, with baby llama fibre diameter being 20–21.5 μm, which is slightly finer than baby alpaca. Wildman[156] suggested 35 μm as the limit (or criterion) for distinguishing between the woolly (undercoat) fibres and the coarse (outer coat) hair. According to tests done by Greaves and Rainsford,[7] the fineness and related quality of llama hair have not changed significantly from 1970 to 2005. The fleece of the llama contains less than 4% grease, less than 25% total impurities and average fibre yield is about 80% (85–90% greasy to clean,[2] quoting Rainsford).

The natural colours of the fleeces are similar to those of the alpaca and there are often different colours within a fleece. It is mostly brown (fawn) in colour, although greys and even white, used alone, are used in blends in knitwear and outerwear chiefly used by the locals in clothing (e.g. ponchos), carpets, rugs, etc., while the coarse llama guard hair is used in ropes, sacking, braids, carpets, etc. The llamas breed, and are also shorn (mainly by hand), during the rainy season, from November to March. A fleece grown over a 2-year period weighs approximately 2.5 kg and only about 1 million kg of normal quality llama fibre is produced annually.

Franck *et al*.[157] have objectively described the different fleece types, subjectively defined as styles, in terms of different fibre types, differentiated according to length, fineness, type of waves and the presence or absence of lustre. Llama fibres are nearly all medullated, only a few of the finest fibres being unmedullated. Cuticular scale frequency is around 10–11 per 100 μm.[35–38]

Llama is de-haired in Bolivia and other countries. Townend *et al*.[5] review the various de-hairing techniques which have been developed over time and report on experimental work on the variables affecting the de-hairing of llama when using a single swift woollen card. They refer to the problems created by the wide range of fibre diameters (10–80 μm) present in un-dehaired llama hair. They conclude that modifying and optimising the carding

arrangement, conditions and settings could lead to significant de-hairing, although probably not to the extent that is commercially desirable.

For more information on llama fibre see the references 2 and 158.

9.9 Mohair

9.9.1 Fibre production, harvesting and properties

Mohair, derived from the Arabic word Makhayar (Mukhayar or Mukhaya), is the fleece of the single coated Angora goat (*Capra hircus aegagrus*), named after the Turkish province of Ankara (Angora or Ancyra) (see Fig. 9.10). The Angora goat is thought to have originated in the Asian Himalayas (Asia Minor) or highlands of Tibet.

The Angora goat is regarded as unique amongst goats in that it is essentially single coated, with the fibres from the primary and secondary follicles not differing very widely, and it does not moult, its fibres growing continuously throughout the year. For centuries, mohair has been regarded as one of the most luxurious and best quality fibres available to man. It is generally a long, straight (uncrimped but often wavy), smooth and naturally lustrous fibre, and predominantly white in colour which can be dyed to deep, brilliant and fast colours. Mohair is characterised by, and renowned for, its high lustre, durability (hard wearing), elasticity, resilience, resistance to soiling, soil shedding, setting, strength, abrasion resistance, comfort (including moisture absorption) and pleasing handle, and by relatively low flammability, felting and pilling. Although mohair has proved extremely popular in many applications, it has some limitations in certain 'close to the skin' apparel applications because of

9.10 Angora goats. (Source: Mohair South Africa, Port Elizabeth, South Africa.)

its coarseness relative to certain other types of luxury animal fibres. Its outstanding properties, such as resilience and durability, also make it particularly suitable for household textiles, such as upholstery fabrics, curtains and carpets. The low flammability of mohair renders it useful in several applications. The Limiting Oxygen Index (LOI) of untreated mohair is about 24, with 27 generally regarded as the minimum required to pass the vertical flame test.

The mohair industry first developed in Ankara, Turkey which was also the first country to supply mohair as a raw material.[159] The first Angora goats to leave Turkey went to South Africa in 1838,[160,161] and Angora goats arrived in the USA around 1849.[162]

Angoras were also introduced into Australia during the 1850s and 1860s,[163] but attracted little interest until around 1970.[163] Angora goats were introduced to Britain in 1881.[163] Today, mohair is largely produced in South Africa (which presently accounts for over 50% of global production), and the USA (Texas), but also in Turkey, Argentina, Lesotho, Australia and New Zealand (Table 9.23).

Angora goats can survive extreme temperatures, but they are very sensitive to cold after shearing, particularly a combination of cold, wind and/or rain. Angora goats can thrive on widely different types of pasture, grazing from about 30 cm to 1.6 m above ground level. It appears that the Angora goat is very efficient in converting feed into fibre[164] and more effective than woolled sheep;[165,166] the latter are more effective in converting feed into body mass. Mohair grows about 20–25 mm in length per month (i.e. 240–300 mm per year), irrespective of age, and Angora goats are generally shorn six-monthly in South Africa and the USA and annually in Turkey and Lesotho. Mohair fibre is generally classified according to the age of the goats and when they are shorn.

According to Van Der Westhuysen *et al.*,[165] the age of the goat is probably the most important factor determining the quantity and quality of mohair produced, with fineness (i.e. diameter), independent of age, also very important. Kids have a birth coat of fibres that grow mainly from the primary follicles, those being the follicles which produce kemp and medullated fibres.[167] From about 3 to 6 months the goats shed their birth coat ('mother hair') as the fibres grow increasingly from the secondary follicles which produce the finer and unmedullated hairs.[167,168] Fibre production increases from birth, reaching a maximum fleece weight at an age of between approximately 3 and 4 years (see Fig. 9.11).

With age, the fibre diameter increases, reaching a maximum at approximately 5 years[165] (see Fig. 9.11). The mohair fibres are finer towards their tips, due to the fact that the fibres become coarser as the goat ages. Fibres from the neck and britch areas of the goat tend to be coarser than those from the other parts of the body.

In South Africa the first shearing takes place around 6 months after birth and the second shearing 6 months later, the goats producing more fibre in the

Table 9.23 World mohair production (million kg greasy)

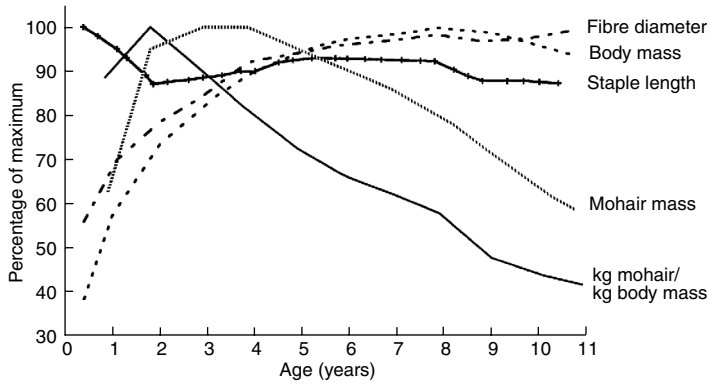
Year	South Africa	Turkey	USA	Argentina	Australia	New Zealand	Lesotho	Other	Total
1972	3.7	4.1	4.6	1.0	–	–	0.8	0.0	14.2
1973	3.4	4.1	4.5	1.0	–	–	0.6	0.0	13.6
1974	3.7	4.1	3.8	1.0	–	–	0.6	0.0	13.2
1975	3.8	3.9	3.9	1.0	–	–	0.6	0.0	13.2
1976	4.1	4.0	3.6	1.0	–	–	0.6	0.0	13.3
1977	4.6	4.1	3.6	1.0	–	–	0.4	0.0	13.7
1978	4.9	4.5	3.7	1.0	–	–	0.5	0.0	14.6
1979	5.4	4.5	4.2	1.0	–	–	0.5	0.0	15.6
1980	6.1	4.5	4.0	1.0	–	–	0.6	0.0	16.2
1981	6.9	4.5	4.5	1.0	–	–	0.6	0.0	17.5
1982	7.6	4.5	4.5	1.0	–	–	0.6	0.0	18.2
1983	7.2	3.8	4.8	1.1	–	–	0.7	0.0	17.6
1984	8.1	3.5	5.0	1.0	–	–	0.7	0.0	18.3
1985	9.2	3.5	6.0	1.0	–	–	0.8	0.0	20.5
1986	11.0	3.0	7.2	1.0	–	–	0.8	0.0	23.0
1987	11.5	3.0	7.3	1.0	1.0	–	0.8	0.0	24.6
1988	12.2	2.9	7.8	1.0	1.0	0.4	0.7	0.0	26.0
1989	11.7	2.0	7.8	1.0	1.2	0.6	0.6	0.0	24.9
1990	10.1	1.8	7.3	1.0	0.6	0.4	0.6	0.0	21.8
1991	7.6	1.2	7.4	0.9	0.5	0.3	0.5	0.0	18.4
1992	6.7	1.2	7.1	0.6	0.5	0.3	0.4	0.0	16.8
1993	6.0	0.8	6.5	0.6	0.4	0.3	0.4	0.0	15.0
1994	5.7	0.8	5.4	0.4	0.4	0.2	0.4	0.0	13.3
1995	5.4	0.6	4.8	0.5	0.4	0.2	0.5	0.0	12.4
1996	5.6	0.4	3.5	0.4	0.4	0.2	0.5	0.0	11.0
1997	5.2	0.4	2.5	0.4	0.3	0.2	0.4	0.0	9.4
1998	5.0	0.4	1.5	0.4	0.3	0.2	0.4	0.0	8.2
1999	4.5	0.4	1.2	0.25	0.25	0.2	0.4	0.0	7.2
2000	4.3	0.4	1.0	0.3	0.3	0.2	0.5	0.0	6.9
2001	4.2	0.3	0.8	0.3	0.3	0.2	0.5	0.3	6.8
2002	4.2	0.3	0.8	0.3	0.2	0.1	0.5	0.3	6.6
2003	4.0	0.3	0.9	0.3	0.3	0.2	0.5	0.3	6.6
2004	3.7	0.2	0.85	0.3	0.3	0.2	0.5	0.2	6.1
2005	3.6	0.3	0.8	0.3	0.2	0.2	0.6	0.3	6.2
2006	3.4	0.3	0.8	0.4	0.2	0.1	0.75	0.2	6.1
2007	3.0	0.35	0.55	0.45	0.2	0.1	0.75	0.2	5.6

(*Continued*)

Table 9.23 Continued

Year	South Africa	Turkey	USA	Argentina	Australia	New Zealand	Lesotho	Other	Total
2008	2.9	0.35	0.5	0.45	0.2	0.05	0.75	0.1	5.3
2009	2.6	0.3	0.5	0.7	0.2	0.1	0.75	0.2	5.3
2010	2.3	0.170	0.48	0.7	0.180	0.05	0.75	0.20	4.8
2011	2.23	0.150	0.35	0.7	0.155	0.045	0.75	0.20	4.6

Source: Cape Mohair South Africa.



9.11 The effect of age on fleece and fibre characteristics in the Angora goat.[165,169]

summer than in the winter. In South Africa, mohair obtained from the first two shearings (i.e. at 6 and 12 months) is generally classified as Kids. That obtained from the third (and also sometimes from the fourth) shearing (i.e. at 18 months and sometimes at 24 months) is classified as Young Goats and after that (i.e. from the fourth or fifth shearing, i.e. from the age of 24 or 30 months) the hair is classified as Adults. Generally, mohair from Kids is finer than 30 μm (varying between about 20 and 30 μm), that from Young Goats finer than 34 μm (varying from about 27 to 34 μm) and that from Adults generally coarser than 34 μm (but ranging from about 30 to 40 μm). Goats are classed as Young Goats up to the age of 3 years in Turkey. The grades of mohair vary in different countries. In general, the best grades of mohair are from Kids (e.g. Super Summer Kids) under 6 months old (i.e. first shearing). In South Africa, mohair to be classified as Kids must be 30 μm or finer, and Young Goats 34 μm and finer. Young goats and Adult goats produce about 2–2.5 kg of greasy mohair every 6 months, rams generally producing considerably more and coarser hair than ewes.[165] In the case of Kids, the fleece barely weighs 1 kg at the first shearing and is generally less than 2 kg at the age of 1 year (i.e. at the second shearing).

Mohair does not have crimp in the true sense of the word but exhibits waviness or curl. Curvature values for mohair and some other animal fibres are given in Table 9.24.[170,171] Table 9.25 shows some average values and ranges of various mohair properties.[173]

The fleece of the Angora goat, when shorn, contains natural and applied impurities; typically a total of 10–15% of non-fibre is present. The sweat or suint, the water soluble component, and grease (wax) combined are termed yolk. The grease (wax) is secreted by the sebaceous glands and the sweat (suint) by the sudoriferous glands. Other natural impurities contained in mohair include sand and dust (i.e. inorganic matter), vegetable matter (e.g. burr, grass seed) and moisture. Applied impurities include branding fluids and dipping compounds. Generally, mohair contains considerably less grease than wool (4–6% on average, compared with an average of about 15% for wool). Because the yolk content of mohair is lower than that of wool, shearers are said to have to change combs and cutters more often than with wool.

Table 9.24 Curvature and diameter values of several different animal fibres[170,171]

	Mean curvature (cm^{-1})		
Fibre	Wet	Dry	Mean diameter (μm)
White alpaca	2.0	8.4	30.2
Fawn alpaca	1.2	6.0	40.0
Lincoln	2.4	5.0	36.0
Mohair	1.2	1.4	43.6
Cashmere	6.8	12.7	13.8
Southdown	18.8	32.0	23.8
Corriedale	10.0	16.4	29.7

Table 9.25 Some average or typical values and ranges of various mohair properties[173]

Property	Range	Average/ typical value
Diameter (μm)	22–42	32
CV (%)	20–33	26
Staple length (mm)	80–140	110
Medullation (%)	0.3–2.8	1.0
Curls per 10 cm	2.5–6.5	4.5
Vegetable matter content (%)	0.1–1.7	0.3
Grease (%)	2.0–8.0	4.5
Suint (%)	1.8–4.2	2.8
pH of suint	3.3–6.2	5.2
Scoured yield (%)	75–95	85
Compressibility (mm)*	10–13	11

*SAWTRI compressibility test.

Mohair, by virtue of its open fleece structure on the goat, is more exposed to weathering than is wool, the tips of the mohair fibres covering the back of the animal being damaged by sunlight or weathering, especially during the summer months.[172] This damage has an influence on the dyeing properties of the affected fibre part. Its wax is also more oxidised than that of wool,[174] making it more difficult to remove during scouring.[169] Ilse[175] compared the composition of mohair, Karakul and Merino wool waxes as shown in Table 9.26 and concluded that the mohair and Karakul waxes had the usual Merino wax components in surprisingly similar proportions.

Mohair from Kids and Young Goats contains more grease than that from Adults, with the grease content higher in winter than in summer[176,177] and also higher towards the root (e.g. tip = 2.0%, middle = 4.6% and root = 6.0%). Uys, quoted by Kriel,[177] found an average grease content of 4.5% for summer hair and 5.8% for the winter hair, with a melting point of 39ºC. He found the acid value to be 14.6 compared with a published value of 14. The unsaponifiable fraction was 46%. Kriel[177] published values (given in Table 9.27) for the chemical constants for mohair grease.

Fibre dimensional and tensile properties

Fibre diameter (fineness) is generally the most important textile quality aspect and price determining factor by far of animal fibres, such as mohair. The finer the fibre generally the more sought after and expensive it is. Fibre fineness determines processing behaviour and performance and product type and quality, having a major influence on the finest yarn and lightest

Table 9.26 Characteristics of the waxes[175]

Characteristics	Merino wax	Mohair wax	Karakul wax
Wax content of the fleece (%)	14–16	5	3
Saponification value (mg KOH/g)	92–102	128	110
Acid value	4	14	9
Hydroxyl value	54	57	58
Iodine value	15–30	36	56
Acids (%)	49	55	50
Unsaponifiable material (%)	51	45	50

Table 9.27 Chemical constants for mohair grease[177]

Characteristics	Value	Literature
Saponification value	126–135	128
Acid value	14.6	14.0
Iodine value	14.8	36
Percentage acids	54	55
Percentage unsaponifiable fraction	46	45
Ester value	117	114

The experimental values of Kriel[177] are shown in the column 'Value'.

Table 9.28 Average values of coefficient of variation (CV) of fibre diameter corresponding to different mean fibre diameters[178]

Mean fibre diameter (μm)	CV of fibre diameter (%)
25	30
30	27
35	26
40	27
45	29

fabric which can be produced, as well as the handle and against body comfort of the fabric.

Hunter *et al*.[178] studied the diameter and variation in diameter, as measured by projection microscope, of some 852 samples of raw and scoured mohair and 380 mohair tops. They found that, although standard deviation tends to increase with increasing mean fibre diameter, the relationship was a tenuous one and the scatter large. There was a tendency for CV to decrease as mean fibre diameter increased up to a mean fibre diameter of somewhere around 35 μm, after which the reverse occurred. For most practical purposes, however, the CV of diameter could be regarded as largely independent of mean fibre diameter, with an average value of approximately 27%. Table 9.28 gives average (typical) values for CV of fibre diameter.

Wang *et al*.[179] showed that there was a relationship between coefficient of variation (CV) of mohair fibre diameter and CV of single fibre strength as predicted theoretically. Fibre length is of secondary importance to fineness, having an important effect on the processing route and behaviour and on yarn quality. Fibre tensile properties are also important from a textile point of view, fibre strength playing an important role in fibre breakage during mechanical processing, including spinning, yarn strength, fabric manufacturing and in the ultimate strength of the fabric. Generally, in the case of animal fibres, fibre strength increases almost linearly with the fibre cross-sectional area, more particularly the cross-sectional area of the thinnest (generally the weakest) place along the fibre. Therefore, fibre diameter, more specifically, that at the point of break, often the thinnest place along the fibre, has the main effect on the absolute strength of the fibre (i.e. uncorrected for the fibre cross-sectional area). The fibre strength divided by the fibre cross-sectional area at the thinnest place or point of break (i.e. the intrinsic strength or intrinsic tenacity) is fairly constant for mohair.

Smuts *et al*.[180] found that mohair generally had a higher single fibre tenacity, initial modulus and extension at break than wool of the same diameter (Table 9.29). The mohair cross-section corrected tensile characteristics (i.e. tenacity) were fairly constant over the whole range of diameters, probably because of the absence of crimp and variations in crimp and associated fibre

Table 9.29 Average values for some tensile properties of wool and mohair*[180]

Property	Mean	SD	CV %	Range	*n*
Wool†					
Fibre diameter (μm)	22.7	3.3	15	18.1–33.1	56
Linear density (dtex)	6.6	2.0	30	3.5–12.8	56
Staple crimp (cm^{-1})	4.2	1.2	27	1.9–6.5	56
Resistance to compression (mm)	17.5	2.8	16	13.6–24.7	56
Bulk/diameter ratio (μm/mm)	0.79	0.19	24	0.41–1.29	56
Tenacity (cN/tex)	12.7	0.9	7	10.9–15.0	56
Initial modulus (cN/tex)	290	27	9	230–392	56
Extension at break (%)	37.0	2.6	7	31.5–41.2	56
Mohair					
Fibre diameter (μm)	32.1	5.8	18	20.7–44.3	29
Linear density (dtex)	11.9	3.3	28	5.8–20.1	29
Tenacity (cN/tex)	16.7	0.7	4	14.6–18.1	29
Initial modulus (cN/tex)	407	13	3	384–430	29
Extension at break (%)	42.7	2.1	5	38.0–45.8	29

* 20 mm test length and rate of extension 20 mm/min.
† Low crimp wool excluded.

Table 9.30 Typical tensile properties of mohair[181]

Property	Bundle test*	Single fibre test
Tenacity (cN/tex)	14.0	16.7
Extension (%)	14.6†	43.0
Initial modulus (cN/tex)	–	407

* Leather linings were used and the tenacity values were multiplied by a correction factor of 1.16.
† The bundle test is not considered to give reliable extension values.

characteristics. Lustre wools (e.g. Lincoln and Buenos Aires) had tenacities and initial moduli close to those of mohair.[180]

Hunter and Smuts[181] found both bundle and single tenacity to be independent of mohair fineness, although the initial modulus increased slightly with an increase in fibre diameter (Table 9.30).

Fibre stiffness

The static bending and extension moduli of mohair fibres are similar, and of the order of 308 cN/tex,[182] with the optical cell densities in the medullae of kemp fibres affecting the bending but not the extension moduli. For kemp, having a virtually empty medulla, the bending and extension moduli were similar at about 77 cN/tex, whereas for the kemp with a virtually filled medullae, the bending modulus was about 365 cN/tex, which was higher than that of mohair.[182] The extension moduli of the two types of kemp fibres were similar, indicating that any material in the medullae did not contribute significantly to the tensile properties of the fibre, confirming the results of Hunter and Kruger.[183,184]

Fibre surface and frictional properties

Mohair, wool and hair are covered by a layer of sheet-like hardened cuticle cells (epidermal scales) which overlap each other, with their exposed edges towards the tip of the fibre. The cuticle plays an important role for the whole fibre because it is, on the one hand, exposed to environmental influences and, on the other hand, responsible for the surface properties of the fibre. Although, under a microscope, mohair is similar in appearance to wool, the epidermal scales (cuticle scales) of mohair are generally less pronounced and only faintly visible. The scale structure described above is responsible for mohair's smooth handle, high lustre, low against-scale friction and low felting propensity. The cuticle scales are quite thin and flat,[185] generally being less than about 0.6 μm (typically 0.4 μm) in thickness and hardly overlap.[172] They are anchored much more closely to the body of the fibre,[172,186–188] i.e. they lie near to the stem or are piled more tightly upon one another,[189] giving the fibre its characteristic lustrous and smooth appearance.

In general, mohair has a relatively low-scale frequency, with a wide distance between the cuticle scale margins. The number of scales per 100 μm is generally in the order of 5 compared with between 9 and 11 in fine wools, with the scale lengths ranging from 18 to 22 μm. In the case of kemp, the number of scales per 100 μm is 10 or more, which is twice that for mohair; and they are arranged in a coronal or ring pattern, with smooth margins.[172] The width to length ratio of mohair fibre scales is of the order 2.[190] Ryder and Gabra-Sanders[191] found that the width to length (W/L) ratios of scales from various goat fibres showed a clear sequence from the wild ancestor (*Capra aegagrus*) to mohair. They defined the scale width as equal to the fibre diameter. Indications were that the W/L ratio was independent of fibre diameter.

As in the case of wool, mohair fibres have a lower friction when rubbed from the root to the tip (i.e. with the scales) than when rubbed in the opposite direction (i.e. from tip to root, termed *against scale*). The low against-scale friction of mohair, relative to wool (Table 9.31),[192] which is one of its distinguishing features, can be largely attributed to its relatively smooth (unpronounced) scale structure. Mohair has a very small directional friction effect (DFE), due to the extremely easy deformation of the thin distal edges in mohair and also to the absence of tilted outer surfaces and other high

Table 9.31 Fibre frictional properties[192]

Fibre	μ_2	μ_1	$\mu_2 - \mu_1$	$\mu_2 + \mu_1$
Wool	0.40	0.22	0.18	0.66
Mohair	0.23	0.15	0.08	0.38
Human hair	0.19	0.09	0.10	0.28

Note: All measured in distilled water against felt.
μ_1 with-scale, μ_2 against-scale.
Source: Frishman *et al.*, quoted by Harris.[192]

asperities. The against-scale (μ_2) to with-scale (μ_1) friction ratio of mohair is about 1:1 compared to about 1:8 for Merino wool.[193] The 'scaliness' ($(\mu_2-\mu_1) \times 100\%/\mu_1$) of mohair, measured dry, is about 5 compared to about 60 for a fine Merino wool (Speakman and Stott, quoted by Onions[193]). When measured wet, the respective values are about 16 for mohair and 120 for Merino wool. It is these characteristics which give mohair its low felting propensity.

Moisture related properties

Although mohair, as in the case of wool, can absorb large quantities of moisture (up to about 30%) without feeling wet or damp, its surface is naturally water repellent, largely due to the presence of a strongly bound thin surface layer of waxy or lipid material which requires strong chemical action to remove it. The moisture-related properties of textile fibres are extremely important as they play a crucial role in the comfort of the fibre and in its behaviour during wet treatment and drying. Temperature and moisture also play an important role in the visco-elastic properties of wool and mohair, which in turn is related to wear properties, such as wrinkling.

Speakman[194] published a table (Table 9.32) illustrating the absorption and desorption of moisture by wool and mohair at different relative humidities. Watt presented a comparative table (Table 9.33) of equilibrium water content (regain) for seven keratins, including mohair.

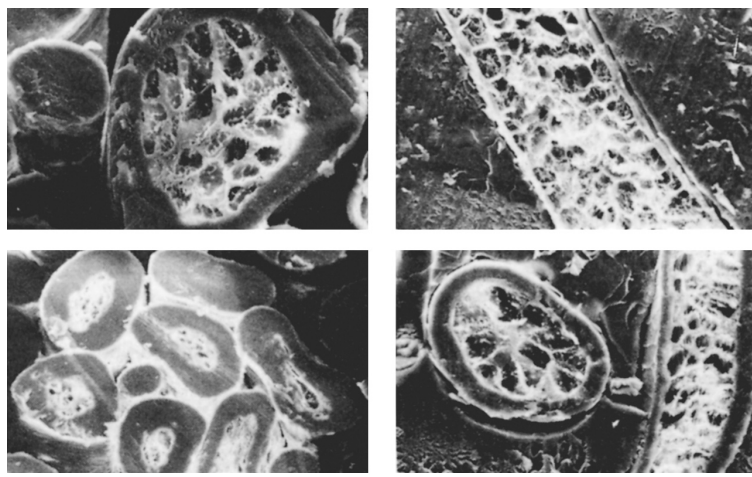
Table 9.32 The absorption and desorption of moisture by wool and mohair at different relative humidities[194]

	Percentage increase in weight of wool					
Relative humidity (%)	Geelong 80s Merino	South down	Oxford down	Leicester	Wensley-dale	Mohair
Absorption						
7.0	3.40	3.37	3.17	3.40	3.46	3.41
25.0	6.96	6.90	7.03	6.96	7.01	6.93
34.2	8.41	8.62	8.79	8.54	8.67	8.64
49.8	11.22	11.48	11.68	11.44	11.59	11.51
63.3	13.97	14.19	14.41	14.46	14.51	14.41
75.0	16.69	17.03	17.30	17.43	17.44	17.33
92.5	23.81	24.17	24.49	24.59	24.90	24.24
100.0	33.3	32.9	35.3	32.9	33.9	31.8
Desorption						
92.5	24.70	25.70	26.33	25.98	26.13	25.82
75.0	18.69	18.79	19.05	19.02	19.16	18.91
63.3	16.12	16.16	16.43	16.28	16.46	16.26
48.7	13.36	13.38	13.47	13.39	13.46	13.46
34.2	10.57	10.55	10.64	10.58	10.63	10.68
7.0	4.77	4.73	4.83	4.79	4.76	4.87

Table 9.33 Equilibrium water contents for seven keratins at 35°C (in percentages)

Relative humidity	Merino wool	Corriedale wool	Lincoln wool	Mohair	Monkey hair	Horse hair	Rhino horn
5	2.6	2.5	2.5	2.5	2.2	2.3	2.5
10	3.9	4.0	4.0	3.7	3.3	3.5	3.8
20	5.9	6.1	6.1	5.7	5.1	5.5	5.6
35	8.6	9.0	9.0	8.3	7.5	7.9	8.4
50	11.3	11.8	11.5	10.7	10.0	10.7	11.4
65	14.4	15.0	14.5	13.7	12.4	13.8	14.8
80	18.6	19.6	19.2	17.5	16.3	18.2	20.1
90	23.6	25.0	25.4	22.2	21.4	22.7	28.0
95	27.7	28.2	29.7	26.1	24.9	26.9	35.5
100	34.2	33.5	36.0	32.3	30.0	32.8	49.0

Source: Watt.



9.12 Cross-sections and longitudinal sections of medullated fibres illustrating the cellular nature of the medullae.[2]

Medullation and kemp

Medullated fibres in mohair can be a source of problems in many end-uses when they differ in appearance from the rest of the fibres which are not medullated. They are characterised by having a central canal (medulla) containing cell residues and air pockets, running in either a continuous or fragmented form along their length (Fig. 9.12[2]). The term 'kemp' is probably more familiar, but this traditionally refers to the more problematic and extreme form of medullated fibres (more recently referred to as 'objectionable medullated' fibres), which are clearly distinguishable by the naked eye, due to their chalky white appearance. Kemp is usually straight, and oval in

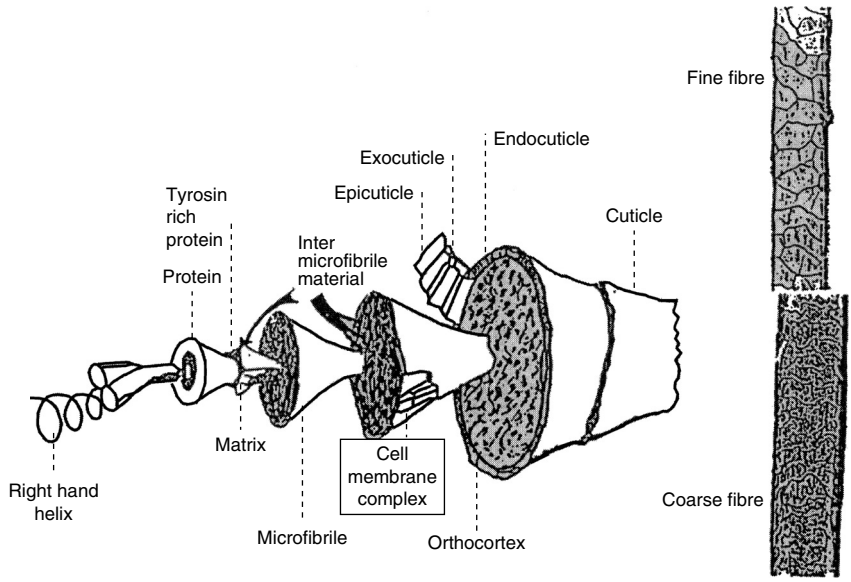
cross-section. Of all the types of medullated fibres that occur in both wool and mohair, those collectively called kemp, which tend to have a relatively large medulla and to be relatively coarse, are the most visible and unwanted in the final product. Kemp occurs as short kemp, long kemp and hetero-type fibres. The 'short kemp' is generally the most common, being short, chalky white, medullated, and pointed at each end when it has fallen out and has not been shorn off. Kemp or 'objectionable medullated fibres' tends to be much coarser than the parent population (on average 1.8 times coarser than the mean fibre diameter of the parent population[195]).

Good quality mohair, such as Cape Mohair, contains very few objectionable (kemp type) fibres, and the presence of even a small amount of such fibres in a high-quality mohair can have a pronounced adverse effect on its value and price. The main problems associated with the presence of kemp (objectionable medullated fibres) are their chalky white appearance and their lighter appearance after dyeing. The chalky white appearance is largely caused by the decreased length of the light path through the dyed fibre material and light refraction at the fibre/medulla interface and within the hollow network of cells (aerian vesicles). This, and not a difference in the dyeability of the solid fibre material (i.e. fibre wall), is considered to be the main cause of the different (paler) appearance of kemp fibres after dyeing.

Fibre chemical, morphological and related structure and properties

The reader is referred to excellent and detailed reviews of this subject by Zahn,[188,196] Spei and Holzem[197] and Tucker *et al*.[8,198] Zahn *et al*.[199,200] reviewed the chemical and biological composite structure of wool, including mohair.

Mohair falls into the class of protein materials known as keratins characterised by its long filament-like molecules and insolubility in dilute acids and alkalis. Keratin can be regarded as a long fibrous composite, comprising crystalline, relatively water-impenetrable micro-fibrils, lying parallel to the fibre axis and embedded in an amorphous, water-penetrable matrix.[201] They generally have a high sulphur content when compared with other proteins.[202] All mammalian keratin fibres contain three main protein fractions,[203] termed low-sulphur, high-sulphur and high-tyrosine proteins, with the low-sulphur proteins generally representing the largest proportion. All animal fibres contain approximately 3–4% sulphur, largely as cystine. The mohair fibre generally consists of a cortex (cortical cells), the solid and main part or bulk of the fibre, which is predominantly ortho-cortex (cortical cells), and epidermis (cuticle cells) of numerous overlapping scales. The cuticle scales form a protective covering for the cortex and consist of three layers; epicuticle, exocuticle and endocuticle (see Fig. 9.13).[204] Each cuticle scale is enveloped by a thin semi-permeable[71,72] membrane called the epicuticle, which comprises protein and lipid.



9.13 Structure of adult mohair fibre.[204]

For further details and discussion of the physical and chemical composition of mohair see the references [1 and 2].

Objective measurement and trading

In practice, the quality of mohair is described as a combination of style and character, freedom from kemp, lustre, handle, yolk and uniformity of length and fineness.[205] The presence of kemp is often the most undesirable quality characteristic of mohair. Handle is largely determined by fineness, although a soft natural yolk and oleaginous dips also improve softness of handle. Mohair characteristics of economic importance are fineness (fibre diameter), length, style and character, contamination (kemp, coloured fibres and vegetable matter), and clean yield, lustre and uniformity in general. Fibre diameter is particularly important, as is the presence and level of kemp, with length having a smaller, though still important, effect on price and processing.

Style and character are judged subjectively, high-quality style being described as solid-twisted ringlets (staples or locks), while character is described as the waviness or crimp shown in the staple.[165,169] Style without character or vice versa is undesirable, and a good balance between these two characteristics is considered to be of paramount importance.[165,169] Table 9.34 is an attempt to consolidate and rationalise some of the different systems of quality, fineness and grades encountered in the literature.

Table 9.34 Some approximate quality types[1,2]

Spinning count									
Tex	Worsted	English grades	Fineness/quality Bradford count	Age group	Crimp* per 10 cm	Maximum mean diameter (μm)	Mean fibre diamter (μm)	Description	Age (years)
14.5–15.5	58–60s	—	8	Kids	6.5–8.0	25	<26	SSK	$\frac{1}{2}$
16	56s	Kid	7	Kids	5.5–6.5	28	26–28	SWK	1
16.7–17.5	50–54s	30	6	Kids	5.5	30	29–30	WSK	—
—	—	—	6/5	—	—	32	—	—	—
18.5–19.5	46–48s	32	5	Young Goat	5.0–5.5	34	31–34	SYG	$1\frac{1}{2}$
20	44s	34	4	Adult	4.0–5.0	36	35–36	SWH	2
22–24.5	36–40s	36	3	Adult	3.0–4.0	39	37–39	SSF	$2\frac{1}{2}$
24.5–27.5	32–36s	38	2	Adult	2.5–3.0	—	>40	SFO	2
31.5	28s	40	1	—	1.5–2.5	—	—	WHO	2
								ARH	—
								CBH	—

* Preliminary.
SSK – Super Summer Kids.
WSK – Winter/Summer Kids.
SWH – Super Winter Hair.
SFO – Summer First and Older.
ARH – Adult (Ram's Hair).
SWK – Super Winter Kids.
SYG – Summer Young Goats.
SSF – Super Summer Ferals.
WHO – Winter Hair and Older.
CBH – Cross-bred Hair (Adult).

The textile processing performance, applications and general quality, and therefore value and price, of mohair are largely determined by the characteristics of the raw (greasy) mohair. It is therefore hardly surprising that considerable effort has been directed over the years towards the objective (i.e. instrumental) measurement of these characteristics to replace the subjective techniques traditionally used. Today, characteristics such as fibre diameter and yield can be, and often are, measured objectively with high accuracy.

Properties that need ultimately to be measured to characterise greasy mohair completely include the following:

1. Fibre diameter and its distribution (variability)
2. Yield (i.e. amount of clean fibre)
3. Staple (or fibre) length and strength, and their variability
4. Vegetable matter content and type
5. Inorganic matter content (e.g. sand, dirt, etc.)
6. Colour
7. Lustre
8. Medullation/kemp
9. Style/character

Douglas[206] discussed the advantages of objective measurement of mohair. He stated that the mohair top must achieve strict specifications to satisfy the spinning requirements such as:

- Quantity of top
- Mean fibre diameter
- Mean fibre length
- Maximum percentage of:
  - Short fibres (shorter than 30 mm)
  - Dark fibres
  - Vegetable matter specks
  - Entanglement (Neps)
  - Fatty matter
- Moisture regain
- Maximum percentage of kemp

In addition to the above, some spinners may have specifications which include:[206]

- Colour
- Distribution (CV%) of fibre diameter
- Bundle strength

Mohair base (i.e. the amount of clean dry fibre, free from all impurities, expressed as a percentage of the greasy fibre mass) is converted into the IWTO scoured yield.[206] This relates the tested yield to normal commercial yields for scoured greasy mohair. This yield is calculated from the mohair base to include all vegetable matter, standard residuals of grease and dirt, which would normally be retained in commercial scouring, and allows for a moisture regain of 17%, which means that yields of over 100% are possible.

There can be little doubt that mohair fineness (diameter) is one of its most important trading characteristics from the point of view of price and textile application and performance, with even a 1 μm change in diameter having a significant effect on price. It is therefore not surprising that mean fibre diameter, which can be measured by airflow, projection microscope, OFDA or Laserscan, is generally the main objectively measured and reported mohair characteristic, although the distribution of fibre diameter, in terms of CV, also has some textile significance. A major step forward in improving and standardising the inter-laboratory measurement of mohair fibre fineness occurred upon the introduction of Mohairlabs International Round Trials and associated issuing of Mohairlabs stamps in the early 1970s. Unfortunately this was terminated in 2003 with the dissolution of the International Mohair Association (IMA). In 2009 a similar body to Mohairlabs, called International Mohair Laboratories (IMLABS), was established in South Africa and became functional in 2011.

Turpie and co-workers[207–210] as well as others[211] reported on the calibration and application of the fibre diameter analyser (FDA)200 for the rapid measurement of mohair fibre diameter and its distribution. It was concluded that, within the ranges covered, kemp level had little effect on the relationship between FDA, projection microscope and airflow diameter values. It was found that different calibrations are required for mohair and wool on both the FDA[200] and the OFDA.

Turpie and co-workers[173,212–216] showed that the staple profile and length distribution could be used to predict the fibre length distribution of the staple and the top. The mohair staple has a very pronounced taper, indicating a fairly wide variation in fibre length within the staple. They found a reasonably good correlation between mohair staple length measured manually and that measured by the automatic staple length/strength tester.

Mohair burr and grass seed contaminants of mohair result in serious price penalties. Coloured (e.g. black or red) fibres, if present, could affect the finished cloth, particularly if light shades are dyed, and thereby the value of the mohair. Burrs or excessive vegetable matter in the fleece also have to be removed.[165] Urine and certain types of soil and vegetable matter contain substances that stain mohair permanently.[165] These affect the dyeing and the value of the mohair and the quality of the final product, Precautions must

be taken to limit such stains, particularly urine stains.[165] Clean yield (i.e. the percentage of actual fibre plus commercially allowed moisture content in raw mohair) generally varies between about 80 and 90% in most fleece classes, but may be as low as 60% in some outsorts, such as lox (locks), the remaining portion being made up of grease, dirt, dust and sweat.

9.9.2 Fibre processing

Scouring and carbonising

Scouring is a critical process in mohair production and often it is at this stage that the ultimate state of the finished article is decided. As previously mentioned, mohair generally contains far fewer impurities than does wool (e.g. 4–6% of grease compared to about 15% for Merino wool) and scouring generally causes a loss in mass of between 15 and 20%. Mohair is generally regarded as more sensitive to alkali than wool. Therefore, less, or even no, soda-ash (alkali) should be used during scouring, non-ionic detergents being preferred today.

Before scouring, individual mohair bales are often sorted on screens for style and quality, efficient sorting and blending playing an important role in the eventual quality of the yarn. The fibre can then be willeyed (opening/cleaning) before it is scoured, and this is advisable. Scouring conditions for mohair are generally gentler than they are for wool, and scouring rates lower; alkali not being necessary, the pH must be strictly controlled. Excess alkali in the fibre can lead to discoloration in dyeing. Care must be taken during scouring not to impair the lustre of mohair. Kriel,[174,217] quoting unpublished work by Veldsman, stated that a higher consumption of detergent was required to remove 1 g of grease from mohair than from wool. The relevant factors were the generally lower level of grease in mohair as well as its more oxidised nature, because of greater weathering than in the case of wool.

For the continental worsted system (French or rectilinear comb) of processing, which is very popular today, scouring to a residual grease content of 0.2–0.3% is advisable, with a total fatty matter level of between 0.7 and 0.9% (up to 1.2% for flexible card clothing) prior to carding.

Very little mohair (±2%) is normally classified as carbonising type, although in high rainfall areas and seasons it can rise to as high as 15%; mohair with vegetable matter exceeding 3% is normally carbonised.

Mechanical processing

Mohair is considered to be difficult to mechanically process because of its smoothness and lack of cohesion. Nevertheless, provided the correct

processing additives (lubricants and anti-statics) and conditions and raw materials are used, very high quality mohair yarn can be spun with acceptable efficiencies. In converting mohair into yarn, similar machinery is used as in the case of wool. Considerable secrecy exists even today concerning the precise processing conditions used; firms which have built up this specialised knowledge and skills do not share it because it provides them with a competitive edge.

It is generally easier to disentangle mohair than wool during carding, with less fibre breakage in this process, although problems with static and fly generation often necessitate lower carding speeds. Mohair's low cohesion often necessitates that the fibres (slivers) be supported, for example by aprons, during processing. Mohair blends well with wool, the wool facilitating its processing, by increasing inter-fibre friction and cohesion. The application of the correct types and levels of processing lubricants and additives (such as anti-statics) and the selection of the most appropriate processing machinery and conditions (including atmospheric) are crucial in the efficient processing of mohair into a quality product.

Traditionally, mohair was processed on the Bradford worsted (oil-combed) system (drafting against twist) followed by flyer spinning.[218] Today, the bulk of mohair is processed on the continental or dry-combed (French/rectilinear combing), as opposed to the oil-combed, system. The French (continental or dry-combed) system of drafting and spinning involves French (rectilinear) combing, intersecting gilling and double apron drafting (drawing). It is possible to use either flyer (twisted) roving or rubbed (twistless) roving for subsequent yarn spinning. Most of the shorter mohair and also a significant amount of longer hair as well as mohair waste, such as carbonised noils, are processed on the woollen system. For the woollen system, a minimum amount of vegetable matter is essential. In woollen spinning, mohair shorter than about 75 mm staple length is generally used while for the worsted system the staple length is generally 90 mm and longer, with a staple length of some 120 mm often required.

The finest yarn which can be spun largely depends upon the mohair fibre diameter or fineness, traditionally expressed in terms of 'quality or quality counts', and these are related to the minimum number of fibres in the yarn cross-section. Wang and Khan[219] have reported on the use of pinned apron and bottom roller for the improved drafting of mohair during ring spinning.

Fancy (novelty) yarns

Mohair is used to particular advantage in fancy or novelty yarns, such as loop, knop, brushed, bouclé, flame, snarl, slub and gimp, where its properties provide outstanding aesthetic appeal and comfort. Such yarns are used in

blankets, stoles, shawls, scarves, knitwear (sweaters, cardigans, jerseys, etc.), travel rugs, curtaining, table coverings, upholstery, furnishings, pram covers, women's dress-wear, suitings and coatings. Adult hair is often used to form the loops of bouclé yarn properly.[220]

Fabric production and machinery

Generally, mohair yarn is converted into knitted and woven fabrics using similar equipment as for wool, though sometimes in a modified or adapted form and under special conditions, which allow for the more hairy, and often weaker, nature of the yarns.

Dyeing and finishing

Dyeing and finishing represent crucial stages in the manufacture of mohair products of the outstanding quality and appearance associated with items bearing the label 'mohair', acid and metal complex dyes being popular. It is generally the case that firms which dye and finish mohair also dye and finish wool and hence similar machinery is used for the two fibres. Furthermore, it is very rare to find pure mohair in yarns and fabrics. It is mostly present in blends with wool, often yarn blends, which means that the dyeing and finishing machinery and conditions used must be suited to both fibres.

There is a vast literature on the dyeing and finishing of wool, much of which is to a large extent also applicable to mohair. There is far smaller literature available on the specialised knowledge of conditions and procedures required for the dyeing and finishing of mohair products because most of such knowledge is a well-kept secret. In general, milder conditions (temperature and time) are used for the dyeing and finishing of mohair than for wool, partly because of the need to conserve the lustre of mohair and partly because mohair is more sensitive to wet treatments than wool. It is common practice to dye at temperatures below the boil, preferably below 90ºC, and to limit the time of dyeing at high temperatures, so as to curtail any adverse effects on lustre and other desirable properties. It is also possible to limit damage to the fibre by using fibre protective agents.

9.9.3 End-uses

The textile application of mohair goes back many thousands of years and the fibre has found application in almost every conceivable textile end-use. Today, up to 80–90% of mohair consumption, especially of the Adult hair, can be affected by fashion.

Mohair, often in blends with other natural fibres, notably wool, with which it blends well, is used to great advantage in knitwear (ladies sweaters)

Table 9.35 Mohair consumption by end-uses[221]

End-uses	Share (%)
Hand-knitting yarns	65
Men's suiting fabrics	15
Women's woven accessories and rugs	12
Woven furnishings and velours	8

and hand knitting, mostly in brushed, loop or some other fancy form where brushing imparts softness and warmth without weight. Knitwear traditionally represented some 80% of mohair's outlets, but this sector is fairly sensitive to cyclical fashion changes.

Mohair also finds significant application in woven suiting and coating type fabrics, particularly in men's lightweight summer (tropical) suits where it provides the wearer with considerable comfort and good wrinkle resistance. Mohair is widely recognised as having very good wrinkle resistance and recovery, which, together with its stiffness, make it an ideal fibre for use in comfortable lightweight tropical type fabrics.

Table 9.35[221] lists some of the end-uses of mohair, and a detailed list of mohair applications is given by Hunter.[1,2] In lean worsted-type lightweight tropical suits, mohair is regarded as a cool fibre, whereas in brushed articles, such as shawls, stoles, rugs, sweaters and blankets, mohair provides warmth without weight. Mohair's characteristics of hard-wearing durability, resilience or springiness, crease resistance, moisture absorption, comfort, lustre and smoothness make it ideally suited to many applications in apparel and interior textiles, such as upholstery and any pile fabric (e.g. plush, velour, velvet and moquette, etc.), furnishings, rugs and curtains and it is virtually unsurpassed for general durability, recovering very quickly after being crushed. Because of its general smoothness and low static propensity, except under dry conditions, the smooth fibres do not allow dirt to collect readily, and stains are generally fairly easily removed.

Mohair, in blends with wool, bamboo and other fibres, has found significant application in comfort socks, particularly for active sports wear (e.g. cricket, mountaineering, etc.) and medical applications (e.g. for diabetics).

Further information on mohair is given in the references. [1 and 2]

9.10 Musk-ox

9.10.1 Fibre production, harvesting, properties, processing and end-uses

The musk-ox (O*vibos moschatus*), a hollow horned ungulate (ruminant), native to the Arctic, is in fact not an ox, and has no musk, its nearest relative

9.14 Musk-ox. (By kind permission of Ms Nancy Bender, The Musk Ox Company, 633 Hatchery Road, Hamilton, Montana 59840, USA.)

being thought to be the goat.[222] It reportedly has the largest and thickest hair of any mammal on earth.[223] The prehistoric animal, also called 'omingmak' by the Eskimo people, is huge, and looks like a Bison[223] (see Fig 9.14). Adult male animals have an average weight of some 360 kg (up to 450 kg), and a shoulder height up to 1.8 m.[222] It originally bred wild and undomesticated in the Northern territories (Arctic lands) of Canada, and wild animals are still found in Canada's Northwest Territory and in Greenland.[222] The musk-ox essentially has a two layered fibrous coat, namely a relatively coarse, dense and long (up to 600 mm) guard hair (outer coat, dark brown in colour) and a relatively fine (≈ 13–17 μm) and short (40–80 mm) undercoat (brown-grey) of down fibres, known as qiviuk; the latter has been gathered and used since time immemorial by the Eskimo people.[223]

The musk-ox skin has an exceptionally high secondary primary follicle ratio between 40:1[224] and 37:1 (Rowell *et al*.[225]), the qiviut being primarily produced by the secondary follicles and the guard hair by the primary follicles. Rowell *et al*.[225] reported, however,[225] that there appears to be an intermediate (medium) group of fibres, coarser than qiviut but finer than the guard hair, variable in diameter and produced by the primary follicles, these being easily visible in combed fleeces. They are fine, like qiviut, near their roots and coarsen considerably towards the tips.[225] They are shed annually and can be medullated.[225] This group of fibres is believed to account for most of the fibres between 30 and 50 μm,[225] and is important in terms of the de-hairing process.

The musk-ox became extinct in Alaska around 1850,[224] but was re-introduced around 1953. The Canadian government placed the animals, which are easily hunted, under some protection in 1926, and in the 1930s the

US Department of Interior obtained animals from Greenland and released them on the uninhabited Nuaivak Island in Alaska.[222] First attempts to domesticate the musk-ox for its fibre occurred in the 1950s. The calves were captured in game sanctuaries in the North West Territories and the first successful musk-ox farm was established in Alaska during the 1960s,[225] when fibre production also started. In the 1980s there were two domesticated herds, one at College, Alaska near Fairbanks, and the other in Northern Quebec.[222] In a joint venture between the United States and the USSR, around the 1980s, musk-ox calves were captured at Nuaivik Island and sent to the Soviet Union, the latter attempting to re-establish the musk-ox in Siberia.[222] Originally mainly hunted for their fat-rich meat, the musk-ox is now mainly valued and bred for its fibre.[226,227] Selective breeding improved the fibre colour and yield and animal domestication, and reduced the size of the animal, thereby increasing its cost effectiveness.[228] In recent times, commercial harvesting of fibre from wild musk-oxen on Banks Island and elsewhere in the North West Territories of Canada has made available relatively large quantities of de-haired qiviut fibre, yarn and finished products,[225] fibre harvesting taking place semi-regularly since around 1982.

The musk-ox sheds its soft 'silvery grey/white' undercoat in a tightly synchronised moult each spring,[225] from around April to June (i.e. around late spring, with temperature playing a role).[228] The animals are placed in pens and the fibre is readily and easily combed out or plucked (pulled off) by hand, over a number of days, the undercoat being easily removed in sheets, which continues until early June.[228] The total collection (plucking) time per animal is slightly more than 2 h.[228] Fibre is also collected from objects against which the animals brush and which retain the fibre, the undercoat being found in great tufts on the bushes of the boreal tundra[223] where the animals graze. The guard hair is separated and removed by hand from the fine down.

The fibre removed from the musk-ox is referred to as raw fibre (i.e. containing a mixture of qiviut and hair).[225] Adult animals annually yield between about 2.5 and 3.5 kg of hair (raw fibre),[222,225] yielding about 2 to 3 kg of qiviut.[226] Females produce about 2 kg of qiviut, while mature bulls yield more fibre than females and younger animals.[228] For the purposes of their study Rowell *et al*.[225] defined qiviut to be all fibres ≤ 30 μm. The fine undercoat (qiviut) of the musk-ox has an average fibre diameter of about 20 μm,[31] numerically, more than 90% of fibres in a 'raw' sample being ≤ 30 μm.[225] Rowell *et al*.[225] reported that the average diameter for raw fibre shorn from the shoulder region of musk-oxen was 21.5 μm for adult males and 19.5 μm for 2-year-old females, and that of the qiviut (taken to be fibres ≤ 30 μm) 16.5 μm for yearling males and 18.2 μm for adult males. The fibre diameter for both the raw fibre and qiviut increased slightly with age. For example, both the raw fibre and qiviut increased by about 1 μm from 1 year to 4 + years of

age. The CV of diameter of the raw fibre was around 65%, and that of the qiviut around 27%,[225] the CV of diameter decreasing with age, for both the raw fibre and qiviut. The female animals tended to have slightly lower mean fibre diameters than the male animals, except at the age of 1 year.[225] The root section of the fibre is generally 1–2 μm finer than the middle section of the fibre.[225] Guard hair and qiviut staple length increases slightly with age, averaging around 120 and 52 mm, respectively, for the shoulder region of the musk-ox. The relatively low qiviut staple length (±50 mm) reported by Rowell *et al*.[225] was attributed to both their sampling site (shoulder) and the fact that the fibres were shorn (i.e. the fleeces were cut from hides) and not shed.[225]

Qiviut fibre quality is judged technically in terms of length and diameter, the fibres being similar in diameter, on average, to cashmere, and considerably longer.[222,228] Musk-ox hair can be de-haired on the same machines as cashmere.[228]

Musk-ox fleeces are virtually free from contamination, such as vegetable matter and dirt, and contain very little grease (< 7%) and suint,[225] it being almost possible to spin it directly from the animal's back.[228] Rowell *et al*.[225] reported an average scoured yield of about 93% for 3-year-old and adult musk-ox, increasing only slightly with age.

Alaskan Inuit (Eskimo) women hand knit caps, scarves, stoles, sweaters, shawls and tunics from qiviut,[222,229] about 115 g of qiviut fibre being sufficient for a sweater for a large man.

The fibre bleaches and dyes well, with garments made from musk-ox fibre being very light and comfortable, and highly suitable for temperatures below freezing. Garments made from musk-ox fibre reportedly do not shrink when washed.[226]

9.11 Vicuña

9.11.1 Fibre production, harvesting and properties

Vicuña (Lama genus: *Vicugna vicugna*), a ruminant (pseudo-ruminant) descended from the guanaco, is the smallest (0.7–0.9 m in height and weighing about 50 kg) and most timid member (see Fig. 9.15) of the South American Camelid family,[80] producing the finest and softest under-hair of any animal. Its fibre, sought after for around 10 000 years,[230] has been termed[231] 'Fibre of the Gods' and 'Silk of the New World'.[232] The individual fibres range from about 6–35 μm in diameter and 12–65 mm in length, the chest hairs being longer and lighter (almost white) in colour.[13] Only some 5000 kg of 'raw' (harvested) fibre is available annually,[233] which is reduced to almost half after scouring and de-hairing.[233] The vicuña originally had to be killed to obtain its fine coat of fibres,[234] although, reportedly,[235] the Incas

9.15 Vicuña. (Reproduced from Pier Giuseppe Alvigini, *The Fibres Nearest the Sky*, Mondadori Editore, Verona, by kind permission of Mr Pier Alvigini at Alvigini S.A.S., 13900 Biella Via Dante, 12 Casella Postale 430, Italy.)

some 500 years ago had captured, shorn and released thousands of vicuñas. During the time of the Inca empire, around 1400 AD, only the royal family and the handful of youthful ladies, chosen by the sovereign as ladies-in-waiting were allowed to wear vicuña garments.[232]

Vicuña is native to the central Andean countries of Peru and Bolivia (also Chile and Argentina), living in the Cordelleros of Peru and surrounding country, at altitudes around 3500–6000 m, and grazing on ichu grass.[12,232] They can exist at higher altitudes than other members of the Camelid family.[230] Millions of vicuña once roamed Peru's 4000 m high Altiplano. During the pre-Columbian Inca civilisation, vicuñas were hunted only once every 4 years.[236] After the Spanish invasion in the early 1530s, the vicuña population dropped dramatically, to only some 5000 in Peru in the mid-1960s,[236] due to uncontrolled hunting for their fibre and meat. Simon Bolivar passed the first law protecting vicuñas in 1825.[236] Peru passed a similar law in 1921, while a South American Agreement was introduced in 1969 which prohibited the commercial use of vicuña. The number of animals increased from some 5000 in Peru in the mid-1960s to between 10 000 and 15 000 by the end of the 1960s,[237] these being kept in the area Pampas Galeras (the 15 000 acre Pampas Galeras Naturel Reserve established in 1967), started as a programme of conservation and managed by the Peruvian government,[234] resulting in the vicuña numbers there increasing to 38 000 by 1978, and the world population to 60 000.[234]

The vicuña was considered endangered between 1960 and 1990.[238] According to one report,[239] the vicuña had been hunted to virtual extinction for its fleece,[239] and by the 1970s the species had virtually been wiped out in certain areas, the fabric fetching up to $1500 per square metre at the time. CONAF, Chile's forestry commission, in 1970 created the Lauca National Park in Northern Chile to protect the vicuña's natural habitat and increase their numbers. As a result of this there were some 28 000 vicuñas in the park around 1990.[239] Attempts also started at around that time to determine whether the animals could be safely trapped and shorn in the wild, leaving enough fleece to protect them from the cold.[239]

Because of the danger of extinction, export bans on fibre in Peru and Bolivia were supported by import bans in manufacturing countries.[12] In the USA, vicuña was listed as endangered on 2 June 1970,[240] reclassifying it to 'threatened' in 2002, other markets having sanctioned imports in 1995. The Peruvian government, and afterwards in 1976, the UN Convention on International Trade in Endangered Species (CITES), stepped in to make trading in vicuña an offence,[232] with a resultant dramatic increase in their numbers. In 1987 CITES changed the designation of the vicuña to Appendix II, allowing fleeces to be harvested and converted into fabric.[52] By 2001 it was estimated that they numbered over 200 000 in Peru[240] alone. In 1994 there were an estimated 66 500 vicuñas in Peru,[230] of the purest bloodline, the number thereafter increasing as shown in Table 9.36. There were estimated to be some 55 000 vicuñas in Northern Chile, Bolivia and Argentina[230] in 2000.

The project for Rational Utilisation of Vicuña, under which thousands of vicuña were culled for their fibre and meat in Peru, was terminated early in the 1980s.[241] The vicuña has never been domesticated, though bred in semi-captivity and herded. The rounding up in Peru is referred to as a Vicuña Chaccu/Chakku (also done by the Incas[230]) and the animals are shorn and released. This is called a 'Chakku'[151] in Bolivia, while the 'Compesinos' herd vicuñas for shearing in Peru.[232] In Peru, vicuña are shorn between 15 May and 15 November,[242] the total production amounting to a little over 6000 kg in 2007. Electric shearing is used where feasible, although clippers are considered preferable[243] for shearing.

Table 9.36 Vicuña population in Peru[230]

Year	Number
1994	66 500
1997	103 000
1998	120 000
1999	140 000
2000 (Est.)	165 000

The fleece of the vicuña ranges in colour from golden to typically a rich, deep fawn (cinnamon) colour, with that of the underbelly and extremities being off-white. Hair from six vicuñas is required to produce one sweater and from 35 to produce one coat.[244] Vicuñas have a double coat, a relatively coarse guard hair and a very fine undercoat or down, the most prized fibre being from the 'apron' of long whitish hair that falls between the front legs.[232] Vicuña fleece ranges from 12 to 15 μm in mean fibre diameter, and from about 15 to 50 mm in mean fibre length,[91] more typically 20–25 mm. The fine (13 μm) vicuña hair is found on the lower chest just behind the front legs and is a short (≈ 50 mm) downy undercoat, the outer hair being much coarser and longer (up to 45 μm in diameter and 65 mm in length[237]). Commercial vicuña typically ranges from 13 to 15 μm.[7] Greaves and Rainsford[7] reported little change in vicuña fibre fineness and quality from 1970 to 2005. An adult vicuña only produces about 200–250 g of fine hair every two years.[232,244]

First quality fibre averages about 37 mm in length and 12 μm in diameter,[245] and contains a small quantity of coarse and dead (kemp) hair. Second quality fibre comes from skirtings, is 3–4 μm coarser[245] and contains a relatively large proportion of coarse and dead hair.[245]

CONACS (Consejo Nacional de Camélidos Sudamericanos), the National Council for South American Camelids, is the government authority of the Ministry of Agriculture in Peru charged with commercialisation of vicuñas and the certification of fibre shorn from live animals.[246] Hair from slaughtered animals is illegal under Peruvian law.

9.11.2 Processing

Vicuña is sorted by hand to remove coarse fibres, a worker taking a week to separate (sort) by hand 1 kg of mixed fibre and down.[232] This is followed by de-hairing. Vicuña is traditionally de-haired by hand, but more recently also by machine. By hand, it can easily take seven 8-hour shifts for a sorter to obtain 700 g of fine fibre,[17] of which only a small percentage is as fine as 13 μm. Its length (typically 20–25 mm) largely limits processing to the woollen system, as opposed to the worsted system.[17] Specially collected vicuña fibres, 35 mm in length, have, however, also been processed, in 100% form, on the worsted system into blazer cloth. This was claimed to be a 'first' in textile history[247] (fibres from 5 vicuñas being required for one blazer length fabric). It was also claimed that the world's first 100% vicuña suiting from worsted spun yarn was produced in 2008.[248]

Woollen yarn as fine as Nm25 (40 tex) and worsted yarn as fine as Nm46 (≈ 26 tex) have been produced from pure vicuna fibre,[249] manual removal of kemp and impurities taking place at the fabric stage.

9.11.3 End-uses

Vicuña re-appeared on the market in the mid-1990s after an absence of 20 years. In 1994 the International vicuña Consortium won the rights to exclusively process and market vicuña globally.[17]. According to the agreement in 1995 to commercialise vicuña hair, only 100% vicuña could be used in fabrics, this being amended around 2000 to include blends with wool,[237] 80% vicuña/20% fine wool (20.5 μm) blend fabric (580–600 g per linear metre), for example, being used for skirts, dresses, scarves, jackets and overcoats.[245,250] Legally exported vicuña fabric is identified by a CITES-designated trademark 'Vicun–Andes'.[236]

Vicuña is popularly used in cardigans and sweaters and is highly sought after due to its very fine and soft nature. Scarves, parkas, throws, shawls, dresses and blankets, in 100% vicuña, also popular.

Vicuña fabric can cost US$4000 per metre,[232] and a full-length 100% vicuña overcoat costs as much as US$12 000.

In the case of woven fabrics, mainly plain weave fabrics are produced, with some common weights and end-uses as follows:[230,237]

- Average fibre length: 20–25 mm
- Yarn count: Nm 18 to Nm 40 (55 to 25 tex)
- Fabrics
  - 430 g/m for suitis
  - 550 g/m for jackets
  - 690 g/m for overcoats
  - 100 g/m (150 × 30 cm) for men's scarves
  - 190 g (180 × 30 cm) for ladies' scarves

9.12 Yak

9.12.1 Production, harvesting and properties

The yak (*Bos grunniens*), a member of the hoofed Bovidae family, is chiefly found in Tibet, living above the snowline on the Qinghai–Tibet Plateau, called 'The Roof of the World'. It is used in the mountainous region as a beast of burden,[251] and for subsistence through its milk and meat,[252] herdsmen in more recent times earning additional income from selling its hair. It was estimated that towards the end of the previous century there were some 13 million domesticated yaks on the plateau flanking the Himalayas, of which some 12 million were in China,[251] one-third of which were in the Qinghai province bordering on Tibet, making up the single biggest herd in the world.[251] Yaks were also dispersed on the vast pastures in the adjoining provinces of Sichuan and Gansu and in Tibet,[251] and there were also over

100 000 in India.[253] It was estimated that there were some 4 million wild yaks in the deep valleys and remote mountain areas. In the Himalayas, the domestic YAK is usually found at altitudes above 2000 m.

The yak weighs about 300–350 kg and its thick coat of long hair almost reaches to the ground (see Fig. 9.16). Wild yaks tend to have black hair but domestic yaks, kept by most herdsmen, have hair which is often a piebald colour, predominantly black and brown, with white markings.[251,252] The yak has a double-coated fleece, consisting of a long coarse guard hair (outer coat), and a soft down (undercoat) which it sheds annually in spring and which is either combed out by hand or shorn.[254] Based on a relatively low yield of 0.5 kg of fine fibre (down) per animal, some 7 million kg per annum of fine fibre should be produced according to the number of yaks from which hair is harvested. In fact, in 2006 the production of yak hair was estimated[254] at 7 million kg, mainly coming from China and Mongolia. A specially selected 'fibre line' of Jiulong yak can yield up to 10–12 kg of fine fibre (without coarse hair) per head which is some 10 times the normal yield (1–1.2 kg)[254] per animal.

The yak hair varies in fineness according to the part of the body from which it comes.[253] The underside of the animal is covered with long coarse hairs, which form a fringe, the outer sides of the legs being covered with the same type of hair.[253] The back none on the neck and spine as well as the occipital and frontal parts of the head are also covered with coarse, but much shorter, hair. The yak's tail is similar to that of a horse,[254] but much thicker. A steel comb is used in Mongolia to harvest the down fibre from yaks. The lower sides and belly of the animals are combed soon after the spring moult,[252] to remove the soft fine undercoat, whereas the neck, back and hind quarters are not

9.16 Yak. (Reproduced from Pier Giuseppe Alvigini, *The Fibres Nearest the Sky*, Mondadori Editore Verona, by kind permission of Mr Pier Alvigini at Alvigini S.A.S., 13900 Biella Via Dante, 12 Casella Postale 430, Italy.)

combed.[252] The hair clip varies from 300 to 900 g, at 2 years being from 400 to 500 g, and under 1 year 500–1300 g. For female yaks aged 3 years or more the figures are 200–600 g and for 2 years it is 300–500 g.[253]

Yak fine hair (down) varies in diameter from roughly 15 to 30 μm and that of the coarse guard hair from about 35 to 80 μm,[130,255] the corresponding fibre length values being about 10–60 mm, and 45–160 mm, respectively. The under-hair (down) of a 1-year-old calf has a diameter of 15–17 μm, and is 40–50 mm in length. The corresponding figures for the adult are 18–20 μm and 30–35 mm, respectively.[252]

It is laborious to remove the hair from the yak, first involving thinning the hair as summer approaches and before the yaks are put out to pasture, the soft hair being pulled from the animal by hand.[251] Shears tend to be forbidden, because they can expose the flanks of the animal as winter approaches.[251] The white hair is the most precious. Fine yak hair is mainly produced in China,[256] where the hair is pulled or combed during the spring[252] moult, and the coarse outer 'guard' hair has to be separated from the down hair thereafter. Well de-haired yak fibre can have a mean fibre diameter between 19 and 21 μm,[76] with a CV between 20% and 25%.

In India, *Clipped yak* fibre refers to fibre from the body of the yak obtained by shearing with hand shears or a shearing machine,[253] while *Pulled yak* fibre refers to fibre removed from the yak by pulling out the fibre, with or without the aid of a soap or depilatory solution.[253] *Mixed fibre* refers to a combination of clipped and pulled yak fibre,[253] with 'Lot' meaning the total quality of yak fibre offered for grading, of one colour and type only.[253] Laboratory yield percentage of yak fibres vary from 65% to 80%. According to one study[253] the percentage of foreign matter varies from about 1% to 6%, with length varying from 28 to 37 mm. A maximum of 3% vegetable matter for different grades of yak fibre has been suggested.[253]

Tentative specifications for Indian yak fibres have been proposed as follows:[253]

Colour of fibres: (a) white, (b) brown, (c) black
Texture: (a) extra fine, (b) soft, (c) coarse
Foreign matter: 2%
Moisture percentage: 3%
Length: 10 cm

Yak fibres tend to be oval to circular in shape and generally do not contain a medulla. Yak fibres have a fine scaled structure, the scales not being very prominent, and are typically deeply pigmented, with an average diameter around 20 μm. In one study[257] yak fibre scale height was found to be 0.36 μm on average, varying from about 0.21 to 0.65 μm, for an average fibre diameter of 20.7 μm, the scale frequency averaging 6.9 scales per 100 μm,

varying from about 4 to 10 per 100 μm, the figures for scale height and frequency being very similar to those found for cashmere fibres (15.6 μm mean fibre diameter) in the same study.[257]

9.12.2 Fibre processing

Yak hair does not appear to be sorted at the time of harvesting, being packed into large sacks (bags) which are sent to the warehouses or sorting factories where it is hand sorted to reduce the quantity of guard hair present in the down and also to separate the natural colours.[252] Sorters are only able to handle some 10 kg per day. After sorting, the fibre is packed into bales and sent to the processor for converting into yarn.[252]

The approximate proportions of the different natural colours are as follows:[252]

- White ≈ 10%, the most valued due to its dyeing potential
- Fawn ≈ 20%
- Dark grey (blue) ≈ 10%
- Dark brown ≈ 60%

Because fine yak hair is much cheaper than cashmere, it is economical to bleach it,[256] although bleaching tends to impair its handle. Reasons for the deterioration in handle and ways of improving it by, for example, adding a chelating agent, have been discussed.[256] The deterioration in handle was ascribed to cleavage of the disulphide bonds. Work has also been carried out[258] to reduce the diameter of yak fibres by a stretching (i.e. slenderising) treatment.

9.12.3 End-uses

The coarse yak hair (outer coat) is used locally for tents, ropes, huts, blankets and mats,[252] while the fine down fibre is used in apparel fabrics.[252] De-haired fine down fibre can be spun into yarn which is comparable to cashmere,[252] and blended with other animal hair and polyamide (nylon), is used in knitwear.[251] Yak hair is used in the felt industry and that of yak calves in the textile industry for the manufacture of high quality, generally thick, fabrics.[253]

9.13 Other animal hair fibres

9.13.1 Bison hair

The properties of hair shed by the North American bison (*Bison americanus*) have been measured[259] and it was found that the guard hairs were hollow and

ranged in diameter from 21 to 110 μm, while the fine down hair was solid (i.e. unmedullated), ranging in diameter from 12 to 29 μm, with a scale structure similar to that of wool. The moisture regain ranged from 13% to 20%.[259]

9.13.2 Cervelt (soft deer hair)

Cervelt is the soft undercoat (down) from the red deer,[260] having a mean fibre diameter of about 13 μm (CV = 18%). Each animal produces only about 20 g of fine fibre and the total world production is some 1000 kg.[260] The down fibre from about 14 deer is required to produce a sweater and from about 40 deer to produce a man's overcoat.[260] The fibre has a curl rather than crimp. It is used in menswear, women's fashion, knitwear, accessories and furnishings.[260] When the name cervelt is used it is in its pure form, i.e. unblended with other fibres.[260]

9.13.3 Common goat hair

Common goats (*Capra aegagrus hircus*) or milk goats generally have a double coat containing very little, but rather fine (≈ 15 μm), down fibre.

Debnath *et al*.[261] gave the following properties for the goat hair waste available from tanneries in India:

Average fineness (tex): 3.6–5.1
Average length (mm): 20–21
Average tenacity (gf/tex): 13–19.4
Average extension at break (%): 49–88

Paul *et al*.[262] obtained the following results on the hair from Gaddi goats:

Average fibre diameter (μm): 76.7 (range = 46–88)
Average fibre fineness (tex): 4.6 (range = 2.1–7.0)
Average medullation (%): 75
Average fibre length (mm): 101 (range 60–121)
Average fibre tenacity (gf/tex): 13.8 (range = 7.8–18.7)
Average fibre elongation (%): 26.0 (range = 2.4–39.0)

9.13.4 Horse hair

Horse (*Equus Caballus*) hair has been used since prehistoric times to make fishing lines and nets, but over the past 200 years it was also commonly used for upholstery (soft furnishing), the fabric being prized for its durability (up to 100 years).[263] It is also used in linings and handbags. Horse hair

fabrics represented one of the most traditional coverings for fine furniture, widely employed by the eighteenth-century masters, like Chippendale and Hepplewhite,[264] but then coming down market in the nineteenth century to become the favourite covering of the parlour sofas of the rising middle class.[264] It can be combined with other natural fibres, such as cotton and silk.

The horse has short (≈ 10–30 mm) and coarse (≈ 80–100 μm) body hair and much longer (≥ 300 mm) mane and tail hair. Horse tail hair generally has an average fibre diameter greater than 140 μm,[265] ranging from about 75 to 280 μm. The mane hair is finer (average ≈ 110 μm) and ranges from about 50 to 150 μm. Bolormaa *et al*.[266] reported that the Mongolian horse tail hair they tested were medullated, and had a mean fibre diameter of around 180 μm, with the diameter increasing from about 130 μm at the tip to about 230 μm at the root. The fat content (according to MNS 379:200 test method) was 2.4% and the moisture content (according to MNS 380:2001 test method) 10.5% for the greasy hair and 16.8% for the scoured hair. The breaking extension ranged between about 43% and 52%, increasing with increasing diameter. Initial breaking modulus ranged from about 2.6 to 4.9 GPa, decreasing with increasing fibre diameter. The cuticle scales of the hair were smooth, with the height (thickness) of the cuticle scale edge lower than that of speciality fibres.[266]

9.13.5 Shatoosh, Shahtoosh, Shah-tus, Shah-tush, Tosh

There is some confusion concerning the precise definitions, origins and sources of fibres or products variously referred to as Shatoosh, Shahtoosh, Shah-tus, Shah-tush and Tosh. Nevertheless, it appears that they are variously applied to fibre and fibre products from wild animals, notably the ibex wild goat and the Tibetan antelope.

The official taxonomy currently lists four wild *Capra* species (*aegagrus*, *ibex*, *falconeri and cylindricornis*), although there may be three or more distinct species of Ibex,[267] the only CITES-listed species being the markhor (*Capra falconeri*), with the *Capra ibex* the typical ibex.[236]

Tosh fibres are obtained from an animal (antelope?) belonging to the ibex family,[268] which is found in Tibet and the adjoining areas of the Ladakh region of India. Its fibre is collected from the bark of trees. It is mainly used to manufacture shawls in the Kashmir Valley, which are known as 'ring' or 'shahtosh'.[268] The following results were obtained on a sample of raw Tosh fibres:

Fine down yield (%): 71
Mean fibre diameter (μm): 11.9 (CV = 3.9%)
Mean fibre length (mm): 42.7 (CV = 6.2%)
Scouring yield (%): 86

The percentage of fine fibres (71%) was reportedly similar to that found for a Pashmina fleece.

The Asiatic ibex or wild goat, Siberian ibex (*Capra ibex sibirica*), also called the Yangir (wild cashmere) mountain goat, is considered to be the wild species most commonly exploited for textile fibres.[267] It lives in the mountainous areas (Himalayas) from India to Mongolia. The fibres are obtained from the skins of dead animals and de-haired manually, fibres from the throat areas being regarded[269] as the most desirable. It is thought 15 000 animals have to be killed to produce 1000 kg of de-haired fibre,[267] i.e. each animal produces around 70 g of de-haired fibre. Tonin *et al.*[267] found a value of 13.6 μm (range 13–15 μm[270]) for the mean fibre diameter of the de-haired Yangir fibre sample they tested, the CV of fibre diameter being 22.6% and coarse fibre (> 30 μm) content 0.1%. They also found the scale density to range from 5/6 to 18/20 scales per 100 μm, and the scale height > 0.8 μm.[270]

According to tradition, and certain reports, Shatoosh (Shah-tus) is the hair from the Tibetan antelope (*Pantholops hodgsoni*), called 'Chiru' in Tibetan,[267] present mainly in the Tibetan region of China. Shahtoosh/shahtus, which means 'king of wools' in Persian or Urdu[271] (reference[52] and CNN Italy, www.cnnitalia.it/1999/STILE/11/04/sciarpa.tibetana, No. 15, 1999, quoted in reference[267]), is one of the finest fibres and used to manufacture 'ring shawls' (they can pass through a wedding ring), each weighing ≈ 120–150 g. The animal is mostly killed to harvest its hair. Hair from at least five animals is necessary to produce a shawl. It was first thought that shah-tus was the shed under-wool of the wild ibex collected during the spring moult, such fibre being used[271] and the shawls referred to as *Shahtoosh.*[272] As much as 30% of ibex guard hair is present in Tosh shawls.[272] According to Ryder[273] the finest fibre in India was named Shah-tush and collected from wild animals, which could include goats, and that it had become illegal to sell shawls made from this fibre because antelopes were being killed to obtain the fibre.

Tibetan antelope is classified by CITES in Appendix I, its highest level of protection, banning all international trade in the species.[267] It was estimated that there were fewer than 75 000 of the antelopes at the beginning of the twenty-first century.

Tonini *et al.*[267] obtained a value of 11.5 μm for the mean fibre diameter of the de-haired Shatoosh they tested, the CV of diameter being 20.1% and coarse fibre (> 30 μm) 0.1%.

The fibres contain 5–6 scales per 100 μm,[274] their natural colour being grey-brown. It takes between 3 and 5 Chiru to produce the 300–600 g of raw fibre required to produce a Shahtoosh shawl,[52] each animal producing about 150 g of fibre. Shamina shawls, made from the fibre of domestic cashmere goats, are sold in India to protect the Chiru.[271] In South Africa a fringed

wrap called a 'shu-shu', consisting of blend of Cape summer kid mohair and superfine Cape Merino wool has been produced.[271]

Shah-tush also refers to very fine fibre shed by wild animals, including wild goats[273] (e.g. wild ibex) and the Tibetan antelope, and the name Shahtoosh (Shatoosh) is also employed to describe shawls from the ibex goat.[272] Rollins and Hall[272] found that the fibres from the Tibetan antelope rarely exceeded 10 μm, generally ranging from 7 to 9 μm, while those from the ibex goat were generally 10 μm or coarser, the guard hair from the ibex having a thicker cell wall (i.e. relatively narrower medulla) than that from the Tibetan antelope (i.e. the latter had a relatively wide medulla). The fibre diameters of the guard hair of the Tibetan antelope varied from 50 to 100 μm or even greater, and those of the ibex goat were rarely over 50–60 μm.[272] The finer fibres generally did not have distinct medulla.

For further reading see the references.[267,269,271, and 274–276]

9.13.6 Opossum (possum) fur

Opossum (*Didelphis* genus, also known as possum) fur has received renewed attention in countries such as New Zealand, where it is blended with superfine Merino wool (e.g. 70%) to produce luxury knitwear. It is also used as duvet filling; it taking 15 bush-tailed possums to produce 1 kg of useable fur,[277] the fur being removed from dead animal skins. It was estimated (in 1999) that there were some 70–80 million possums in New Zealand. The fur ranges in colour from almost black to grey, in mean fibre diameter from about 12 to 16 μm and in mean fibre length from about 15 to 25 mm,[277] the latter making it suitable for spinning on the woollen system, although processing on the worsted system also takes place for the long fibres.[277] The fibre need not be scoured, and is blended with wool prior to carding.

Gore and Laing[278] obtained the following results on the fibres (excluding re-growth) from New Zealand possum (*Trichosurus vulpecula*) fur:

Mean fibre diameter (μm): 19.9 (CV = 49%)
Mean fibre length (mm): 26 (CV = 46%)

For re-growth fibres, they found a typical fibre length of 10 mm and diameter of 18.9 μm (CV = 61%).

9.13.7 Pashmina

Pashmina is a Persian word[271] used to describe cashmere in India, Kashmir and Nepal,[279] but is often also used to describe products containing a blend of cashmere and silk (e.g. 70% cashmere/30% silk).[271] The 'Pashmina' goat

(*Capra hircus*) is bred by nomadic herdsman (Tchang Tang shepherds) in the Ladakh region of Northern India, at altitudes of some 4800 m[223].

Pashmina (Pashm, Pashim, Pushima) can be taken as the fine, downy fibre (undercoat) obtained from, or shed by, the Himalayan Pashmina goat and several other species of goats (ordinary and mountain) in Kashmir and other parts of India.[271] It is generally combed out or gathered from the ground or bushes when the warmer season arrives.

The fibre properties, including diameter (12–14 μm)[52] (or 14–16 μm)[271] and length, processing and applications are generally the same as for cashmere.

Pashmina shawls and scarves were originally produced in Kashmir from hand-spun and woven fine cashmere fibre gathered from the ground and bushes where the goats had been feeding.[2]

9.13.8 Karakul

The karakul (*Ovis*) breed of sheep, also known as Persian lamb, originally came from fat-tail sheep in the Bokhara district of Russia, which resembled the karakul sheep in their zoological characteristics, and were found in this region as early as 1600 BC.[280] The word 'Karakkul' originated from the ancient Assyrian word 'Kara-Gjull' which means a flower (black rose)[281] or something else of beauty.[280] For many centuries this breed was found only in Russia, and it was only at the end of the nineteenth century that some of these sheep were exported to Austria, the first karakul show in the world being held in Vienna in 1898.[280] In 1907 the German Governor of South West Africa (now Namibia) imported 10 ewes and 2 rams from Austria and the country became the largest supplier of karakul pelts in the Western world.[280] The karakul pelts were the main purpose of farming with the breed, the wool being considered as only a by-product. The adult animals were normally shorn twice a year producing about 2–3 kg of wool annually,[282] the wool having a clean yield of some 65%. The colour of the natural pigmented karakul fibre varies from white to grey, brown and black, the pigment being fast to water if initially scoured under neutral or acid conditions.[280]

In South Africa, karakul wool was sorted into 54 different types prior to sale. Production was some 7.1 million kg in the 1978/79 season[281] and in 1996 some 278 000 kg of karakul wool was sold in South Africa and Namibia.[282] The karakul wool staples consist of a short fine under-growth and coarse long fibres, the latter having a diameter up to 70 μm, whereas the under-growth can be as fine as 20 μm. The mean fibre diameter of karakul wool ranges from about 30 to 40 μm, with an overall average (typical) value of about 35 μm (CV ≈ 35%).

The length of karakul wool varies from about 20 to 150 mm (typically 50–100 mm), the length being important in determining the type into which it is sorted. The shorter, free from vegetable matter, karakul wool is

processed on the woollen system and the longer types on the worsted system. Karakul processed on the woollen system, often in blends with other wool, were mainly used in carpets, blankets, upholstery and curtaining, but also in protective clothing[280] and mulch fabrics.

Karakul types, 65 mm and longer, lend themselves to combing, with the noils produced being used in the woollen system, needle punching (e.g. protective clothing, insulation, carpeting) or in industrial felts. The good felting properties of karakul have been ascribed to the presence of the fine undercoat fibres with the coarser outer coat fibres. Yarns produced on the worsted system were mainly (some 80%) used in interlinings. The processing of karakul on the woollen, worsted and semi-worsted systems as well as the manufacturing of products have been investigated by various researchers.[283–290]

The natural pigmentation (colours) of karakul limited its application, although the lighter types of karakul wool (e.g. white, steel, silver and fawn) could be dyed to almost any colour, the most suitable dyes being reactives and chrome. Because of the limitations resulting from its natural colours, considerable work was carried out on the cost-effective bleaching of Karakul[291–293] with the minimum amount of damage. A number of processes were developed, mordant bleaching in particular,[292] and ferrous mordanting prior to peroxide bleaching.

China is the largest producer of karakul today. The virtual demise of the karakul pelt industry, largely resulting from pressures by animal lovers and environmentalists, drastically reduced the production of karakul wool in South Africa and Namibia. Current production is only some 250 000 kg clean wool in Namibia.

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Bioengineered natural textile fibres

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Abstract: Cellulose is a biological renewable resource whose biomass can be used in the production of paper, textiles, biofuels and chemical compounds. Cellulose rarely occurs in its pure form and is usually found in the form of lignocellulosic biomass. Recently bacterial cellulose (BC) of microbiological origin, synthesised by microorganisms, has also been developed. The transformation of lignocellulosic biomass is not a simple task and typically involves two stages: first, pre-treatment, and second, hydrolysis of the pre-treated cellulose and hemicellulose to form simple sugars (saccharification). Further understanding of the complex mechanisms that allow termites to decompose cellulose-based material of this type may make it possible to apply similar solutions on an industrial scale. One potential future direction in this area is the development of modified natural fibres and biomaterials such as biosilk and fibres based on polylactic acid (PLA) and polyhydroxybutyric acid (PHB), but this is very much in the early stages at present.

Key words: lignocellulosic biomass, bacterial cellulose, lignocellulose pre-treatment, termite symbiotic system, modified natural fibres.

10.1 Introduction

Cellulose is a commonly occurring renewable biological resource, used to manufacture fibers, paper, polymers, lacquers, photographic films and thermal insulation materials as well as biofuels such as ethanol. Cellulose $(C_6H_{10}O_5)_n$ is a non-branched polysaccharide biopolymer, composed of anywhere between a few to several hundred thousand glucose molecules linked by β-1,4-glycoside bonds; it is the principal constituent of plant cell walls. It is insoluble in water, acids and alkalis. In the presence of oxygen, cellulose is decomposed by many fungal species as well as cellulosic bacteria, resulting in water and carbon dioxide production. In anaerobic conditions, cellulose is broken down to methane by bacteria of the *Clostridium* genus found in the rumen of ruminants, while bacteria of the *Cellulomonas* genus hydrolyse cellulose to shorter chains, including glucose. However, due to the absence of cellulose-decomposing bacteria in their gastrointestinal tracts, mammals are unable to use this compound as a source of energy, and can only use it as a bulk food component.

Once cellulose biomass has been enzymatically hydrolysed by natural or artificial means, it can be utilised for the production of paper, textiles, other plant fibres, fuels and chemical compounds. The hydrolysis of cellulose into glucose is by no means a simple process and requires synergistic action by endonucleases, cellobiohydrolases and, later, β-glycosidases (Zhang and Lynd, 2004, 2006). One method of hydrolysis uses a system of cellulases (cellobiohydrolase I, cellobiohydrolase II and endoglucanase I) derived from *Trichoderma*.

Cellulose, the basic material of all plant substances, rarely occurs in its pure form (e.g. flax, cotton, hemp, kenaf, juta, abaca, etc.) and is usually found in the form of lignocellulosic biomass in which cellulose and hemicellulose are closely bound with lignin (see Chapter 1: *Introduction to natural textile fibres*).

Green algae (*Valonia*) and some bacteria, mostly those of the *Acetobacter*, *Sarcina* and *Agrobacterium* genera, also produce cellulose. *Acetobacter*, and particularly *Acetobacter xylinum*, is the most widely studied of these, as these strains are known to able to oxidise alcohols to acids and ketones, and are commonly used in the production of vinegars using ethanol, wine or cider as carbon sources. When *acetobacter* are used, the cellulose pellicle forms on the upper surface of the supernatant film, and it has been observed that cellulose production increases when lactic acid, methionine, tea infusion and corn steep liquor are added. New non-conventional bioreactors have been developed for this production process; however, static cultures are still preferred. Factors affecting productivity include surface area (the larger the surface area, the better the productivity) and glucose concentration (lower glucose concentrations give better productivity as well as increased yield). Bacterial cellulose can be used in applications where plant cellulose is not suitable, for example, as a thickener to maintain viscosity in food, cosmetics, etc., as a nonwoven fabric or paper for repairing old documents, and as food additives, among other uses. Cellulose films could also be used as a temporary substitute for human skin in the treatment of, for example, burns and ulcers. In surgery, and for dental implants, three products of microbial cellulose that have become commonly used are Biofill®, Bioprocess® and Gengiflex® (Jonas and Farah, 1998).

Lignin, together with cellulose and hemicelluloses, is one of the principal constituents of wood. It is an adhesive substance that preconditions wood cell structure compactness, providing wood with compression strength and maintaining its stiffness. The elimination of lignin from wood (delignification) through the addition of sodium compounds results in a softening of the wood substance. Lignin is a polymer whose monomers are organic compounds derived from phenol aromatic alcohols (coniferyl, synapyl and coumaryl alcohols). The chemical structure of lignin is cross-linked with ether and covalent carbon–carbon (C–C) bonds. Lignin is an important source of

the aromatic part of the humic acids found in soil, peats, lignites and coals. Following incomplete lignin decomposition, the soil is also richer in nitrogen compounds.

Hemicelluloses constitute a diverse group of polysaccharides and their derivatives, bound with β-glycoside bonds, and are one of the principal components of plant cell walls. Compared to cellulose, they display a worse resistance to the action of diluted acids and, also in contrast to cellulose, they dissolve well in diluted alkalis. Hemicellulose macromolecules are composed not only of glucose residues but also of other residues of simple sugars such as xylose, mannose and galactose. Hemicelluloses are characterised by a lower degree of polymerisation (DP < 300) than cellulose, as well as lower structural regularity and degree of structural arrangement, all of which makes them less resistant to degradation.

Currently, the most significant challenge for industries using lignocellulose is the separation of lignin and cellulose fractions; unfortunately, due to bonds between polysaccharides (cellulose and hemicellulose) and lignin, the process is somewhat complex.

10.2 Bacterial cellulose

Thanks to the development of nanobiotechnology, certain nanomaterials can be obtained with the assistance of enzymes or live cells, most frequently microorganisms. One of these nanomaterials of microbiological origin is bacterial cellulose (BC), one of many polysaccharides synthesised by microorganisms. These polymers provide an excellent example of ready-to-use nanobiomaterials whose properties can further be improved by way of genetic modification (at the level of the manufacturer's strain) either by the application of appropriate conditions during the culturing of microorganisms or through chemical and/or enzymatic modification of the polymer obtained using culturing.

Bacterial cellulose, first obtained in 1957 by Colvin, is a nanomaterial because its microfibrils, which are made up of β-1,4-glucan chains arranged collinearly with the degree of polymerisation close to 15 000, are characterised by cross-section of 5–10 nm × 30–35 nm (Bielecki *et al.*, 2002). They form ribbons 70–150 nm wide. This polysaccharide is manufactured most efficiently by strains of *Gluconoacetobacter* (formerly classified as *Acetobacter xylinum*), and differs from plant cellulose in the following ways: smaller cross-section of fibres, high degree of crystallinity (over 60%) and lack of impurities such as hemicelluloses (plant heteropolysaccharides) and lignin (strongly cross-linked, plant aromatic polymer). *Gluconoacetobacter* are gram-negative aerobic bacteria that synthesise a white, skin-like, strongly hydrated and elastic membrane on the surface of liquid media in order to guarantee cells access to oxygen. Each strain of *G. xylinus* and other

BC-manufacturing bacteria synthesise membranes with differing elasticity, water-binding capacity, degree of polymerisation (usually 2000–6000, although in some cases even 16 000 or 20 000) and degree of crystallinity. These properties are affected by culturing conditions (stationary or shaken), culturing duration and medium composition (source of C and N, presence of other polymers, e.g. collagen, hemicelluloses). The ultimate properties of the BC produced are further dependent on the method of purification (removal of bacterial cells and medium residues) and the modifications possible after the culturing process is complete.

The unique properties of BC produced by *Acetobacter* (namely: high mechanical strength, high water absorption capacity, high crystallinity, and an ultra-fine and highly pure fibre network structure) mean that it has great potential to become a new commodity biochemical that can be used for a wide range of applications across different industries. Indeed, it is already used in the following applications: as a food matrix in *nata de coco*, a jelly-like food product produced by fermentation of coconut water; as a source of dietary fibre; in temporary dressings used in burn healing; as an acoustic or filter membrane; as ultra-strength paper; and as a reticulated fine fibre network with coating, binding, thickening and suspending characteristics. However, for more extensive industrial use to be feasible, the mass production process used for BC needs to be improved and made more efficient, most probably through the use of submerged fermentation technology.

A means of wet spinning bacterial cellulose to produce textile fibres has also been developed, and studies are currently underway into their use as a superconducting and optical fibre matrix. Bacterial cellulose production in surface culture (up to 28 g/l) and submerged culture (up to 9 g/l) was improved by Vandamme *et al.* (1998) via strain selection, mutation, medium composition optimisation and the control of physico-chemical fermentation parameters. A significant improvement in cellulose yields was achieved through the use of glucose and fructose as the carbon source and acetic acid as the energy source, combined with careful control of both the pH and the levels of dissolved oxygen. An appropriate fructose:glucose:acetic acid ratio allowed for control of the internal pH of the stationary surface cultures.

It has been shown that the use of insoluble microparticles such as diatomaceous earth, silica, small glass beads and loam particles can improve the cellulose formation process. Vandamme *et al.* (1998) added these microparticles to submerged, agitated/aerated *Acetobacter* cultures, resulting in enhanced cellulose synthesis, possibly as a result of *Acetobacter* cells attaching to the particles as a biofilm, which in turn leads to the formation of microenvironments with locally lowered dissolved oxygen levels. The result of this is that more glucose is available for cellulose formation, since less

is lost in the form of gluconate. The pH profile can then be kept within the desirable range. A UV-mutation and proton enrichment strategy was also developed by the same authors: this allows *A. xylinum* mutants to be selected: these have only low levels of (keto)gluconate synthesis, and are able to produce cellulose more efficiently, even under oxidative culture conditions. As above, however, further improvements in the process are still required for mass production of bacterial cellulose by this means to be feasible (Vandamme *et al*., 1998).

Shibazaki *et al*. (1993) tested a thin membrane of bacterial cellulose (BC) obtained from *Acetobacter* culture to investigate its performance as a dialysis membrane in aqueous systems. The BC membrane proved to have superior properties when compared to a dialysis-grade regenerated cellulose membrane, particularly in terms of its mechanical strength. As a result, a thinner BC membrane can be used, which leads to higher permeation rates for (poly)ethylene glycols as probe solutes. The original BC membrane also had a significantly larger cut-off molecular weight than the regenerated cellulose membrane, and this could further be modified through use of concentrated alkali treatments. For further information on the precise nature of the ultrastructural changes caused by these alkali treatments, see Shibazaki *et al*. (1993), who carried out investigations using X-ray diffraction and scanning electron microscopy.

The nanofibrillar spatial structure of BC makes it an excellent dressing material that can adjust to closely fit all kinds of wounds (Czaja *et al*., 2006). When purified well, i.e. deprived of any cell residues and all kinds of culture medium components, bacterial cellulose meets all the requirements expected from biomaterials, i.e. it is biofunctional, biocompatible and non-allergenic, and is not mutagenous or teratogenic. Another equally important characteristic of BC as a dressing is its strong and permanent hydration as well as its capability to bind wound secretions. Moist BC dressings offer good protection against infections from the immediate surroundings, as well as having a cooling effect and reducing the pain associated with the healing process. In addition, it does not stick to the regenerating tissue; this means that changing the dressing does not harm the tissue and is consequently not painful. BC dressings can be any shape and size, and have been shown to decrease wound healing time and prevent scar formation. The therapeutic effects of the above dressings can further be enhanced by including various drugs and substances accelerating angiogenesis into the BC. Antibacterial agents such as, for example, nanosilver, lysozyme or antibiotics can also be added to the BC. If the surface on which *G. xylinus* develops the compact membrane during the culturing process is correctly formed, pipes of any diameter and length can be obtained, which can act as excellent blood vessel implants or as sheaths of broken nerve fibres to speed up their regeneration. Appropriate processing after culturing makes it possible to obtain material

similar to cartilage from BC; this can be used in implant production in place of cartilage of animal origin Table 10.1 presents the potential medical and non-medical applications of BC (Bielecki and Kalinowska, 2008).

Svensson *et al*. (2005) described tissue constructs for cartilage with native mechanical properties, exploring the potential for BC to be used as a novel scaffold material due to its unusual material properties and degradability. To this end, bovine chondrocytes were used to evaluate both modified and unmodified BC, with the results indicating that unmodified BC, which provides substantial advantages over the collagen type II substrate in terms of mechanical properties, allows approximately half the level of chondrocyte proliferation. Unmodified BC showed significantly higher levels of chondrocyte growth than was observed in tissue culture plastic and calcium alginate. Chondrocyte growth and viability did not appear to be affected by either chemical sulfation and phosphorylation of the BC (performed to mimic the glucosaminoglycans of native cartilage) or by the porosity of the material. As proinflammatory cytokine production was not activated by the use of BC during *in vitro* macrophage screening, human chondrocytes were used in the next experiments. The results laid out in Svensson *et al*. (2005), on the basis of transmission electron microscopic (TEM) analysis and ribonucleic acid (RNA) expression of the collagen II from human chondrocytes, demonstrate that unmodified BC does support the proliferation of human chondrocytes. The same authors also showed that the chondrocytes

Table 10.1 Applications of bacterial cellulose

Field	Application
Medicine	Dressing material (artificial skin) accelerating the healing process of all types of wounds, implants of blood vessel, trachea, etc., dentistry implants
Cosmetics	Emulsion stabilisers (creams, tonic agents, etc.), constituents of artificial nails, etc.
Textile industry	Fabrics, artificial skin, materials of high absorption capacities, materials for tent production and tourist equipment
Paper and pulp industry	Paper of special properties, repair of antique books, more durable banknotes and other paper products
Environmental protection	Water ultra filtration, sewage treatment, absorption of petroleum-based contaminations, toxins, etc.
Production of dietary food	Edible cellulase of '*nata de coco*' type
Acoustics	Loudspeaker membranes
Scientific investigations	Protein immobilisation, chromatography, constituents of culturing media

Source: Bielecki and Kalinowska (2008).

grew into the scaffold, suggesting that BC could be suitable as a scaffold for tissue engineering of cartilage (Svensson *et al.*, 2005).

10.2.1 Biosynthesis of bacterial cellulose

The most important problem encountered during the production of bacterial cellulose on a large scale is inefficient synthesis and the appearance of Cel$^-$ mutants, i.e. *G. xylinus* cells which are incapable of producing this glucan and, simultaneously, make use of nutrients present in the culturing medium (Bielecki *et al.*, 2002). These mutations appear particularly frequently during submerged cultures with shaking and aeration and are considerably rarer during stationary cultures. The most probable cause of this difference is a relatively high oxygen concentration in the entire volume of the aerated medium in the fermenter; bacterial cells therefore do not have to synthesise a cellulose mat ensuring growth on the medium/air interphase which takes place in the course of stationary culturing. However, the precise mechanism of development of Cel$^-$ forms has not yet been established, hence the increasing interest in understanding the regulatory mechanisms of BC biosynthesis at a molecular level. Moreover, investigations into the impact of other culturing parameters on the efficiency of the process are ongoing. A huge advantage of *G. xylinus* strains is their good growth on different sources of carbon and nitrogen; therefore, in order to reduce costs, BC is frequently cultured on waste-containing media such as glycerol, molasses, whey or by-products of vegetable processing and so on (Bae and Shoda, 2005; Krystynowicz *et al.*, 2000; Thompson and Hamilton, 2001).

Aside from the investigations into optimising the composition of the culturing media, further studies were also carried out with the aim of assessing the impact of high hydrostatic pressure on the growth rate of *G. xylinus* and on the quantity of manufactured cellulose produced. It was found that *G. xylinus* is a barotolerant microorganism, capable of adapting even to the pressure of 100 MPa without losing its capacity to biosynthesise BC, although with the increase of pressure the efficiency of the process decreases (Karakoti *et al.*, 2008). In addition, *G. xylinus* strains are capable of synthesising BC growing on the same medium with lactic acid bacteria (Seto *et al.*, 2006). Furthermore, it was observed that strains of *G. xylinus* synthesise BC efficiently on both solid substrates or on gel substrates and, therefore, change the mutual position of pores through which cellulose chains are secreted outside cells (Yamanaka *et al.*, 2000).

The process of cellulose biosynthesis begins with the transformation of glucose into uridine diphosphate glucose (UDP-glucose) which serves as a donor of β-D-glucopyranose residues which are linked to the growing β-glucan chain. At least eight proteins take part in the process, including four

key enzymes such as glucokinase, phosphoglucomutase, glucoso-1-phosphatic uridine transpherase and cellulose synthase. These proteins, together with sequences of genes coding them, are well recognised (Bielecki *et al*., 2002; Kawano *et al*., 2002), although it is still not clear how their expression is regulated despite the fact that cyclic diguanosine monophosphate (c-di-GMP) (Edgar, 2007) is known to act as the stimulator of BC biosynthesis.

c-di-GMP is a compound which regulates many different cell processes and makes their rate conditional on signals coming from the environment. Its concentration is regulated by the activity of the diguanylate cyclase (CDG), which catalyses the conversion of 2 GTP molecules into c-di-GMP, and of two enzymes that respectively transform the latter compound into either linear di-GMP or two molecules of GMP such as phosphodiesterases A and B (PDEA and PDEB). Genes of these three enzymes form a common operon (despite opposing activities) occurring in three different variants whose expression supplies, respectively, 80%, 15% and 5% of these proteins.

In many *G. xylinus* strains the efficiency of the BC biosynthesis increases if ethanol, generally considered to create stress conditions, is added to the medium; it is thus very likely that BC production is stimulated by the sigma B factor (SigB) whose gene undergoes increased expression in different stress conditions (Nies, 2004).

The genetic material of *G. xylinus* strains applied to the biosynthesis of cellulose has not yet been completely sequenced. Sixty-seven nucleotide sequences as well as 138 sequences of proteins of various *G. xylinus* strains have so far been collected in databases. These are primarily sequences of genes and proteins associated with cellulose biosynthesis. The majority of these genes have already been expressed in cells of appropriate hosts. This was the case with genes encoding subunits that make up cellulose synthase and which constitute part of one operon. Other genes have also been expressed in this way. Cellulose synthase is an oligomeric protein (Bielecki *et al*., 2002) occurring in all organisms that synthesise cellulose (with the exception of the gram-positive bacterium *S. ventriculi*) and is made up of three (AB, C and D) or four (A, B, C and D) subunits. Each of the above subunits plays a different role during the course of cellulose synthesis, e.g. subunit A is a glycosyltransferase elongating glucan chain, whereas subunits C and D are responsible for its transport outside the cell. Cellulose synthesis requires the cooperation of the cellulose synthase and enzymatic proteins coded by certain genes placed above and below its operon such as: β-glucosidase, β-1,4-glucanase and CcpAX protein (cellulose complementing protein *Acetobacter xylinum*).

Comparative DNA analysis of individual *G. xylinus* strains revealed a high degree of homology and conservatism of gene sequences of the cellulose biosynthesis process. However, despite this, comparable *G. xylinus* strains did not demonstrate similar levels of cellulose productivity.

The last parameter was successfully increased by the introduction of mutations within genes not connected with the cellulose synthase operon such as glucose dehydrogenase (which converts glucose into gluconic and ketogluconic acids) (Shigematsu and Kida, 2005) and levansucrase (which catalyses levan synthese, i.e. β-D-fructofuranose polymer) genes (Tonouchi *et al*., 1998) because the inactivation of these two enzymes blocks processes competing with cellulose synthase.

The efficiency of BC biosynthesis is also influenced by mobile insertion elements (IS) that make up part of *G. xylinus* plasmid DNA and are capable of building into chromosomal DNA (Valla *et al*., 1983). Their presence is associated with frequent mutations leading to the reduction of BC biosynthesis. *G. xylinus* strains usually contain a set of several plasmids of differing sizes (from 16 to 300 bp). Generally speaking, Cel^+ and Cel^- cells differ with regard to plasmid profiles, although it should be emphasised that bacterial strains synthesising BC and deprived of plasmids were also found.

Further and more intensive research into genetic preconditioning and regulation of the BC biosynthesis process by *G. xylinus* strains is quite likely to not only increase its efficiency but also to modify the properties of this nanomaterial such that it will be able to meet specific requirements.

10.2.2 Modification of bacterial cellulose

Nanocrystalline, arranged bacterial cellulose structure was utilised in the production of nanomaterials including nanoconductors made of titanium dioxide (TiO_2) (Zhang and Qi, 2005) or zinc oxide (ZnO) (Hussein *et al*., 2005) nanocrystals bound with cellulose nanomatrix. In addition, a number of composite materials are obtained on the basis of BC, including collagen (Wiegand *et al*., 2006), chitosan (Ciechańska, 2004), modified starch (Orts *et al*., 2005), phenolic resins (Nakagaito *et al*., 2005), alginate and polyvinylpyrrolidone (Ramana *et al*., 2006), polyphosphates (Barud *et al*., 2007), silica (Maeda *et al*., 2006), plant cellulose (Mormino and Bungay, 2003) and so on. A slightly different type of composite is material containing built-in polypropylene networks, which is particularly useful as a material for internal application.

BC-containing composites are utilised both for medicinal purposes (for instance, composites with sodium polyphosphate or hydroxyapatite of high mechanical resistance were employed for bone regeneration and for the treatment of changes caused by osteoporosis) and as constituents designed to increase the mechanical strength of other materials. It should be emphasised that the production of composite materials from bacterial and plant cellulose is difficult due to their extremely limited solubility and high hydrophilic properties. Investigations have consequently begun into composites

containing cellulose binding modules (CBMs) derived from cellulase molecules. The characteristic structure of these modules, which are divided into 43 families, is well known, allowing their laboratory-scale production (Pinto *et al*., 2006). They can act as links and can be used to bind BC with peptides of biological activity and with proteins.

In addition to composite materials, cellulose esters also play an important role both in medicine and in the production of packaging materials, plastics and adhesives. These are also manufactured from bacterial cellulose (Edgar, 2007) and include carboxymethylcellulose (CMC) (Ruzene *et al*., 2007), cellulose acetate (Kim *et al*., 2002) and esters of higher organic acids (Jandura *et al*., 2000).

A large group of cellulose derivatives was obtained through oxidation with sodium periodate ($NaJO_2$) which causes disruption of the C2–C3 bond in the glucopyranose ring as well as development of reactive aldehyde groups, allowing covalent binding of compounds that contain free amino groups, including proteins, e.g. lysozyme. On the other hand, treatment with tosyl chloride yielding a tosyl derivative substituted at C6 allows the introduction of a number of other functional groups into glucose rings (Heinze *et al*., 2001). Moreover, BC modifications also include controlled enzymatic hydrolysis (Josefsson *et al*., 2008).

10.3 Enzymatic treatment of cellulose

The transformation of lignocellulosic biomass typically consists of pretreatment aiming at disruption of the crystalline structure, followed by hydrolysis of the pretreated cellulose and hemicellulose to simple sugars. In the stage of enzymatic hydrolysis cellulases obtained from Trichoderma reesei fungi are employed most frequently. High doses of cellulases and β-glucosidases are frequently used to increase the hydrolytic rate and efficiency. Termite as a model for the development of an efficient method of cellulose digestion is being considered.

10.3.1 Enhancement of enzymatic saccharification of cellulose by cellulose dissolution pre-treatments

The principal lignocellulose fraction is cellulose, which can be hydrolysed by cellulases into glucose. Generally speaking, we can distinguish three categories of cellulosic enzymes required for total cellulose breakdown into simple sugars, namely: (a) endoglucanases randomly cutting glycoside bonds inside the polymer chain; (b) exoglucanases acting gradually on the reducing and non-reducing ends of the cellulose chain, liberating cellobiose or glucose as the main products; and (c) β-glucosidases decomposing

soluble cellodextrines and cellobiose to glucose. Enzymatic hydrolysis, in other words saccharification, constitutes a critical stage of sugar production because it is dependent on many factors such as the porosity of the lignocellulose biomass, the degree of cellulose crystallinity and the lignin and hemicellulose content. Pre-treatment procedures are important for the removal of lignin and hemicelluloses, reduction of cellulose crystallinity and increase of porosity. Enzymatic saccharification of the cellulosic biomass is an environmentally friendly process in which enzymes manufactured by *Trametes hirsuta* fungi containing endoglucanases, cellobiohydrolases and β-glucosidases can be used (Jeya *et al*., 2009).

The transformation of lignocellulosic biomass is not a simple task and typically consists of two stages, as follows: first, pre-treatment aiming at disruption of the crystalline structure, followed by hydrolysis of the pre-treated cellulose and hemicellulose to simple sugars, where the process of liberation of simple sugars from carbohydrate polymers is known as saccharification. The main obstacle in the large-scale exploitation of biomass is the lack of cheap technologies for the initial treatment of the starting material. The development of a strategy to initially process and remove lignin from the biomass is important, as this would allow separation to be achieved without damage to the cellulose and chemically non-modified lignins which can provide a source of polyaromatic compounds. Recent investigations have been focused on ionic solutions as well as similar solvents for dissolving and pre-treating cellulose. An ionic solution of 1-ethyl-3-methylimidazolium acetate ([Emim][CH_3COO]) can be used for extraction. The observed cellulose regeneration indicates a significant removal of lignins from the recovered polysaccharides (Lee *et al*., 2009; Singh *et al*., 2009).

Cellulose must be completely regenerated, and solvent residues removed, which increases the overall cost of the process and, simultaneously, rules out the possibility of developing one continuous transformation stage for lignocellulosic biomass. The choice of solvent for the biomass pre-treatment which ensures the activity of both cellulases and microorganisms is a key factor in allowing the development of the continuous conversion process. *N*-methyl morpholine oxide (NMMO) is an attractive alternative to volatile and unstable organic solvents, similar to ionic solution, which is able to dissolve cellulose effectively. Furthermore, saccharification *in situ* eliminates the need to regenerate the recovered cellulose (Ramakrishnan *et al*., 2010). The advantages of the application of this solvent are that it does not affect the composition of the initial material and that more than 98% of it can be recovered: this is an important consideration in industry (Shafiei *et al*., 2010).

Another solvent used during the pre-treatment stage is sulphuric acid. Diluted sulphuric acid was most frequently used due to its lower cost, convenience and effectiveness with respect to a wide range of lignocellulosic

biomass (green material, softwood and hardwood). The applied acid effectively hydrolyses hemicelluloses to monosaccharides (arabinose, galactose, mannose, xylose) and soluble oligomers, which improves cellulose conversion. The next stage involves enzymatic hydrolysis; effective enzyme utilisation, through creation of favourable environmental conditions, is extremely important. Cellulases obtained from *Trichoderma reesei* fungi are employed most frequently; *Aspergillus niger* or cellulases from a modified *T. ressei* strain characterised by a higher activity of β-glucosidases provide an additional source of β-glucosidase. High doses of cellulases and β-glucosidases are frequently used to increase the hydrolytic rate and efficiency, which is connected with costs; therefore it is important to establish the optimal enzyme concentration required for the process of hydrolysis (Zheng *et al*., 2009; Zhu *et al*., 2010).

Enzymatic digestion depends on the lignin content of the material subjected to pre-treatment, and on the concentration of sulphuric acid used during this phase. Investigations showed that low lignin content results in high sugar yields as well as a better sugar liberation rate during the course of enzymatic saccharification. That is why it is necessary to characterise the initial biomass, as this may help to explain interrelations between the functional groups present in lignin and their inhibiting impact on cellulases (Guo *et al*., 2009).

The application of alkaline pre-treatment at low temperature resulted in increased efficiency of the enzymatic hydrolysis of lignocelluloses. Sodium hydroxide solution made it possible to remove lignin, hemicelluloses and cellulose from the material by disrupting the bonds between them; a structure modified in this way is associated with greater sensitivity of cellulose to the hydrolytic process (Zhao *et al*., 2008).

10.3.2 The application of a termite symbiotic system for efficient cellulose digestion

Termites are a commonly occurring group of social insects. Thanks to the presence of symbionts in their intestines, they can decompose considerable quantities of cellulose and hemicellulose into glucose and other simple sugars. Termite intestines constitute a bioreactor which may serve as a model for the development of an efficient method of cellulose digestion (Cao *et al*., 2010).

In order to elucidate the process of lignocellulose degradation in lower termites, a cDNA library was established of symbiotic *Protista* (single-cell *Eucaryota*) occurring in insect intestines. The experiments performed revealed the presence of numerous enzymes: cellulases, xylanases, β-glucosidase, arabinosidase, mannosidase and arabinofuranosidase. This

indicates that the lignocellulose degradation process that takes place in termite intestines is very complex. High level production of cellobiohydrolase indicates that this enzyme, alone or in cooperation with other enzymes, plays a key role in the process (Todaka *et al*., 2007).

In termites it is primarily the intestinal microflora that are responsible for cellulose digestion. However, the discovery of cellulosic activity as well as the cellulase gene in termite salivary glands and in the central intestine appears to suggest that termites are also equipped with endogenous cellulases which can play a specific role in the transformation of cellulose into simple oligosaccharides (Zhang *et al*., 2009). Among the very important challenges facing bioengineering is the understanding of the detailed mechanisms which allow termites to decompose material based on cellulose, as this may make it possible to apply similar solutions on an industrial scale.

10.4 Future trends

The excellent properties of BC have led to a search for new applications of this nanobiomaterial and its derivatives. The prospective applications of bacterial cellulose include the production of electronic paper for use in lightweight displays that are both flexible and durable, designed to replace computer monitors. The bounding of metal ions and colour-changing dyes under the influence of electric current around the cellulose nanofibrils allows electrical conduction between transparent electrodes and reversible change of colour pixels. The result will be a unique high-contrast screen, which can replace newspapers, maps, wallpaper, etc.

The most important challenge for any industry using lignocellulose is the separation of lignin and cellulose fractions during the pre-treatment process. The choice of solvent for the biomass pre-treatment, which ensures the activity of both cellulases and microorganisms, is key to allowing a one-phase continuous conversion process to be developed. Currently the enzyme-based process is the dominant method. The costs associated with enzymatic treatment are high, but the method offers potential for the development of new and less expensive techniques. An understanding of the complex process by which termites decompose cellulose-based material in their intestines could allow similar solutions to be developed for use on an industrial scale.

At the 10th International Cotton Conference (Gdynia 2009) several ideas were presented concerning twenty-first-century fibres such as spider and nanocellulosic fibre, basalt fibres, 'hollow fibres', carbon nanotubes and novel biopolymers based on these advanced and unique fibres. Studies are currently being conducted into the development of biosilk, fibres based on polylactic acid (PLA), polyhydroxybutyric acid (PHB) (in the early stages), and fibroin, modified starch, natural nanofibres and nanofillers (Kozłowski

et al., 2009). Developments in technology, materials, information and biological science can provide us with new economically usable fibres (Aneja and O'Brien, 1999).

Plant fibres are generally inexpensive and easily recyclable; there is therefore a tendency to use them as a compound in composite materials. Natural fibres alone could also be considered as natural composites of cellulose fibrils in a lignin and hemicellulose matrix (John and Thomas, 2008). The biocomposites consisting of natural fibres and biodegradable polymers like polylactic acid (PLA) and polyhydroxyalkanoate (PHA), are eco-friendly (green composites), fully degradable and sustainable alternatives to glass fibre composites. Biofibres such as sisal, flax, hemp, jute, banana, wood and various grasses with polymer matrices from renewable resources are considered as biocomposites. Cellulose is one of the best known renewable resources for biodegradable plastics.

10.4.1 Polyhydroxyalkanoate

Polyhydroxyalkanoates (PHAs) are natural polyesters with a wide range of thermoplastic and elastomeric properties. A number of bacteria have been used for the production of PHAs at high level as an intracellular carbon and energy storage compound. Biodegradable polymers based on PHAs offer excellent potential for use in industry, but the very high cost of the bacterial fermentation production method limits their usage. Alternative options are improving the fermentation and processing techniques or developing transgenic plants that would synthesise PHAs. Transgenic plants, including crops, accumulate PHAs directly in their structure. Extracting these PHAs is not straightforward, but requires fewer steps than traditional production by bacterial fermentation. The production of PHAs using engineered plants can currently be carried out on a laboratory scale, but production is being extended to an industrial scale. The synthesis of PHA in plants was first demonstrated in 1992 by the accumulation of PHB in the cytoplasm of cells of *Arabidopsis thaliana*. PHA copolymers with a wide spectrum of physical properties have been synthesised using transgenic plants like *Arabidopsis*, rape, cotton, corn, potato, sugar beet, tobacco and sugarcane, through the development of new metabolic pathways either in the cytoplasm, plastid or peroxisome (de Oliveira *et al.*, 2004; Khanna and Srivastava, 2005; Poirier, 2002; Steinbüchel and Füchtenbusch, 1998). There is currently considerable interest in engineering plant metabolic pathways for the synthesis of PHA copolymers with better features.

The production of PHB polymer in plants changes the physical properties of the fibre. A very small amount of PHB significantly decreases the rate of heat uptake and cooling of the fibre, leading to higher heat capacity and

improved insulating properties. The synthesis of PHAs in plants can also be used as a unique tool in basic studies of plant biochemistry. High level production of a limited number of useful PHAs and the development of extraction procedures are the tasks for future research (Poirier, 1999), but one attractive alternative for the industry is the production of PHA-based polymers to modify plant fibre properties, which could eliminate the need for an extraction process. Plants naturally synthesise carbohydrate-based polymers like cellulose, and through modification biodegradable plastics can be produced.

Biodegradable composites based on natural fibre plants and poly-β-hydroxybutyrate (PHB) were synthesised on flax (Wróbel *et al.*, 2004). Modified flax stems revealed better resistance to tensile loads than plants which had not been modified: this suggests significant commercial potential for the production of biocomposites. Polyhydroxybutyrate, a biodegradable bacterial polymer, is fragile and has limited potential for use in industry. However, composites containing polypropylene and biofibres from transgenic plants with incorporated PHB have better mechanical properties than those based on natural fibres (Szopa *et al.*, 2009). The improvement of the properties of the biocomposites indicates that biofibres were bonded more strongly to the matrix and adhered to the polypropylene matrix significantly better than fibres from non-engineered plants. Typical biomass crops such as switchgrass could also produce PHB after genetic transformation (Somleva *et al.*, 2008).

The efficient production of PHB and its copolymers is reliant on the correct construction of the genetic vectors used for transformation. Gene expression in a specific compartment of plant tissue, for example, in the leaf of transgenic oil palms, is an important issue for the production and commercialisation of biodegradable plastics in transgenic plants (Masani *et al.*, 2009). Generally, for the manufacture of PHB in plants three enzymes are required. The production of a fusion protein comprising all three enzymes for the PHB biosynthesis pathway by *Escherichia coli* provides a powerful tool for the construction of transgenic organisms that produce PHB (Mullaney and Rehm, 2010).

10.4.2 Polylactide

Polylactides (PLAs) produced from renewable resources (starch) are biodegradable and have very low or no toxicity, high mechanical performance and low thermal stability. Biopolymers such as PLA are not acceptable alone for use as an engineering resin because of their rather low resistance to impact and low heat distortion temperature, which has excluded PLA from many commercial applications. However, it is currently a most promising

and highly popular material with excellent development prospects and is considered to be the 'green' choice for eco-friendly material (Nampoothiri *et al*., 2010; Yu *et al*., 2006). The substitution of natural fibres matrix by PLA, the newly created biocomposite, could lead to high mechanical properties being achieved (Bledzki *et al*., 2009; Yu *et al*., 2006). The biocomposites produced from PLA and abaca fibres have enhanced tensile strength in comparison to native PLA. Optimisation of the process parameters is necessary to allow these biocomposites to be used in industry. Moreover, combining the biocomposites with jute fibre mats improved their tensile properties (John and Thomas, 2008). Fibre-reinforced plastics have high strength and stiffness and low weight, making them excellent, biodegradable substitutes for glass fibres.

10.4.3 Biosilk

Spider dragline silks consist of spidroin and, as they are the strongest natural materials, they are also attractive for industry (Sponner *et al*., 2005). Unlike silkworms, spiders cannot be used for the commercial production of spider silk. The usage of genetically modified plants for the production of new biopolymers is an alternative to traditional non-degradable polymers but involves a more complicated production method. Novel compounds in transgenic plants require the direction of biosynthetic pathways to specific intracellular compartments and efficient production (Börnke and Broer, 2010). Fibrous proteins such as silk have very good properties (elasticity, strength and toughness) for industry but commercial use requires production by transgenic plants able to synthesise biosilk (Keller *et al*., 2001).

Agricultural crops could produce biopolymers on a larger scale than microbial biotechnology allows (Omenetto and Kaplan, 2010; van Beilen, 2008; van Beilen and Poirier, 2008). The production of spider silks in microorganisms, cell cultures, animals and plants is an attractive but complex solution, because silks are a broad class of polymers based on proteins. Silk fibre from silkworms, composed of fibroin (fibrous protein core) and sericin (glue-like protein) that serves to bond fibres together, is one of the most studied structural proteins to be used as a biopolymeric material (Mandal and Kundu, 2008a, 2008b). Silk could be produced by transgenic plants in the form of pure silk proteins or a new generation of silk-like proteins, but for this process it is necessary to control the material properties. Synthetic spider silk genes, producing silk-like proteins, have been expressed in transgenic tobacco, potato (Schallau *et al.*, 2004; Scheller, 2002) and *Arabidopsis thaliana*. Improvements in fibrous protein synthesis in plants may require several approaches, such as increasing the pool of essential amino acids (lysine and threonine), simultaneous expression of several fibrous proteins

and accumulation in proper compartment. Silk-based materials have been used in the textile industry and in surgery and have the potential to be used for biomedical and biotechnological applications not only as silk sutures but also in fibre-based tissue products. The discovery of the silk-like substance produced by the feet of zebra tarantulas (which allows them to attach to smooth vertical surfaces) could broaden the application of silk proteins for safety purposes to enhance locomotor ability and avert catastrophic falls (Gorb *et al.*, 2006).

Transgenic plants have the attractive capability to produce polymers and chemicals but commercial exploitation is not yet possible. Competition between genetically modified plants and conventional production in terms of economic efficiency and sustainability depends on a number of factors such as future trends in oil and energy prices, public acceptance, the regulatory framework for their commercialisation and long-term political commitment to providing impetus into research and commercialisation of transgenic plants for biomaterial production (Börnke and Broer, 2010).

10.5 Conclusions

Thanks to the development of nanobiotechnology, certain nanomaterials can be obtained with the assistance of enzymes or live cells, most frequently microorganisms. One of these nanomaterials of microbiological origin is bacterial cellulose (BC), one of many polysaccharides synthesised by microorganisms. These polymers provide an excellent example of ready-to-use nanobiomaterials, whose properties can further be improved by way of genetic modification (at the level of the manufacturer's strain), either by the application of appropriate conditions during the culturing of microorganisms or through chemical and/or enzymatic modification of the polymer obtained using culturing.

A nanocrystalline arranged BC structure was utilised in the production of nanomaterials including nanoconductors made of TiO_2 or ZnO nanocrystals bound with a cellulose nanomatrix. In addition, a number of composite materials can be obtained on the basis of BC including collagen, chitosan, modified starch, phenolic resins, alginate and polyvinylpyrrolidone, polyphosphates, silica, plant cellulose, etc. A slightly different type of composite is material containing built-in polypropylene networks: this composite is particularly useful as a material for internal application.

Cellulose biomass is a commonly occurring biological renewable resource and generally exists in the form of lignocellulosic biomass. The transformation of lignocellulose is not simple and involves pre-treatment to disrupt the structure, as well as hydrolysis of the pre-treated cellulose and hemicellulose to simple sugars. Nowadays, the dominant method is an enzyme-based process. Simple sugars can also be obtained from commonly occurring

non-food plants of considerable biomass. Furthermore, termites, with the help of symbionts in their intestines and their own enzymes, can decompose cellulose and hemicellulose to simple sugars. In this case the termite intestine, as a natural bioreactor, may serve as a model for the development of efficient method of cellulose digestion.

The development of modified natural fibres and biomaterials is a very important issue for future research. Plant fibres are generally inexpensive and easy to recycle; consequently there is a tendency to use them as a compound in composite materials. Moreover, natural fibres alone could be regarded as natural composites of cellulose fibrils in a lignin and hemicellulose matrix.

10.6 References

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10.7 Appendix: abbreviations

BC	bacterial cellulose
CBMs	cellulose binding modules
C–C	covalent carbon-carbon bonds
CcpAX	cellulose complementing protein *Acetobacter xylinum*
CDG	diguanylate cyclase
c-di-GMP	cyclic diguanosine monophosphate
Cel^+	cellulose-positive mutant
Cel^-	cellulose-negative mutant
CMC	carboxymethylcellulose
CSG	diguanylate cyclase
di-GMP	linear diguanosine monophosphate
DP	degree of polymerisation
[Emim] [CH3COO]	1-ethyl-3-methylimidazolium acetate
GMP	guanosine monophosphate
GTP	guanosine-5'-triphosphate
IS	insertion elements
PDEA	phosphodiesterase A
PDEB	phosphodiesterase B
PHA	polyhydroxyalkanoate
PHB	poly-β-hydroxybutyrate, polyhydroxybutyrate
RNA	ribonucleic acid
PLA	polylactide, polylactic acid
SigB	sigma B factor
TiO_2	titanium dioxide
TEM	transmission electron microscopy
UDP-glucose	uridine diphosphate glucose
ZnO	zinc oxide

11

Identification of natural textile fibres

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Abstract: Fibre identification is a very important component to textile industries, fashion and design houses, and forensic science. The identification methods, however common across disparate industries, are conducted very differently in each. The technological changes create a constant need to improve the identification methods. In spite of the increased pace of new technology, the old methods are often the best. This chapter covers the distinguishing features of natural textile fibres; their structure and properties; and various methods devised for their identification. Also the recent developments in fibre identification techniques and the forensic methods of fibre identification have been explained.

Key words: natural fibre, fibre identification, microscopy, spectroscopy, forensic analysis.

11.1 Introduction

The identification of textile fibres is important for textile and apparel manufacturers, fashion designers, automotive industries and forensic science. The identification methods, however common across disparate industries, are conducted very differently in each. Some methods are exclusively used by one group; others are shared, while some shared methods have greater or lesser utility for the analyst. Differences in physical and chemical properties are the basis for fibre identification. Generally, simple tests are sufficient to enable a fibre to be categorised into the main groups of natural fibres where the differences in properties are relatively large. More complicated tests are essential to distinguish between fibres within a group. Many fibres can be identified by fairly simple tests needing only inexpensive equipment.

Although some new instrumental identification techniques originated with the technical developments, the old methods are often considered to be the best. Most laboratories will still rely on the simplest microscopical examination that provides a range of analysis barely possible with any other method. Combined with spectroscopy, microscopy is the quintessential fibre identification tool. Instrumental methods of identification provide reliable means to distinguish between the main types of fibres according to their

chemical structure and closely related fibres with only minor differences in properties.

The American Association of Textile Chemists and Colorists (AATCC) technical manual lists microscopy as a useful method for fibre identification, whereas the American Society for Testing and Materials (ASTM) reports infrared (IR) spectroscopy with additional physical properties as the preferred method for fibre identification. AATCC also lists reaction of fibres to flame (such as melts near flame, shrinks from flame and burns in flame) as a test method for fibre identification. The amount of sample for analysis is nearly unlimited for producers, while it is minimal in the case of forensic analysis. Therefore, different methods are devised for fibre identification. For both, the purpose of the analysis of common parameters is to determine the individual fibre's morphological, optical and chemical properties as distinctly as possible.

11.2 Natural textile fibres

A natural fibre is any fibre that exists as such in the natural state, such as cotton, wool or silk (Houck, 2009). Natural fibres differ from each other in cross-sectional shape, colour, surface contour, chemical structure as well as length and width (Ziabicki, 1976). The natural textile fibres can be classified into three groups – vegetable, animal and mineral – according to their origin. But only the vegetable and animal fibres will be discussed in this chapter.

11.2.1 Vegetable fibres

Vegetable fibres are mainly collected from different parts such as: seed (cotton, akund, kapok, coir); bast (flax, jute, hemp, ramie, kenaf); and leaf (sisal, abaca) of various plants. The vegetable fibres are also called cellulosic fibres as cellulose is the main component of the fibres. The identification of vegetable fibres includes the analysis of: the size and relative thickness of cell wall; shape and thickness of lumen; the presence, type and distribution of dislocations; and the direction of twist of the cellulose in the cell wall (Greaves, 1990).

Seed fibres

Cotton is the most common seed fibre and accounts for approximately half of all textile fibres processed annually. The unique feature of cotton fibre is the presence of lumen and convolutions, which makes identification easier. Convolutions are twists in the fibre with many reversals along the fibre length, which are clearly visible in the polarised light of the microscope. Cotton can

be distinguished from other fibres under the microscope between crossed polars (Perry *et al.*, 1975). It remains substantially bright in all orientations, except that in the orthogonal positions dark bands cross the fibre at frequent intervals indicating the reversal points of the underlying spiral structure.

The appearance of cotton can be modified by specific chemical finishing to achieve the desired result, e.g. mercerisation of cotton causes fibre swelling and untwisting. Therefore, the convolutions in mercerised cotton may not be easily visible (because of fibre swelling). Generally, the cross-section (CS) of cotton is kidney shaped and in the case of mercerised cotton it is circular. Resin treatment and some other chemical finishes can alter the appearance of cotton fibre.

For preliminary identification of cotton, add a drop of cuprammonium hydroxide to the fibres mounted on a glass slide and examine under the microscope: (a) if the fibres dissolve readily and completely, it is scoured cotton or mercerised cotton, (b) if the fibres swell irregularly, forming beads and finally dissolve leaving a residue of particles that float away in the solvent, it is raw cotton and (c) if the fibres remain undissolved, it is chemically modified cotton. An iodine test can also be used to distinguish between mercerised and unmercerised cotton (Marsh, 1941). In this test cotton fibre is treated with a specific iodine solution and if the fibre decolourises immediately, it is unmercerised cotton. Mercerised cotton takes several hours for discoloration.

Other seed hairs such as akund, kapok and coir are readily distinguished from cotton by a thin cell wall and absence of convolutions. Akund is fine, soft and lustrous, but very weak like kapok. The appearance of akund under the microscope is similar to kapok but the base does not show the net-like thickenings seen in kapok. Kapok fibres are smooth, hollow, cylindrical, thin-walled, brittle and frequently bent over on it. Coir fibres are very stiff, dense and have a distinct cross-section that makes it easier to identify by microscopy. Coir appears dark brown or opaque with very large, coarse ultimates under the microscope.

Bast fibres

The colour of the bast fibres depends upon the retting conditions, e.g. the colour of water retted fibre is from white to yellow and the colour of dew retted fibre is from grey to dark silver. Flax has clockwise twist, polygonal cross-section of ultimates with thick walls and small lumina. The feature of flax fibre that is common to other bast fibres is the presence of dark dislocations, often in the form of an 'X' under the microscope that are roughly perpendicular to the longitudinal axis of the fibre. On the other hand, jute has counter-clockwise twist; microscopically it appears bundled and may have a yellowish cast. It has polygonal cross-section of the ultimates which are angular with medium sized lumina.

The characteristic features of hemp differ from jute with more bundled ultimates, wider lumen and fewer nodes. Also the rounder and more flattened cross-section of hemp helps to distinguish it from jute. Hemp has counter-clockwise twist like jute and may also have a brownish cast to it. Ramie has a thick wall, flattened cross-section and very wide ultimates with the width ranging from 25 to 75 μm. Microscopically ramie has frequent, short dislocations, longer transverse striations and radical cracks may be present in cross-section. Kenaf, with an ultimate fibre length from 2 to 6 mm is more lustrous, harder and stronger than jute, and lighter in colour.

Leaf fibres

The irregular lumen size, acicular crystals, spiral elements and annular vessels are the features for identification of sisal. The cross-section of sisal looks like cut celery and it has counter-clockwise twist. The features for identification of abaca are uniform diameter of ultimates, waxy appearance, polygonal cross-section and counter-clockwise twist. Abaca may contain spiral elements but often will have stegmata, which are visible as small crown-like structures. Sisal is distinguished from abaca by Billinghame's test (Perry *et al*., 1975).

11.2.2 Animal fibres

The animal fibres with protein as the main component include wool, hair and silk fibres. Although the chemical structures of hair fibres are like that of wool, the physical characteristics are different, i.e. they differ in length, fineness, shapes and internal structure, which can be used for their identification.

Wool

Wool is obtained from the hair of sheep and certain other animals, including cashmere from goats; mohair from goats, alpaca and animals in the camel family; and angora from rabbits. Wool can be identified by careful microscopical examination of features such as cross-sectional outline, pattern formed by the scale margins, regularity of fibre diameter, type of medulla and thickness of cuticle. The morphology of these fibres is complex, consisting of a cortex covered by a cuticle of scales and in some instances a central medulla, often showing different patterns in different fibres. Finer wool fibres (e.g. merino wool) have no detectable medulla. The finest merino

wool appears strikingly irregular in diameter when seen under the microscope. The medulla in long wools and cross-bred wools is simple, unbroken and medium to narrow in type.

At the beginning, it should be confirmed that the animal hair fibre to be identified is not human in origin. This is done from the banding characteristics (abrupt and profound colour changes along the hair shaft) of the animal hair. The complete identification of animal hair fibres mainly relies on the morphological features of the cortex, the size and shape of the scales and their pattern around the hair. The scale patterns may be visualised by mounting the hairs in a semi-permanent mounting medium with a refractive index lower than that of keratin (1.55) or it can be done by a scanning electron microscope (SEM). The advantage of SEM is its very high resolution and relatively large depth of field which enables a better discrimination of the surface characteristics. However, in SEM no internal features are visible, and due to the thin coating, further examination of the fibre is not possible.

If the origin of the animal fibre is unknown, determining the diameter of the fibre assists in the identification (Houck, 2009). The medulla types and scale patterns vary in hairs of different species, which can be used for differentiation. Heat treatment and some chemical finish such as alkaline treatment, bleaching or chlorination can modify the structural characteristics of wool. Chlorination causes the scale margins to gradually become less prominent and may be completely removed with severe treatment.

Hamlyn *et al*. (1992) applied molecular biology techniques for the identification of animal hair fibres (wool, cashmere and mohair). In this technique specific DNA probes were developed which can distinguish between DNA isolated from closely related species. This technique is helpful for forensic analysis and biomolecular palaeontology. The chemical resistance of similar type of fibres such as wool, cashmere and mohair to solvents is very similar. Therefore, the solubility test cannot be used to distinguish among these fibres. In these cases scale height (Kusch and Arns, 1982; Robson, 2000), scale pattern (She *et al*., 2001), scale frequency (Robson, 2000) and evenness of diameter (Langley and Kennedy, 1981) may be used to distinguish between wool, cashmere and mohair fibres. She *et al*. (2001) used image processing and artificial neural network model to classify mohair and merino fibres on the basis of the scale pattern.

Silk

Silk is a natural protein fibre mainly produced by the larvae of insects that complete metamorphosis. The best type of silk is obtained from the cocoons of the mulberry silkworm (*Bombyx mori*). Unlike wool fibres, silk fibres are generally easy to identify. The filaments of *Bombyx mori* in gummed state

are stuck together in pairs and covered with sericin. These silk filaments when degummed have a smooth and uniform surface, semi-transparent appearance and approximate to equilateral triangles with rounded apices in cross-section.

Mulberry silk shows a triangular cross-section and a smooth surface, which markedly differ from the non-mulberry (wild) silk (Sen and Babu, 2004). Wild silk appears flat and ribbon-like with fine longitudinal lines. The cross-section of other silks can vary in shape, e.g. crescent-like for anaphe and elongated wedge-shaped for tussah.

Spider silk is a protein fibre spun by spiders with mechanical properties superior to silkworm silk (Cunniff *et al.*, 1994). It has a unique combination of strength, elasticity and resistance to compression, which is judged to be superior to that of synthetic fibres made of polyamide or polyester. Some specimens exhibit over 200% elongation and others possess tensile strengths equivalent to high performance fibres. Also compared to silkworm silk, it is more waterproof. In spite of many superior properties, spider silk has not been domesticated for textile applications because of difficulties associated in raising dense populations of spiders due to their solitary and predatory nature; reeling of a single fibre from the spider webs is difficult; and the amount of silk generated by spiders is small in comparison to the silkworm cocoon silk (Mukhopadhyay and Sakthivel, 2005).

Artificial spider silk has already been spun successfully. The race is now on for the perfection of spinning technology. The most promising methods until now include: conventional solvent spinning of recombinant spider silk protein analogue (Du Pont Ltd) (O'Brien *et al.*, 1998) and extruding regenerated silk into a coagulation bath using a nanofabricated silicon spinneret (Seidel *et al.*, 1998).

Care must be taken while identifying the silk fibre because of its similarities with nylon fibre in fibre diameter and infrared spectra. However, silk is less regular in appearance along its length than a nylon fibre. If difficulties are faced in identifying these fibres, place a short segment or cross-section of the fibre in question in a hot stage. Nylon will melt and silk will not. Fluorescence microscopy may provide additional features based on the fluorescence of the dyes.

11.3 Identification methods

The identification of textile fibres is an important and challenging task. As there are many ways of identifying textile fibres, there is no strict routine test method followed by the textile analyst. Fibres are identified by one or more test methods in combination such as physical appearance, burning test, microscopic analysis, solubility test, staining test and physical property analysis. Although conventional approaches such as microscopy, burning

test, staining test and solubility test are easy to conduct, they have several limitations such as:

- these methods are subjective and rely heavily on expertise, and
- these methods are destructive as there is fibre damage during the test.

11.3.1 Identification by physical appearance

It may be said with some justification that the simplest and most obvious way to identify a fibre is to look at it. As the physical appearances of fibres differ from each other, it can give a preliminary indication for fibre identification. Successful identification of fibres depends on experience and familiarity with the fibres. This method is subjective, however, and often not sufficient for complete identification.

11.3.2 Burning test

The burning test is the first step in fibre identification as mentioned in Bureau of Indian Standards (BIS) (BIS, 1981). This test relies on the behaviour of the fibre when exposed to a flame depending on the chemical composition of the fibre. In this method a small tuft of fibre is held by forceps for about 10 in the flame of a burner and then removed (BIS, 1981). The behaviour of the fibre to the flame (whether it burns, shrinks, forms any bead, the type of smell emitted, nature of remnant ash) helps in identifying the generic type of the fibre. This method is still used in industry where the composition of an unlabelled bale of fibre needs to be identified. This method is very quick and can be performed almost anywhere. However, the test is subjective, destructive, consumes a considerable amount of material and may produce toxic fumes. Table 11.1 describes the burning behaviour of different natural textile fibres.

11.3.3 Microscopic analysis

Microscopic analysis is indispensable for positive identification of several types of cellulosic and animal fibres and also for distinguishing these fibres from man-made fibres (ASTM, 2000). The distinguishing features of each fibre though not sufficient for confirmatory identification, help to categorise them into the correct group. The features include convolutions, lumen and reversal zones (cotton); fibre bundles with or without cross-markings (bast and leaf fibres); presence or absence of scales and scale margins (animal fibres); and smooth profile without scales or convolutions (silk). Other features include longitudinal shape, regularity of diameter and any

Table 11.1 The burning behaviour of natural textile fibres

Behaviour of fibre	Cellulosic fibres	Wool	Silk
When approaching flame	Does not fuse or shrink away from flame	Fuses and curls away from flame	Fuses and curls away from flame
When in flame	Burns quickly without melting	Burns slowly with some melting	Burns slowly with some melting
After removal of flame	Continues to burn without melting after glow	Burns very slowly, usually self-extinguishing	Burns very slowly, sometimes self-extinguishing
Melts near flame	No	Yes	Yes
Shrinks from flame	No	Yes	Yes
Burns in flame	Yes	Yes	Yes
Continues to burn	Yes	Slowly	Slowly
Smell	Burnt paper	Burnt hair	Burnt hair
Appearance of ash	Small, light grey	Lumpy, blistered, irregular, black ash, brittle, breaks easily	Soft, round, black bead, brittle, pulverises easily

Adapted from Lyle, D. S., *Performance of Textiles*, John Wiley and Sons, New York, USA, 1977.

distinguishing characteristics. But sometimes, the morphological differences among some species are not sufficiently distinctive for precise identification since nature tends to use the same repertoire of cells in constructing the fibres of different plants (Goodway, 1987).

Microscopy has and will remain an important aspect of fibre examination (Palenik, 1999) primarily for both vegetable and animal fibres (Brunner, 1974; Catling and Grayson, 1982; Cook, 1969; Palenik, 1983). This method applies various microscopies such as light microscopy, stereomicroscopy, scanning electron microscopy and infrared microspectroscopy. Light microscopy usually provides information about the shape and interior structure of fibres, not about the external features. A stereomicroscope can record fibre characteristics such as size, crimp, colour and lustre.

In contrast to the optical microscope the external features of fibres can be observed by SEM with very high resolution and relatively large depth of field. In SEM images, the complete surface of a fibre can be seen in high detail, which gives a better discrimination of the surface characteristics. In this process, the fibres are made conductive by coating with a layer of conducting metal to allow the charge acquired from the incident electron beam to run to earth. The ability to portray surface topographies with such clarity has targeted the main application of SEM in fibre identification towards natural rather than synthetic fibres. IR microspectroscopy is widely used in forensic analysis.

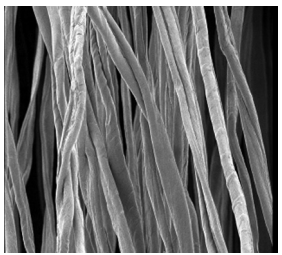
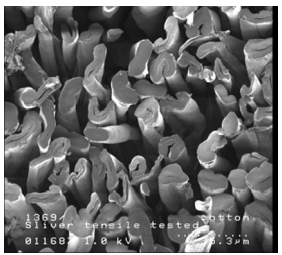
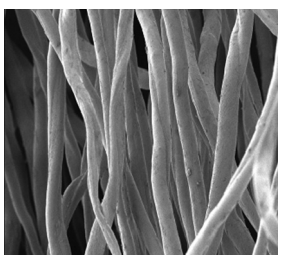
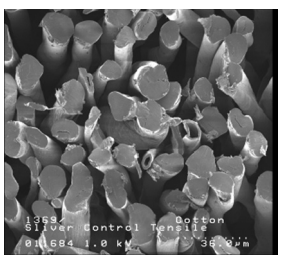
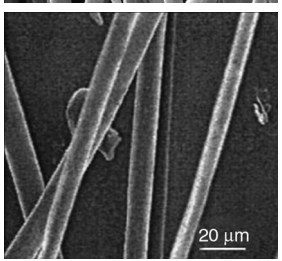
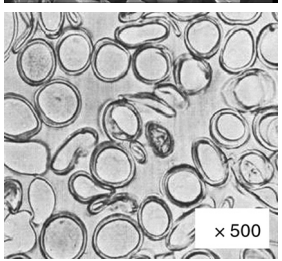
A mounting medium (such as liquid paraffin) with a refractive index about a tenth higher than that of natural fibres, can be used as a convenient method for enhancing contrast at the surface of the fibre so that surface details become distinctly visible in light microscopy (Perry *et al.*, 1975). Microscopic analysis includes longitudinal and cross-sectional examination. In longitudinal examination a small amount of fibre is placed on a glass slide with a suitable mounting medium and covered with a covering glass. The fibres are examined at a specific magnification that provides a strong indication of the type of fibre present. If the longitudinal analysis fails for identification of a fibre, it is necessary to use the cross-sectional analysis. The cross-section (CS) of different types of natural fibres are different and they vary in diameter, i.e. from 10–13 μm (cultivated silk) up to 40 μm (US wool).

In cross-sectional analysis, the CS of a fibre is prepared with a cross-sectioning device, placed on a glass slide with a suitable mounting medium, covered with a covering glass and examined at a specific magnification. The CS can be prepared by microtome, hand-sectioning (by the use of a cork, a C-clamp and a razor blade) and the plate technique (Wildman, 1947). For better quality, the hardy microtome is used for cutting the CS. In some cases despite cutting the fibre CS, the CS can be inferred by slow focusing upon the fibre at fairly high powers. This method is called optical sectioning, which is a quicker and much less tedious procedure than cutting the CS. The longitudinal and cross-sectional images along with the features of different natural textile fibres are shown in Table 11.2.

By using a combination of light microscopy and SEM examinations, Langley and Kennedy (1981) suggested that the earlier criteria for identifying wool, mohair and cashmere were unreliable. The combination may be considered to be the correct and most reliable means of identification by microscopy, which reveals different characteristics of the fibres involved. However, Kadikis (1987) argued that light microscopy reveals more information about the structure and internal characteristics of animal fibres than SEM, and hence is the optimum instrument for such analysis. He also stated that the analyst working in this field should possess years of experience, a good visual memory, patience and the intellectual honesty to recognise when a fibre is difficult to identify.

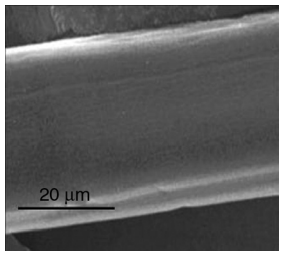
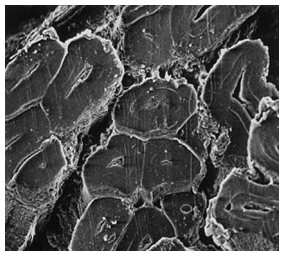
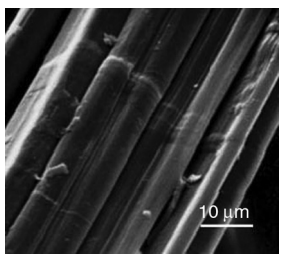
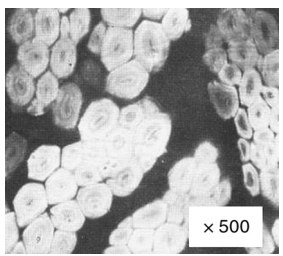
Both light microscopy and SEM should be used in conjunction rather than in competition, as each will have its advantages for particular circumstances. Extensions of the SEM technique include energy dispersive X-ray (EDX), scanning tunnelling microscopy (STM) (Spurny and Stöber, 1981) and plasma etching (Alfy and Blakey, 1980). Microscopic analysis is the quickest, most accurate and least destructive method of determining the types of fibres. Forensic analysers first use microscopy in preliminary identification and then other methods for confirmatory identification.

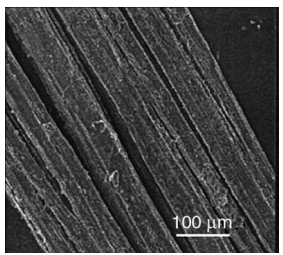
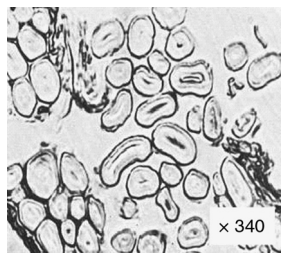
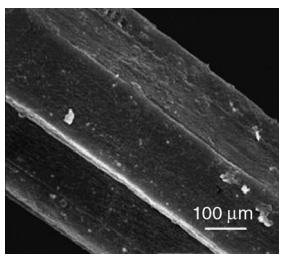
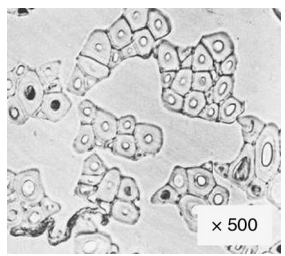
Table 11.2 Microscopic views and features of natural fibres

Longitudinal view	Cross-sectional view	Longitudinal features	Cross-sectional features	Fibre name
		Ribbon like, flattened structure with frequent convolutions, sometimes changing directions; distinct but small lumen, containing protoplasm in raw fibre. Thin walls and few convolutions in immature fibres.	Flat, elongated or kidney bean shaped, seldom round or oval; lumen as a line or oval. Some very thin-walled sections of immature or dead fibres.	*Cotton* (raw and bleached)
		Ribbon-like fibres and fibre regions or less frequent depending on degree of mercerisation; with greater part cylindrical and smooth; very small lumen or disappeared.	Most fibres round or oval (number depending on degree of mercerisation); very small lumen as a small central spot or no lumen.	*Cotton* (mercerised)
		Smooth and cylindrical with rounded base; no convolutions or other structure except at the ends. When mounted in water, shows large, elongated air-bubbles.	Circular or slightly elliptical; thin wall and large lumen.	*Kapok and akund*

(*Continued*)

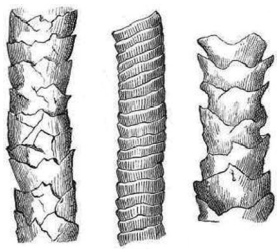
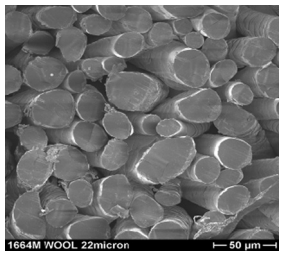
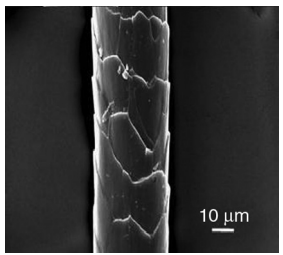
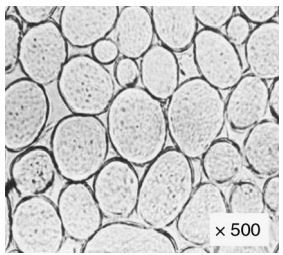
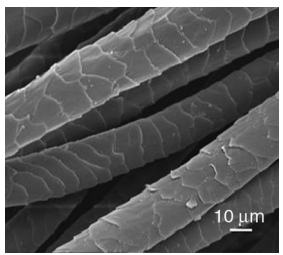
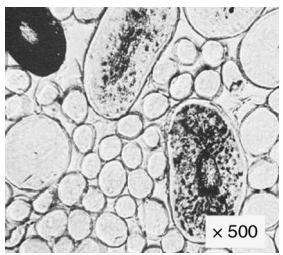
Table 11.2 Continued

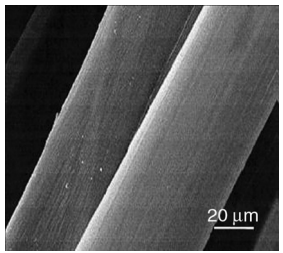
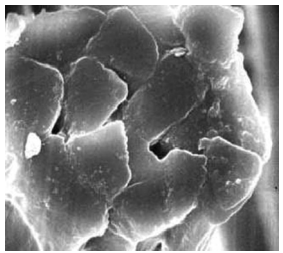
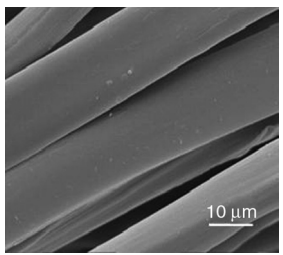
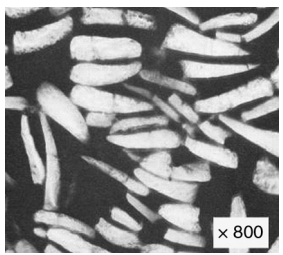
Longitudinal view	Cross-sectional view	Longitudinal features	Cross-sectional features	Fibre name
		Fibre bundles with cross-markings, longitudinal and transverse fissures.	Bundles (and possibly some individual fibres).	*Ramie* (raw, before degumming)
		Isolated individual fibres, very broad and ribbon-like with infrequent twists; cross-markings, longitudinal and transverse fissures.	Long and narrow lumen or same shape as fibre section. Elongated polygons, often with curved side-lines, and sometimes rounded; thick wall and radial fissures.	*Ramie* (degummed, and possibly bleached)
		Fibre bundles, cross-markings, nodes, fissures, but otherwise smooth.	Shape and size of fibre bundles partly depends on preparation; ultimate fibres mainly sharply polygonal with narrow, round or oval lumen; also rounded oblong forms with larger lumen.	*Flax* (raw)

		More or less isolated ultimate fibres depending on degree of bleaching; cross-markings, nodes, fissures but otherwise smooth.	*Flax* (bleached)
		Fibres cylindrical, smooth, few cross-markings and nodes visible.	*Flax* (mercerised)
		Mixture of bundles and single fibres.	*Flax* (cottonised)
		Same as mercerised.	*Flax* (crease-resistant)
 		Similar to flax.	Similar to flax; often the lumen is as a mere line and indistinct.	*Hemp* (raw, bleached and cottonised)
 		Fibre bundles, very rarely cross-markings; nodes or fissures; ultimate fibres (bleached or macerated) with lumen considerably varying in size along the same fibre.	Fibre bundles of varying size; ultimate fibres mainly sharply polygonal, some with rounded corners; lumen round to oval with varying size.	*Jute*

(*Continued*)

Table 11.2 Continued

Longitudinal view	Cross-sectional view	Longitudinal features	Cross-sectional features	Fibre name
		Irregular diameter and prominent scale margins. Medulla present in some medium and coarse fibres; may be fragmental, interrupted, or continuous.	Oval to circular, variable in diameter. Medulla (if present) is concentric and variable in size.	*Wool*
		Regular diameter and smooth profile; scales very shallow; small vacuoles appearing black in some fibres.	Circular to oval, medulla occasionally fragmental or continuous in the coarser fibres.	*Mohair*
		Fairly regular diameter with prominent scale margins. Medulla interrupted or continuous in coarse fibres.	Fine fibres: almost circular; coarse fibres: oval to circular, some flattened, often medullated.	*Cashmere*

		Fine fibres or filaments, cemented in pairs by silk gum. The gum layer is not always continuous.	Triangular, with rounded corners, in pairs.	*Bombyx silk* (raw)
		Fine fibres or filaments variable in diameter, single, smooth, nearly structure-less, sometimes flattened. Occasionally very fine fibres are seen, formed by the superficial splitting of the original fibre.	Separated fibres, triangular, rounded corners.	*Bombyx silk* (degummed)
		Flat irregular ribbons, usually separate, sometimes twisted and with longitudinal striations.	Very elongated triangles, usually separate.	*Tussah silk*

Adapted from Luniak, B., *The Identification of Textile Fibres*, Sir Isaac Pitman and Sons, London, UK, 1951; and *Identification of Textile Materials*, 7th Edition, The Textile Institute, Manchester, UK, 1975.

11.3.4 Solubility test

Solubility tests were widely used for fibre identification before the development of modern instrumental methods. The generic fibre types are identified by the solubility of the fibres in various reagents and comparing the data to the known solubility of several fibres. The reactivity of textile fibres to various solvents is different depending upon the chemical composition of the fibres. A suitable solvent can be selected for each fibre which will dissolve only that fibre. Therefore, fibres which are soluble can be distinguished from those that are not. The list of commonly used solvents for different natural fibres is given in Table 11.3. Solubility tests are easy to perform and can be carried out in test tubes on macro-scale or on the stage of a microscope (ideally used for just such testing).

In the test tube method, a small tuft of fibre is placed in a test tube containing the suitable solvent of the fibre. The fibres are identified by the solubility of the fibre. In the microscopic method, a small amount of the fibre is placed under a cover slip with a drop of the appropriate solvent and the changes are observed under the microscope. For each solubility test, a fresh sample must be used in order to avoid any risk of cumulative damage effects. Great care must be taken to ensure that corrosive or harsh organic solvents do not contaminate any of the optical and mechanical parts of the microscope.

11.3.5 Staining test

In the staining test method a small tuft of fibres is dyed with a mixture of dyestuffs for 1–5 min and washed thoroughly. The coloured fibres are compared with the known dyed samples or viewed carefully to identify the fibre. The chemical composition and reactivity of natural fibres differ from each other leading them to have particular affinity for specific stain or dye types.

Table 11.3 Solubility of natural fibres in different reagents

	Sulphuric acid	Sulphuric acid	Sodium hypochlorite	Sodium hydroxide	Cuprammonium
Concentration (%)	60	75	0.25	10	–
Temperature (°C)	20	20	20	Boil	20
Time (min)	20	30	20	20	5
Cellulosic fibres	I	S	I	Sw	S
Wool	I	I	S	S	Sw
Silk	S	S	S	S	S

Note: S, soluble; I, insoluble; Sw, swell.
Adapted from Perry, D. R., Appleyard, H. M., Cartridge, G., Cobb, P. G. W., Coop, G. E., Lomas, B., Ritchie, G. G., Taylor, C., Welch, M. J. and Farnfield, C. A. *Identification of Textile Materials*, 7th Edition, The Textile Institute, Manchester, UK, 1975 and Garner, W., *Textile Laboratory Manual, Vol. 5*, 3rd Edition, American Elsevier Publishing Company Inc.

Therefore, mixtures of dyes have been developed for fibre identification, which produce known colours for individual fibre types. The best known stains for natural fibre identification are undoubtedly the Shirlastains (A and D) from Shirley Developments Ltd (UK) (Ford and Warwicker, 1961) and Kayastain from Nippon Kayaku (Japan).

The satisfactory identification of natural fibres by a single identification stain 'Shirlastain A' is still widely used for identification of these fibres since it was developed in 1932. The identification between cotton and viscose obtained by Shirlastain A is not entirely satisfactory, however, and an improved distinction may now be obtained by a new product, Shirlastain D.

In this method a small fibre tuft is stained in Shirlastain A at room temperature for 1 min, rinsed under cold tap water and compared with the colour chart for identification. The similar orange hues of chlorinated wool and degummed silk create a problem in distinguishing them. This can be avoided by staining a fresh sample in Shirlastain A at the boil for 1 min. Chlorinated wool appears black while degummed silk is dark, reddish brown. Although the same stain can be used two or three times, the recommendation is to use a fresh portion of the stain for each test.

The main limitation of the staining test is that the fibre samples for staining should be undyed (pale-dyed samples are sometimes usable), wettable and free from any surface finish. Dyed fibres need to be stripped with strong reducing or oxidising agents. Also any chemical damage to the fibre may also affect the colour of the fibre produced. Nevertheless staining tests can be very useful particularly if combined with light microscopical examination.

11.3.6 Identification from physical properties

Various physical properties of unknown fibres such as density, moisture regain, refractive index and birefringence are compared with the reference standards from which the fibres are identified. The physical properties of natural fibres are described in Table 11.4.

Gravimetric method (density)

Different fibres have varying densities according to the polymer from which they are made and this can be used as a diagnostic test. Generally, the densities of natural textile fibres range from 1.3 (wool) to 1.6 g/cm^3 (weighted silk), which may be used to confirm the identity of a fibre. Three methods – density gradient column, pycnometer and a technique based on Archimedes' principle – are described in the ASTM standard (ASTM, 2000).

The simplest approach for determining fibre density is by using a density gradient column. In a glass tube, a liquid of high density (pentachloroethane – 1.71 g/cm^3) and a liquid of low density (xylene – 0.87 g/cm^3) are used to form a linear density gradient region with the density increasing from the top to the

Table 11.4 Physical properties of natural fibres

Fibre	Density (g/cm³)	Standard moisture regain (%)	Refractive index $n_{\parallel}$	Refractive index $n_{\perp}$	Birefringence Δn	Tenacity (g/den)	Extension at break (%)
Cotton	1.52–1.56	8.5	1.577	1.529	0.048	1.7–6.3	3–12
Flax	1.48–1.50	12.0	1.58–1.60	1.52–1.53	0.060	2.6–8.0	1.5–5
Hemp	1.48–1.49	12.0				3.0–7.0	1.5–5
Jute	1.44–1.50	17.0				2.0–6.3	1–2
Sisal	1.33	14.0					
Ramie	1.51–1.55	8.5	1.599	1.529	0.070	4.5–8.8	1.5–5
Wool	1.31	18.25	1.557	1.547	0.010		
Silk	1.32–1.60	11.0	1.591	1.538	0.053		

Remark: Physico-mechanical properties of natural fibres, especially lignocellulosic fibres, depend on cultivation conditions and mainly on primary processing (especially on fibre extracting) methods.

Adapted from Houck, M. M., *Identification of Textile Fibres*, Woodhead Publishing Limited and CRC Press, 2009.

bottom. The other commonly used liquids include ethanol/water mixture and varying strengths of salt solutions. A fibre of known density is used as a calibration marker. Fibre of unknown density is allowed to sink in the tube until it reaches the region of equivalent density, i.e. where the density of the fibre matches that of the liquid. The density value of the fibre may be read off and compared with the standard values for identification (Table 11.4).

Thermogravimetric methods, titrimetric methods, kinetic measurements (rate of fall) and centrifuging techniques are variants of this method. The use of a single instrument to simultaneously identify cotton, wool, silk, viscose and synthetic fibres by thermogravimetry and differential scanning calorimetry has been reported (Crighton and Holmes, 1998; Perkins *et al.*, 1966). In the thermogravimetric method, the sample mass is monitored in a thermal balance as the temperature is raised linearly and the mass-temperature curves are plotted. The fibres are identified from the degree of mass change with temperature. In the titrimetric method the volume of liquid adjusting the density is metered through a burette and the 'end point' of non-movement of the sample is noted.

The gravimetric method requires a very small sample and the colour of the fibre does not interfere with the test. Also, this method is applicable to all fibre types, i.e. thermoplastic and non-thermoplastic. However, the possible interactions (mainly sorption or swelling) between the liquid and the fibre, as well as the trapped air bubbles must be avoided as these lead to a change in the density. This method cannot be used solely for confirmatory test as there is considerable overlapping of densities between different fibre types and difficulty in determining exact density values. Also, in all the density-based measurements, the temperature should be controlled accurately.

Moisture regain

Different textile fibres can absorb and hold different amounts of moisture from the atmosphere depending on the chemical structure. The standard measure of the moisture termed as regain of natural fibres ranges from 8.5% (cotton) to 18.25% (wool). The standard regain (Table 11.4) of an unknown fibre can be determined, which can help in identifying the fibre.

Refractive index

The refractive index of a fibre is directly related to its specific chemical composition. As the fibres are optically anisotropic, the refractive indices along and across the fibre axis will be different. Hence, the fibres are said to be birefringent. The refractive index is an extension of the microscopical method and can be used directly or in combination with melting and solubility behaviour for complete fibre identification. The birefringence (Δn) is the difference between the two refractive indices:

$$\Delta n = n_{\parallel} - n_{\perp}$$

where $n_{\parallel}$ and $n_{\perp}$ are the refractive index parallel and perpendicular to the fibre axis, respectively.

Refractive index and birefringence are the two main distinguishing features for the identification of a fibre's generic class. Although the refractive indices can be obtained by a light microscope with a polariser, the most accurate method is to use monochromatic light and an interference microscope. The refractive indices ($n_{\parallel}$ and $n_{\perp}$) of undyed fibres can be determined by simply mounting the fibres in different liquids of known refractive index until the two are found, which causes the fibre to become invisible for polarised light along and across the fibre axis. The refractive index and birefringence values of different natural fibres are shown in Table 11.4.

The fibres dyed with dark shade require dye removal or stripping before their refractive index can be determined. The refractive index of some fibres (such as cotton and nylon) and the varieties of one group of fibre (wool and mohair) lie very close to one another which necessitates the measurement of some other properties such as melting point and solubility for complete fibre identification. More training and experience are required in this method compared to other methods such as staining and solubility tests.

Other physical properties

Other properties not generally recognised as relevant in fibre identification (such as tensile strength, modulus and melting point) can also be used for identification of the fibres. Subramaniam *et al*. (1983) proposed a method to distinguish between wool and mohair fibres by the examination of their

Table 11.5 Length and diameter of natural fibres

Fibre	Length of commercial fibre (mm)	Length of ultimate fibre (mm)	Diameter (μm)
Cotton	15–56		14–21
Jute	750–1500	0.8–6	5–25
Flax	700–900	13–60	12–30
Ramie	800	40–250	16–125
Kenaf	750–1500	2–6	12–36
Hemp	2500	5–55	16–50
Sisal	600–1000	0.8–8	100–400
Wool	25–300		18–55
Silk	Very long		8–15

Remark: Length and diameter of natural fibres, especially lignocellulosic fibres, depend on cultivation conditions and mainly on primary processing (especially on fibre extracting) methods.
Adapted from Perry, D. R., Appleyard, H. M., Cartridge, G., Cobb, P. G. W., Coop, G. E., Lomas, B., Ritchie, G. G., Taylor, C., Welch, M. J. and Farnfield, C. A. *Identification of Textile Materials*, 7th Edition, The Textile Institute, Manchester, UK, 1975.

Table 11.6 Chemical composition (%) of plant fibres

Fibre	Cellulose	Hemi-cellulose	Pectin	Lignin	Water soluble	Fat and wax	Moisture
Cotton	82.70	5.70			1.00	0.60	10.00
Jute	64.40	12.00	0.20	11.90	1.10	0.50	10.00
Flax	64.10	16.70	1.80	2.00	3.90	1.50	10.00
Ramie	68.60	13.10	1.90	0.60	5.50	0.30	10.00
Hemp	67.00	16.10	0.80	3.30	2.10	0.70	10.00
Sisal	65.80	12.00	0.80	9.90	1.20	0.30	10.00
Abaca	63.20	19.60	0.50	5.10	1.40	0.20	10.00

Source: Batra, S. K. and Turner, A. J., 'The structure of textile fibres'. In C. Jarman, *Plant Fibre Processing: A Handbook*, Intermediate Technology Publications, UK, 1988.

tensile properties. The tensile strength and extension at break values of different natural fibres are shown in Table 11.4. The determination of melting point for fibre identification is mainly used for synthetic fibre identification as natural fibres do not melt. Some other properties such as the fibre length, the length of ultimates and fibre diameter (Table 11.5) can be used for fibre identification. Also the plant fibres can be distinguished by their chemical composition (mainly the cellulose and lignin content) as shown in Table 11.6.

There are significant differences in the burning characteristics of major natural fibres and hence can be used for their identification. One of the important properties used to compare the flammability of textile fibres is the limiting oxygen index (LOI). LOI is the quantity of minimum oxygen content (%) in nitrogen, necessary to sustain candle-like burning. The LOI values for some important natural fibres are: Cotton – 18, Silk – 23 and Wool – 25.

11.3.7 Infrared (IR) and Raman spectra analysis

The IR spectra of fibres are obtained by a double beam spectrophotometer or by a Fourier transform infrared (FTIR) spectrophotometer. The identification of generic fibre types is made by comparing the absorption spectrum of these fibres with reference spectra of known samples done on the same instrument until a match is found. This technique is more valuable for distinguishing between different fibres of the same generic class (Tungol *et al.*, 1990) and subclass (Bartick and Tungol, 1993; Grieve *et al.*, 1988; Tungol *et al.*, 1991, 1993). When the IR radiation interacts with a fibre, certain frequencies of energy are absorbed while others are transmitted or reflected depending on the functional groups (bonds) of the fibre which is used as a means for identification. Although the IR spectrophotometer is expensive,

it offers an unambiguous means of chemically identifying unknown fibres. Also when only a few fibres are available, this method is found to be the most valuable single test.

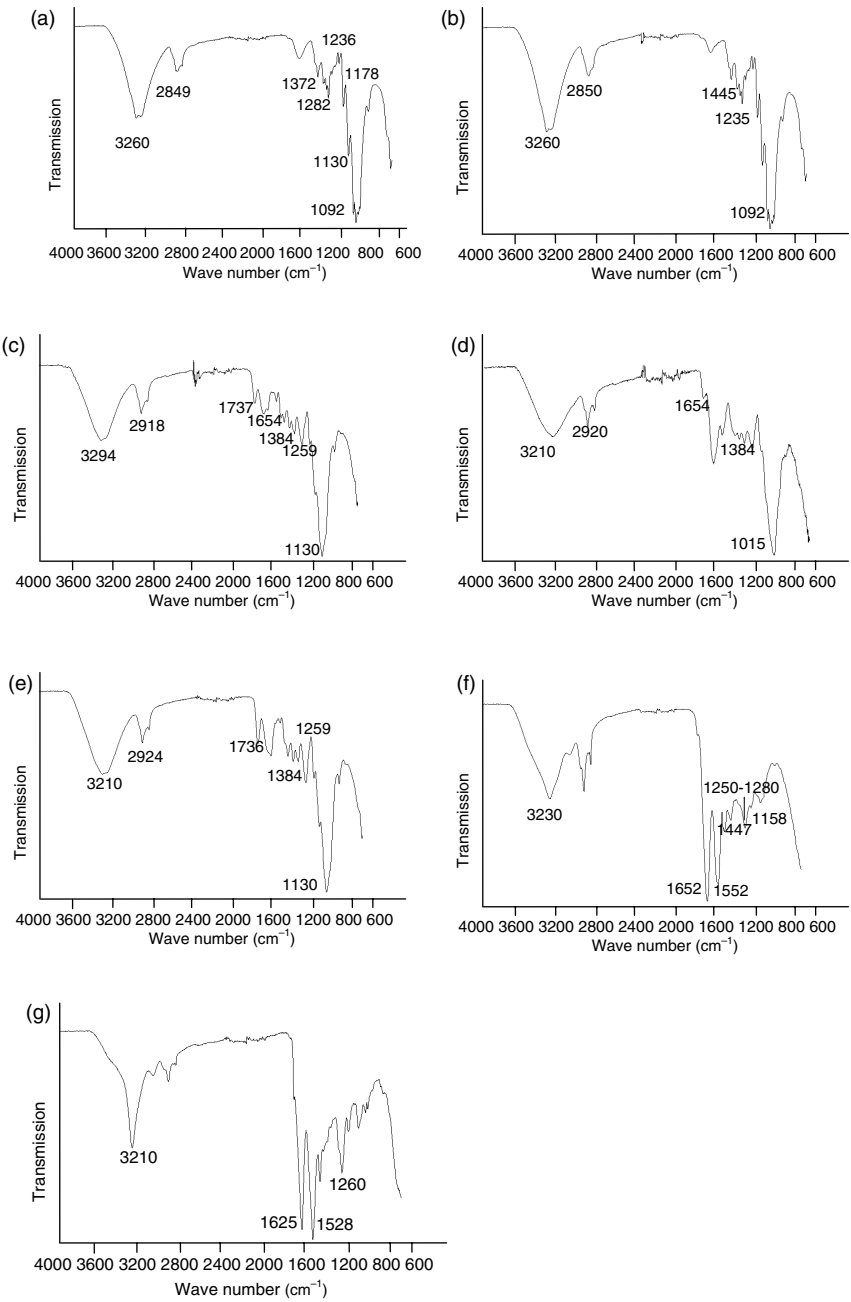
The IR spectrum of a sample is obtained by passing a beam of IR through the sample over a range of energies within the IR region; either a monochromatic beam or a Fourier transform instrument is used. In modern instruments, energy from an IR source is directed through the fibre by an interferometer. The track of the wavelength and the amount of light transmitted through the fibre at each wavelength are saved by a computer and the result is represented by a plot of wave number (cm^{-1}) against intensity (T %). IR spectroscopy along with a microscope can be used for the complete analysis of a single textile fibre.

The natural fibres exhibit more intra-sample variation in the absorption spectrum compared to synthetic fibres. The amount of twist in the fibres should be reduced by teasing apart and straightening the fibres before the FTIR test. The IR absorption spectrum of various natural textile fibres is shown in Fig. 11.1. Table 11.7 shows the assignments of absorbance frequencies of natural fibres.

Sometimes the fibre may have to be flattened to reduce the effects of spectral distortion due to the diffraction caused by fibre's cross-section. A metal roller in combination with a clean glass microscope slide is used for flattening the fibre, which is then carefully transferred to the specimen holder. As the fibres are flattened during the test and the morphology is destroyed, the minimum fibre length should be used. In some cases the available fibre is too short or too thin to obtain an FTIR spectrum by conventional means. In these cases, attenuated total reflectance (ATR), also known as internal reflection spectroscopy, may be used. In ATR mode, the fibre is placed in tight contact with an IR transparent, high refractive index crystal. IR spectrum is obtained by passing the IR radiation into the sample and crystal at angles greater than the critical angle. FTIR plays an important role in the forensic analysis of fibres (Grieve, 2000).

Garside and Wyeth (2003) developed an ATR technique using FTIR for the identification of natural fibres and used the peak intensity ratio for their differentiation on the basis of relative lignin content. If the composition of cellulosic fibres is considered, the relative proportion of lignin with respect to other cellular components seems distinctive. According to harvesting time and degree of maturity of lignocellulosic fibrous plants, the lignin content in the fibre is changeable (labile). The authors applied ATR spectroscopy for the comparison of the lignin-to-cellulose content of the plant fibres for identification.

Raman spectroscopy is complementary to IR spectroscopy and is also a powerful tool that can be applied in routine fibre analysis following optical microscopy and microspectrophotometry measurements. The fundamental



11.1 The infrared absorption spectrum along with the assignment of vibrations of various natural fibres: (a) cotton, (b) flax, (c) jute, (d) hemp, (e) sisal, (f) wool and (g) silk.

Table 11.7 The FTIR assignments (absorbance frequencies) of natural textile fibres

Cotton	Hemp	Flax	Jute	Sisal	Wool	Silk	Assignment
1042							C–O stretching
1092	1000–1162	1000–1162	1000–1162	1000–1162			C–C stretching
1130							Asym. bridge C–O–C
					1158		C–N stretching
1178							Asym. bridge C–O–C
1236		1235					O–H in-plane bending
					1250–1280		C–N stretching
			1259	1259			C–H bending
						1260	Absorption by amide III
1282							C–H stretching
1372	1384	1372	1384	1384	1447		C–H bending
		1445					$C–H_3$ asym. stretching
					1552	1528	Absorption by amide II
					1652	1625	Absorption by amide I
	1654		1654	1654			C=C stretching
			1737	1736			C=O stretching
2849		2850					Sym. $C–H_2$ stretching
	2920		2918	2924			C–H vibration
3260	3200–3570	3200–3570	3200–3570	3200–3570	3200–3570	3200–3570	O–H stretching

principles of Raman spectroscopy and IR spectroscopy are different, the former being a scattering process while the latter is a pure absorption phenomenon. The spectroscopic selection rules for Raman and IR activities are different and they produce complementary vibrational data. Many IR modes that are weak or not permitted in IR spectrum are very strong in Raman spectrum, e.g. molecular vibrations which involve nonpolar bonds are strong in the Raman spectrum, and those vibrations involving polar bonds are strong in the IR spectrum.

Raman spectroscopy can be conducted while the fibre remains mounted on a glass slide under cover slip as glass produces little response to Raman spectroscopy. But FTIR cannot be conducted with the fibres mounted to the glass slide due to the strong absorption of IR radiation by glass. Raman spectroscopy is very useful for the discrimination of dyed fibres and for complete fibre characterisation sequence.

Raman spectroscopy was used to discriminate amongst untreated plant fibres on the basis of peak ratios derived from the associated C–H and C–O–C vibrations (Edwards *et al.*, 1997). Raman spectroscopy has been used for the analysis (industrial and forensic) of fibres and has potential as an analytical tool for fibre examination. The application of Raman spectroscopy for characterising natural and synthetic fibres, and forensic analysis by the European Fibres Group (EFG) (Wiggins, 2003) has been reported. Raman spectroscopy can also be used to identify the main dye type present in a coloured fibre.

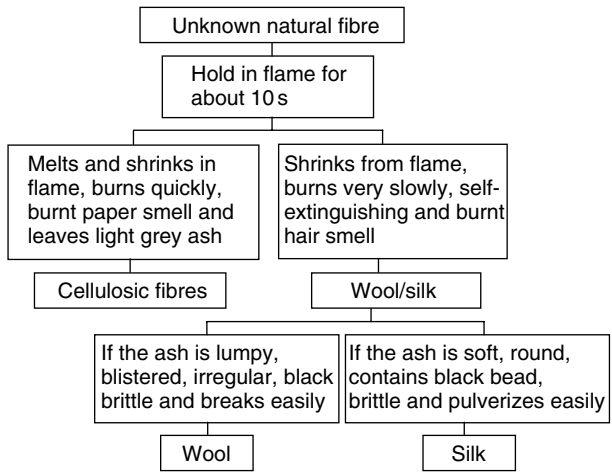
11.3.8 Other approaches

In several cases, other techniques such as transmission electron microscopy (TEM), chromatography and pyrolysis can be used for fibre analysis. These techniques along with some other methods (burning or solubility test) can be used for complete fibre identification. Unlike the light microscope and SEM, the TEM cannot be used to examine whole textile fibres directly. TEM is mainly confined in studying the fine internal structure of single fibres or parts of fibres and chemical reactivity. TEM is not directly used for fibre identification (Johnson and Sikorski, 1965); rather it is used indirectly by resolving the structure of the crystalline regions in the fibre (Sikorski, 1975).

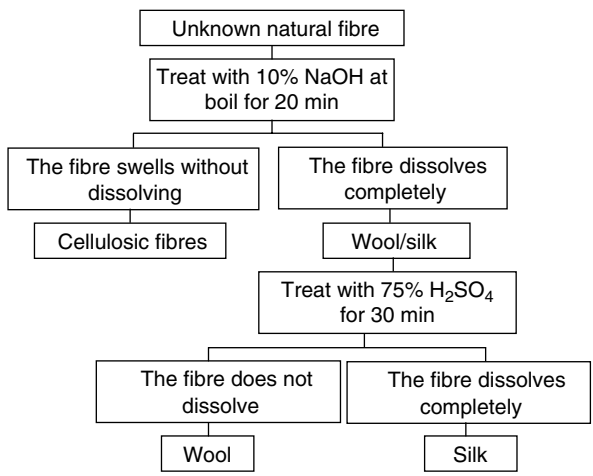
Various chromatography methods such as gas, high-pressure liquid and thin-layer techniques can be used for the chemical analysis of textile materials (Weaver, 1984). Pyrolysis is mainly used for the identification of synthetic fibres. A method of qualitatively identifying a fibre without any elaborate equipment, with the aid of a simple solvent separation process combined with preliminary tests was suggested by Rist (1987).

11.4 Practical approach

Microscopy and burning test are described (Garner, 1967) as the first step towards fibre identification. An unknown fibre is investigated under the microscope for the longitudinal and cross-sectional appearance (see Table 11.2). Then the burning behaviour of the fibre is observed by holding a small tuft of the unknown fibre in forceps into the flames of a micro-burner for about 10 s (see Fig. 11.2). For the confirmation of the fibres solubility test (Fig. 11.3) and staining test (Section 11.3.5) should be used. In the staining



11.2 Scheme for identification of fibres according to burning test.



11.3 Scheme for identification of fibres according to solubility test.

test a small fibre tuft is stained in Shirlastain A at room temperature for 1 min, rinsed under cold tap water and compared with the colour chart for identification. The ultimate fibre identification is done by repeating the above tests, carefully considering the information given on various methods of identification in Section 11.3 and considering the physical properties.

ASTM (2000) describes the solubility test for the preliminary identification of the generic type of an unknown fibre. Alternatively, the IR spectra (obtained by an FTIR or a double-beam spectrophotometer) can be compared with the decision chart for the preliminary identification. The plant (native cellulose) and animal hair fibres are distinguished by the microscopical examination of longitudinal and cross-sections. Additional physical properties such as density, melting point, regain, refractive indices and birefringence are used for confirmatory identification.

11.5 Forensic analysis

Forensic analysis of fibres, which rarely follows the industrial practice, requires knowledge of fibre manufacturing processes and methods. Textile fibres are one of the most important forms of trace evidence in the context of criminal evidence. Forensic scientists can examine this trace evidence and compare the fibres to a known fibre in order to discover possible common origins. The factors influencing the nature of fibre adherence to materials include type of fibre transferred, type of receiving material and the extent to which the receiving material is used after transfer.

In the case of forensic analysis, apart from the murky origin and uncertain provenance, the sample size is limited (sometimes only 1 or 2 fibres). Therefore, microscopy is the primary method of fibre identification. The preservation of the sample is the main concern. The focus of forensic scientists is fast, non-destructive, selective and sensitive examination methods.

In forensic analysis, IR spectroscopy is mainly used to determine the chemical composition of fibres, providing a generic fibre class and subclass. Thomas *et al*. (2005) used Raman spectroscopy for the forensic analysis of black/grey and blue cotton fibres (reactively dyed) by varying the laser wavelength. The study has shown that this technique can be used for dyed fibre identification without interference from the cotton substrate, thereby providing molecular information about the main dye used. However, caution must be taken in the choice of wavelengths, as the fluorescence and changes in sample spectra can occur with laser wavelength.

Wiggins *et al*. (2007) investigated the use of calculating the first derivative of the absorbance spectra of fibres as a tool for forensic analysis. The first derivative of the absorbance spectra provided more points of comparison which facilitated discrimination when the absorbance spectra produced for some samples were broad and featureless. Although it was found that the

calculation of the first derivative can be a useful aid in the comparison of spectra, a high degree of caution is required while applying this method to fibres that exhibit a large intra-sample variation in colour.

The application of the new combined μ-Raman and μ-X-ray fluorescence (XRF) spectrometer (PRAXIS apparatus) in the forensic analysis of fibres and other materials has been reported (Zieba-Palus *et al.*, 2008). The study confirmed the usefulness of the apparatus in criminalistic traces and the strong point of the instrument is that it provides a combination of two non-destructive methods in one instrument. The analysis is characterised by good repeatability, reproducibility and compatibility. Lepot *et al*. (2008) analysed five forensic fibre cases by Raman spectroscopy and found it as a good complementary method for molecular spectroscopy (MSP). The forensic analysis of dyed textile fibres by using chromatography, spectroscopy and mass spectrometry has been reported. There are also other important works in forensic analysis by several researchers (Causin *et al.*, 2006; Goodpaster and Liszewski, 2009; Lang *et al.*, 1986; Paulsson and Stocklassa, 1999; Miller and Bartick, 2002).

11.6 Future trends

Recently, a wide range of analytical methods such as FTIR, Raman spectroscopy, electron microscopy, atomic force microscopy and vibrational spectroscopy are available in addition to optical microscopy for fibre identification. Yet as far as the analysis is concerned, the old methods are the best. Microscopy still dominates in fibre identification by providing a range of analysis barely possible with any other methods. Combined with an analytical bench, such as IR or Raman, it is the quintessential fibre identification tool. Microscopy should be the first method of choice for any fibre scientist.

New developments in technical and medical textiles, micro- and nanofibres, and speciality fibres broaden and deepen the analytical world of the fibre analyst. Some other new techniques include multiphoton fluorescence microscopy, optical coherence tomography and confocal microscopy. Although the first two instruments were developed especially for bio-sciences, it is likely that these will become more valuable in relation to fibre identification. Confocal microscopy is an advanced form of optical microscopy which can measure lower than 200 nm (not feasible in optical microscopy).

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11.8 Appendix: abbreviations

AATCC	American Association of Textile Chemists and Colorists
ASTM	American Society for Testing and Materials
ATR	attenuated total reflectance (a sampling technique used to enable infrared spectroscopy to be carried out directly on samples in the solid state)
BIS	Bureau of Indian Standards
CS	cross-section
EDX	energy dispersive X-ray (an analytical technique used for the elemental analysis or chemical characterization of a sample)
EFG	European Fibres Group
FTIR	Fourier transform infrared (a technique used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas)
IR	infrared (the electromagnetic radiation with a wavelength between 0.7 and 300 μm)
LOI	limiting oxygen index
SEM	scanning electron microscope (a microscope which uses an electron beam rather than light)

STM	scanning tunnelling microscope (an instrument for imaging surfaces at the atomic level)
TEM	transmission electron microscope (a microscope in which an image is formed by the interaction of the electrons transmitted through the specimen. The image is magnified and focused onto an imaging device such as a fluorescent screen on a layer of photographic film, or to be detected by a sensor such as a CCD camera)
XRF	X-ray fluorescence (an analytical technique used for elemental and chemical analysis especially in the investigation of metals, glass, ceramics and building materials; and for research in geochemistry, forensic science and archaeology)

12

Testing of natural textile fibres

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Abstract: The range of natural fibres and the diversity of their important properties are so broad that it is impossible to provide a comprehensive coverage of appropriate testing methods in one short chapter. For the most important fibres, cotton and wool, many excellent books have been previously published. The characteristics of natural fibres are diverse between the different fibre types. The variability within any specific type poses challenges for sampling to ensure that results are representative and reliable; sampling therefore forms an important aspect of the early content of the chapter. The remaining parts deal, in Section 12.4, with testing for the key physical properties common to most natural fibres and some of the testing methods available; in Section 12.5, with a limited range of tests for chemical properties applied to natural cellulosic fibres; and finally, in Section 12.6, with examples of modern physical and chemical analytical methods with the focus upon examples of how these have been adopted to investigate fibre properties. The importance of testing to meet the demands of the international trading environment and process automation leads to the conclusion that global standardisation of test methods and quality specifications is likely to become even more important in the future.

Key words: natural fibres, testing, fibre properties.

12.1 Introduction

Testing of natural fibres is undertaken for a range of reasons and is a necessary aspect of the determination of fibre quality in respect to the suitability of fibres for particular end uses. The need for characterisation of the physical properties of fibres, e.g. length, fineness, strength, elongation, colour, trash content, etc. are generally directly related to subsequent conversion into products, such as yarns, felts, woven and knitted fabrics, industrial and agro-textiles, and are related to the suitability of the fibres to provide the products which meet the end-user specifications. Fibre properties fall into two groups. First those properties which are characteristics of the fibres themselves and which can be related directly to the quality of the fibre in specific areas of application and their performance in production processes. Fibre fineness and length are important examples of these intrinsic fibre

properties: they are fundamental characteristics which affect, for example, fibre spinnability, the quality of yarns and the limit of yarn count that can be achieved. Second, natural fibres usually contain non-fibre contaminants generated in production or left over from the extraction processes, e.g. plant debris such as shives and cuticular matter, dust, grease, soil and vegetable matter. Such contaminants are important when determining fibre yield but can be of more fundamental importance when they limit the uses of fibres in target applications.

The use and usefulness of test data are, naturally, dependent upon the fibre type and end use. Skill and experience play an essential part in the interpretation of the data within the diverse specialist areas of the natural fibre industries and on the particular testing required for production and quality control. Testing plays a very different role in research and development where the speed of generating test results, and the costs involved, can often be of less importance than the quality of the data generated. As a result, many of the test methods used in research have not been adopted for general use in the industrial environment.

Chemical testing of natural fibres offers a diverse set of tools which fall into two broad areas of application: testing for the extent and causes of 'chemical' damage and for characterisation of fibres so that subsequent chemical and other wet processes can be optimised. However, this review focuses mainly on physical methods of testing and chemical methods are not considered here.

There are many sources which detail standard procedures for routine testing of fibres. The International Organization for Standardization (ISO) designates standards; the membership of the ISO comprises representatives from various national standards organisations. As well as publishing international standards they also produce technical reports and technical specifications. The European Committee for Standardisation (CEN) exists to develop European Standards (ENs) in various sectors to build a European internal market for goods and services and to position Europe in the global economy. National standards organisations include the British Standards Institution (BSI) and the German Institute for Standardisation (DIN); both are members of ISO. ASTM, originally known as the American Society for Testing and Materials, predates both BSI and DIN and is an international standards organisation in its own right but it is not a national body in the USA, this is the American National Standards Institute (ANSI) in the USA. However, ASTM does develop standards in the USA in collaboration with volunteer committees across the world.

Note, it is not the intention of this chapter to be a catalogue of standards information but to focus upon some of the more important testing methods, recent innovations and their uses. There are many excellent publications on textile testing, some of which are focused on natural fibres, which contain

extensive information on test methods; some of these are listed at the end of the chapter.

12.2 Key issues in testing natural fibres

In comparison with manmade fibres, which are produced with known uniform properties, natural fibres lack consistency due to the fact they are produced under varying conditions. For example variations in climate, soil type, and local environment, mean that fibre properties will vary between locations. For this reason there is a need to test a range of fibre characteristics to assess their suitability for designated end uses.

12.2.1 Reasons for testing

By their very nature, natural fibres vary widely in their properties depending upon a whole range of factors including agronomic, environmental, geographical and geographic variables, species varieties and breeds. Because of the international dimensions to trade and manufacturing it is important to have recognised quality standards for raw materials. For cotton and wool, classification and grading standards have been established for many years, and there are comprehensive sets of international standards for testing and quality evaluation. However, manual grading is still commonplace in many of the smaller production countries of the world. Despite the existence of grading standards, processors will often undertake in-house testing of raw materials in order to optimise their processing and to confirm that input materials are appropriate for their requirements.

Quality classification systems exist for all fibres. Table 12.1 shows the factors upon which the gradings of a range of fibres are based. Internationally recognised methods exist for testing the characteristics of many fibres but for many of the low-volume fibres quality is evaluated subjectively and testing is not automated and is hence labour intensive; as a result grading of fibres at source is generally carried out subjectively and manually by skilled graders; testing by end-users is often an essential part of their production activity.

12.2.2 Sampling

As with almost all products obtained from animal or plant origins**,** natural fibres show a wide variation in their properties, therefore proper selection of representative samples and statistical analysis are necessary to cope with the variation. Obviously it is not possible, or reasonable, to sample the total amount of raw material; it would be too expensive and time consuming,

Table 12.1 Grading factors for a range of fibres

	Strength	Length/length distribution	Diameter	Fineness	Elasticity	Colour	Cleanliness
Cotton*		√	√	√		√	√
Linen flax	√	√		√		√	√
Short fibre flax		√	√	√		√	√
Hemp	√	√		√			√
Ramie		√		√		√	√
Nettle	√	√	√	√		√	√
Coir							
Jute	√			√		√	√
Wool*	√	√		√	√	√	
Silk				√			

*Automated methods used routinely in some countries.

furthermore many tests are destructive. Therefore samples of the material have to be taken for evaluation and the amount of material actually tested is often a very small proportion of the total. It is, however, imperative that the sample is an appropriate representation of the whole. For this reason one must obtain an unbiased sample from the bulk material so that the properties to be evaluated are adequately representative of the properties of the whole. Test data can then be used with confidence. For example, if fibre diameter is the property being examined, the results should be an adequate representation of the bulk material and enable the prediction of performance in subsequent processes and products.

The total amount of a material under test can be seen as a collection of individual units (e.g. fibres) and in statistical terms this is termed the *population*. The characteristics of a population which differ between the individuals are termed the *variables*; for clean fibres these will be properties such as fibre length, diameter and strength. A *sample* is a subset of a relatively small number of *individual*s selected to represent that population. Because of the variation found in natural materials a sample must consist of a sufficient number of individuals for data to be meaningful. For this reason selection of the individuals comprising the sample should be done in such a way to ensure that a *random sample* is obtained and *sample bias* is avoided. A very variable sample will require a large number of individuals in the sample to obtain an adequate representation.

A *numerical* sample is defined as one where all the fibres in the population have an equal chance of selection. In a perfect numerical sample the proportion by number of, say, long, medium and short fibres would be the same in the sample as in the whole population. However, such perfection is practically impossible and therefore a random sample is normally chosen. A random sample will have a *random error* associated with it, i.e. repeated measurements of the same property will produce different values scattered randomly around a central (mean) value. This error can be estimated by statistical analysis.

When the sample selection is influenced by factors other than chance, a sample ceases to be truly representative of the bulk and a *biased* sample results; bias may be attributed to the person doing the sampling. The effect of bias may be negligible or considerable and is characterised by test measurements being either consistently higher or lower than expected. Bias is also referred to as *systematic error* and cannot be detected by statistical analysis of test results.

Sampling methods for fibre will depend on the form in which the fibre is available, e.g. fibre in bales, sliver, carded web, yarn, etc., will demand different sampling techniques. The aim of sampling is to minimise random and bias/(systematic) errors. Fibre sampling will be affected by the degree to which fibres have been mixed or blended. If the bulk sample has been thoroughly

mixed or blended then the sample can be selected from one area with the assumption that fibres from all the original sources of supply are represented. However, if mixing was only partial, or if it is known that the material may vary in different regions of the bulk material, then samples should be taken from all parts of the bulk to ensure a representative test sample.

Fibre sampling methods are outlined below; a comprehensive explanation of different fibre sampling methods is given by Booth (1968) and Saville (1999).

Fibre sampling from bulk

When a contract is established between a buyer and a seller of fibre or yarn, it is usually a requirement that an independent third party determine the invoice mass of a consignment. The method of sampling to be used for commercial bales of fibre and containers of yarn is described in BS ISO 6741-2:1987. In order to assess fibre, yarn and fabric quality from bulk consignments a range of different sampling methods will be required. BS EN12751:1999 describes sampling for acceptance testing to estimate without bias the desired property of the lot being evaluated.

Zoning

Zoning fibre in bulk form is likely to vary significantly from place to place throughout the bulk and the zoning method is used for selecting samples. A sample of fibre is taken from at least 40 different places, or zones, in the bulk material; these should be widely spaced apart. Each of these samples is divided into two sub-samples and one sub-sample is discarded. The process is repeated with the remaining sub-samples, i.e. each is divided in two and one half is discarded; the process is repeated again until about n/x fibres remain in the sub-sample (n = total number of fibres required in the sample, x = the number of original samples). Each of the 40 or so samples originally selected are treated thus, the fibres remaining are collected together to give the required sample containing 'n' fibres. This method can be used for selecting samples from bulk raw cotton or other loose fibre.

Core sampling

The core sampling method was developed to obtain a laboratory sample representative of fibre in unopened bales of raw wool, and may be used to assess the moisture content, grease and vegetable matter present and can be adapted for other fibre types. The density of the packaged fibre must be suitable for core sampling to take place: too loose or too dense and it is not applicable. A predetermined number of core samples are withdrawn from the bulk material by forcing a 60 cm tube with a sharpened tip into the bale.

The tubes are sufficiently long to penetrate halfway into the bale, and coring is carried out from both ends and the sides of bales. All cores are combined when removed from the bale and stored in an air-tight vessel, and the weight of the bale and moisture content can be determined.

Fibre sampling from combed slivers, roving and yarn

Sampling sliver or yarn may lead to a length-biased sample as the fibres are laying approximately parallel to each other. This means that the longer fibres are more likely to be selected during sampling. Length-bias should be avoided where fibre length is the property to be measured, but length-bias will also impact upon fineness and strength measurements as these often vary with fibre length. Length-bias can be overcome by preparing a numerical sample, or alternatively a length-biased sample which allows for the bias to be incorporated into any calculation.

Textile testing is carried out for a wide range of particular reasons which depend upon the specific circumstances and the information required. Commonly, testing is carried out to give an indication of performance of a material, to assess and compare qualities of materials, to estimate the content of useful fibre, to determine the effects of different processes or to see whether the material meets a particular specification. Whatever the purpose, it is imperative that test methods are reproducible. For example, when the same material is tested by a different operator or in a different laboratory the results should be sufficiently similar, within experimental error, for the same conclusions to be obtained. Different operators may each have their own individual way of carrying out a test procedure, for example there may be variations in how different people take measurements, or make instrumental adjustments; in all situations the procedures used should be specified in order to minimise operator-based variations. It should also be borne in mind that when testing natural fibres they are responsive to different atmospheric conditions and their properties may vary according to local temperature and humidity; standard conditions are commonly required for testing and should be specified (normally 20 ± 2°C and 65 ± 2% RH). Similarly reproducibility in test procedures should be ensured between different laboratories and different dates. If test results vary significantly between laboratories then the test method must be deemed unreliable. However, values obtained from repetitive testing of textile materials would not be expected to be identical and therefore statistical analysis needs to be carried out.

12.2.3 Standardisation of test methods

To ensure reproducibility of test results, and facilitate trading in the global marketplace, test methods need to be standardised and, as mentioned

earlier, most countries have their own standards organisations e.g. the British Standards Organisation, ANSI, ASTM International (USA) and Deutsches Institut für Normung (DIN, Germany). The European Committee for Standardisation (CEN) comprises national members from 31 European countries with the remit to develop voluntary European Standards (ENs). These European standards are also national standards in each of the 31 member countries, and replace any conflicting national standards. The aim of CEN is to remove trade barriers for European industry and consumers, and enable products to reach a wider market with lower development and testing costs. Similarly on a global scale the Vienna Agreement of 1991 is a joint agreement on technical cooperation between ISO and CEN for harmonisation of international and European standards.

12.3 Test methods for natural fibres

Historically the assessment of the 'quality' of natural fibres was carried out subjectively by skilled individuals, often aided by physical reference 'standards'. This approach is still commonly used in most of the less-developed fibre-producing countries. Today's international trade has led to the development, and wide acceptance, of internationally approved standard test methods including automated instrumental methods for the assessment of quality parameters. Standard methods are highly developed for cotton and wool but for many other fibres there is still a long way to go before objective testing of natural fibre materials becomes the norm. The USA have led the development of test methods for cotton and, more recently, the principles for cotton trade 'harmonisation' have been established (HS 52) as part of the harmonised commodity description and coding system. The methods developed for wool grading have been established primarily from research undertaken in Australia and New Zealand.

Some progress has been made for international grading for the quality of other natural fibres but their importance in international trade is often localised and has not received the attention given to cotton and wool. That said, the current exploitation and studies of the potential uses of flax and other bast fibres require a standardisation system, and this has been addressed by the FAO/ESCORENA European Cooperative Research Network on Flax and other Bast Plants (Kessler and Sharma, 2011). Procedures adopted for testing wool have been relatively easily adapted to other hair fibres but most of the cotton tests that are based upon objective methods have not been easy to apply to other cellulosic fibres. The need for the establishment of standard test methods for flax has been recognised in the main producing countries for many years but the only real progress has come from ASTM International (e.g. ASTM D-6961-03; ASTM D6961/D6961M-09; ASTM D-7025-09; ASTM D-7076-10).

The end use of fibres determines the relative importance of testing. For example, it is not necessary to test the quality of coir or jute using the rigour and range of methods applied to high-value fibres intended for use in fine woven or knitted apparel. Table 12.1 shows the more important characteristics which are important in the assessment of quality for a range of natural fibres.

12.4 Measuring the physical properties of natural fibres

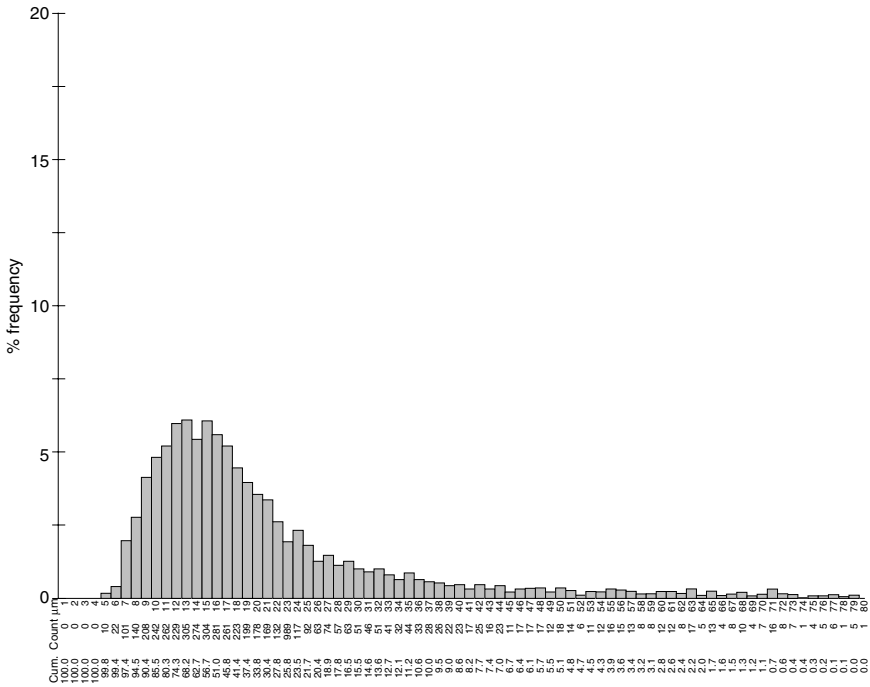
Consideration of the physical properties of fibres such as fibre diameter, fineness and stiffness is imperative for quality control purposes. Fine fibres are used for the production of fine yarns and therefore are of high value. Similarly the stiffness of a fibre impacts on its spinnability and therefore affects yarn and fabric properties. Fabric drape, handle, prickliness and softness are all dependent on the stiffness of a fibre used in its production.

12.4.1 Fibre diameter and fineness

Fibre fineness/diameter is one of the most important properties of fibres and is influential in determining end use. Fibre diameter is normally measured in micrometers, commonly abbreviated to microns (μm). The visual appearance and handle of fibre are influenced by the fineness and this will obviously have an impact on the value of the fibre. Generally the value of a natural fibre for use in apparel and many domestic textiles increases with decreasing diameter but for some industrial and technical textile applications the relationship can be reversed. A simplified example is that, because there is a minimum number of fibres needed in the cross-section of yarns to provide strength and uniformity, fine yarns can only be satisfactorily made from fine fibres; fine yarns are more highly valued and are used in high-value products so, other factors being constant, fine fibres generally have higher values than coarse, thicker, fibres.

Fibre stiffness (resistance to bending) increases with increasing fibre diameter, i.e. the coarser the fibre, the stiffer it is. Bending rigidity of perfectly elastic fibres of circular cross-section is proportional to the fourth power of the fibre diameter ($B \propto d^4$); whilst most natural fibres are not perfectly circular, or perfectly elastic, the bending stiffness is still very sensitive to fibre fineness. The stiffness of a fibre affects the 'spinnability' and yarn properties; fibre stiffness also affects properties of fabrics such as drape, handle, prickliness and softness.

The diameters of natural fibres vary considerably; fibre diameter does not have a single absolute value and can range widely about the mean value even when fibre is from the same source; because of this the use of single values to characterise fibre diameter (e.g. mean, median or airflow) is often inadequate for assessing diameter characteristics, for example short flax fibres will always

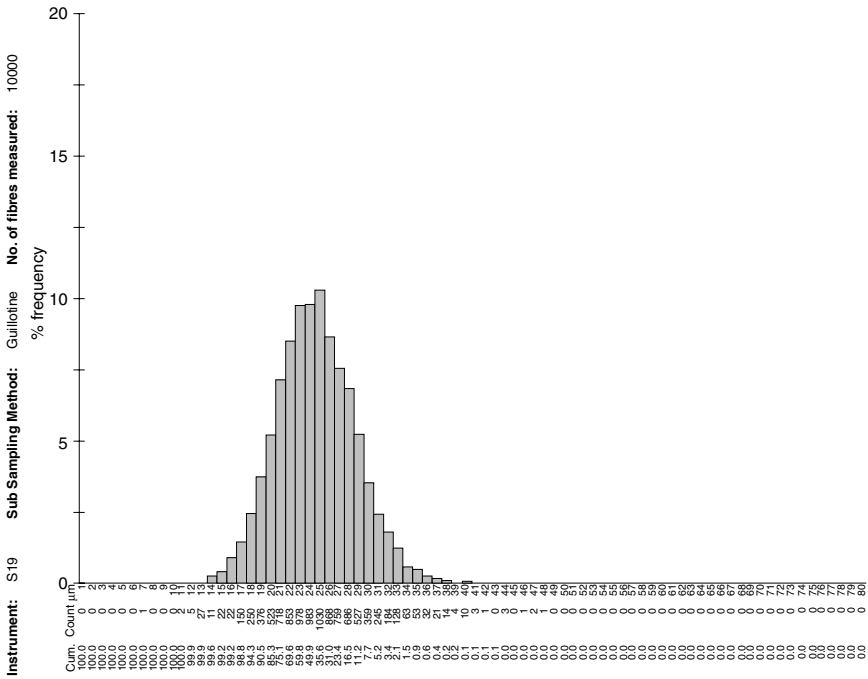


12.1 Fibre diameter distribution of short fibre flax (Laserscan).

have a wide, skewed, distribution in diameter values even within batches from the same crop (see Fig. 12.1). Similarly, wool obtained from a single sheep will show variation but the distribution of diameters is less skewed (Fig. 12.2).

Another factor that needs to be considered when measuring fibre fineness is that the cross-section of different fibre types varies in shape; wool has an approximately circular cross-section, raw cotton has a collapsed tubular cross-section and bast fibres are often irregular polygonial in cross-section (Fig. 12.3(a)–(f)). Furthermore natural fibres usually exhibit cross-section variability along their length.

Fibre diameter is not a particularly useful measurement in all circumstances, particularly when the deviations from circular cross-sections are significant and alternative ways have been developed to represent fibre fineness. Fibre fineness is often measured in terms of fibre linear density, e.g. decitex (mass in grams of 10 000 m of fibre) or denier (a historical term originally used to express the fineness of silk, defined as the mass in grams of 9000 m of fibre). Indirect assessments of fibre fineness were some of the earliest to be developed, especially for quality control purposes, and have been particularly valuable in the cotton and wool industries; the most common of these are the 'airflow' methods, based upon the resistance of a standard mass of fibre to air flowing through the fibres.



12.2 Relative fibre diameter of a wool sample (Laserscan).

12.4.2 Methods for measuring fibre fineness

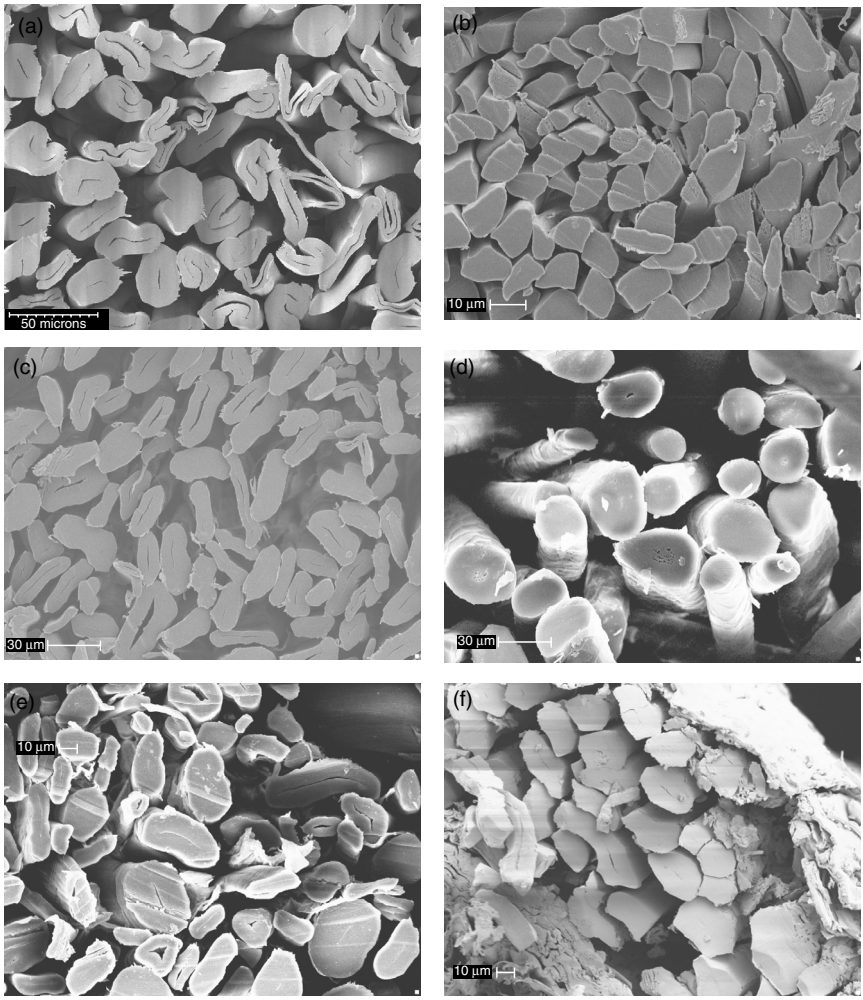
A series of 14 papers by Sommerville (2001–2007) reviewing methods of measuring fibre fineness are essential reading for those interested in this topic.

Cross-sections

Microscopy offers the only direct method for measuring the absolute characteristics of fibre cross-sections. Because of the irregularity of the cross-sections of most natural fibres, values of 'diameter' are usually an average based upon a large number of 'diameter' measurements. Examination of fibre cross-sections is rarely used nowadays as it requires skill and experience, and is slow, expensive and highly labour intensive.

Fibre diameter

The most common methods for measuring diameter are based upon determining the width of short snippets of fibre using a projection microscope but the processes are still laborious and are now only used for the characterisation of 'standard' or 'reference' samples which are to be used as calibration standards for other methods.



12.3 (a) Cotton fibre cross-section; (b) silk fibre cross-section; (c) ramie fibre cross-section; (d) wool fibre cross-section; (e) nettle fibre cross-section; and (f) flax fibre cross-section.

Automation of diameter measurement for wool fibres has passed through many stages of development, reviewed by Sommerville (2001–2007) and today there are a number of commercially available instruments which include the Optical Fibre Diameter Analyser (OFDA) (Australia), the Sirolan Laserscan® (Australia), and the FibreShape (Switzerland) systems.

OFDA

The potential for using image analysis for automating the measurement of fibre diameter from microscope images was developed at the

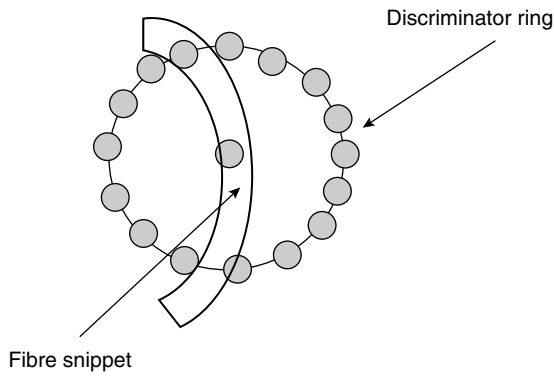
Australian Wool Testing Authority Ltd in the early 1980s. This led to the current range of OFDA systems produced by BSC Electronics Pty Ltd. OFDA 100 was originally developed to determine the average fibre diameter and standard deviation of wool fibres and is widely used by testing houses and mills across the world. The OFDA 100 system was developed for wool; its successor, OFDA 2000, determines a wide range of quality parameters including: mean diameter (range 4–300 μm), standard deviation, and curvature for fibre types which have a circular, or near-circular cross-section including wool, cashmere, mohair, glass and synthetic fibres (Stobart and McColl, 2000). Recently OFDA 5000 has been introduced for measurements on micro-fibres (diameter range 0.5–60 μm). OFDA 100 has been used for determining useful characteristics of other fibres including some bast fibres, but the data have to be interpreted to take account of the irregularity of the fibre cross-sections; nevertheless the 'relative' fibre diameter data has proved very useful in a range of applications (Akin, 2010; Drieling *et al.*, 1999; Grishanov *et al.*, 2006; Wang and Wang, 2004).

The OFDA 2000 comprises an automated microscope which captures images of a moving sample of fibre snippets (normally 2 mm long) which are spread automatically onto a 70 mm square glass slide which is then scanned by the microscope. Software identifies and measures individual fibres to a resolution of 1 μm; the mean diameter and standard deviation are calculated to a resolution of 0.01 μm. The whole process takes a few minutes for, typically, 4000–20 000 measurements. The results are printed out as a histogram of the fibre diameter distribution and software generates values for coefficient of variation, prickle factor and spinning fineness (the last two relating only to wool and similar near-circular fibres).

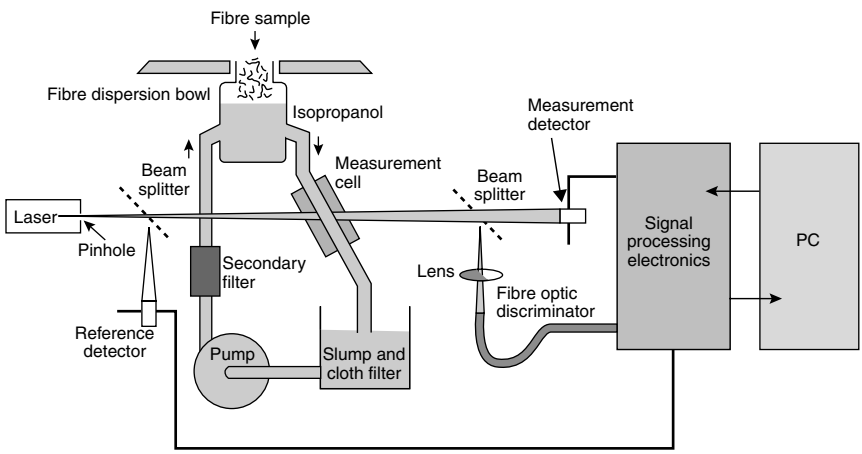
Sirolan™ Laserscan

Research on the use of photometry to determine fibre diameter was undertaken by the AWTA alongside the image analysis approach which led to the early OFDA machines. The principle is relatively simple: when a beam of parallel light (from a laser) shines upon a photo-detector, fibres at right angles to the beam of light will cast a shadow on the photo-detector which, if the fibres are of constant length, will have an area directly proportional to the fibre diameter. The key to the success of this approach was the development of an optical discrimination system (Fig. 12.4), which ensures that only single fibre snippets that fully intercept the laser beam are measured. Figure 12.4 shows a schematic diagram of the principle of the 'optical discriminator'. Fibres to be measured must cross the central detector and two (but only two) of the outer detectors. In this way only single whole fibres with an appropriate alignment are measured.

In order to measure fibre diameter using this principle samples should be appropriately prepared in terms of cleanliness and fibre separation (e.g. wool is scoured to remove grease and dirt, dried and conditioned). When the samples are in suitable condition fibre snippets of 2 mm length are cut from an aligned random sample of fibre using a special guillotine. Figure 12.5 illustrates the general features of the Sirolan™ Laserscan system: small quantities of the snippets are introduced into a pre-measurement dispersion bowl containing an isopropanol/water mixture (which emulates standard moisture regain conditions) until the required number of measurements have been taken. The dispersion of fibres flows through a measurement cell where fibre snippets intersect a laser beam; those correctly aligned are



12.4 Principle of the Laserscan optical discriminator (courtesy CSIRO).



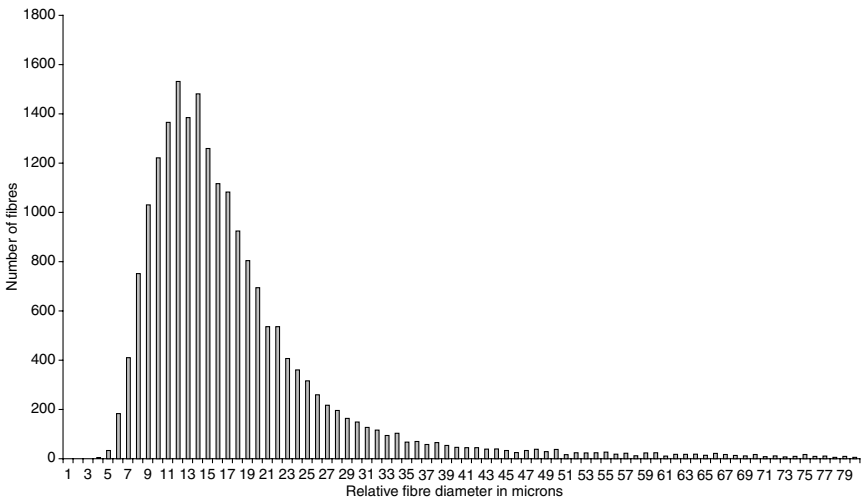
12.5 Principle of operation of the Sirolan Laserscan system.

detected and measured. Typically 2000 to 10 000 measurements are taken and the software yields values of mean diameter, standard deviation, CV%, percentage of fibres less than 30 μm (comfort factor for wool), fibre curvature and a histogram (and data file) of the diameter distribution (Fig. 12.6) of fibre diameters up to 80 μm.

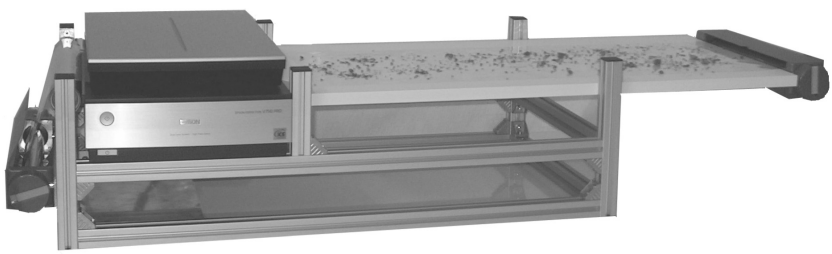
As with the OFDA systems, the Sirolan™ Laserscan was targeted at wool but has been successfully used for many other fibres including other protein fibres, flax and hemp (Grishanov *et al.*, 2006; Lamb and Denning 2003; Miao and Finn 2008; Wang and Wang 2004).

FibreShape™

FibreShape (Fig. 12.7) is a versatile quality control and characterisation system which combines a high-resolution scanner with advanced image analysis



12.6 Distribution of relative fibre diameter of a cottonised flax sample (Laserscan).



12.7 FibreShape instrument (courtesy FibreShape™).

software. The analysis of a scanned image provides mean fibre diameter, standard deviation and a histogram of diameter distribution; fibre length and shape information can also be determined. The FibreShape system is available with either manual or automatic sample preparation; it is a relatively low-cost system useful for quality control measurement of a wide range of characteristics for many natural fibres (Schmidt *et al.*, 2002, 2010). FibreShape offers a rapid characterisation process but the resolution is lower than with other methods, though there is good general correlation. Fibres of a fineness range from 5 μm up to 5000 μm (5 mm) can be measured (using appropriate software, fibre lengths from 2 μm to 30 cm can also be measured).

The performance of automated diameter measurement systems need to be checked and recalibrated using standard reference materials. For the OFDA and Laserscan machines this is achieved using standard wool tops where the mean fibre diameter and diameter distribution have been previously determined by direct measurement using a projection microscope (available from INTERWOOLLABS), but other reference materials may also be used. In some instances recalibration can be more troublesome, e.g. returning the machine to the manufacturer for recalibration.

The Laserscan, OFDA and FibreShape systems have many uses in the natural fibre industry beyond wool characterisation as they enable the measurement of mean fibre diameters and diameter distributions for quality specification, quality analysis, research and development for many fibres.

Flax and hemp have received a lot of attention in recent years particularly towards modification of flax and hemp tow and short fibre crops by enzyme and chemical 'retting' to produce cottonised fibre for use on short-staple spinning systems. The main issue is that flax and hemp fibre are not, as harvested and decorticated, single fibres but a mixture of single, elementary, fibres and bundles of elementary fibres held together by inter-fibre cement. During the carding and spinning processes these bundles partially split into single fibres, but a proportion of bundles will remain intact. Enzyme and chemical retting treatments have been shown to improve bast fibre separation; high energy ultrasound and shock waves have also proved successful for cottonising bast fibres (Akin *et al.*, 2001; Brown, 1984; Fila *et al.*, 2001; Henriksson *et al.*, 1997; Harwood *et al.*, 2008b; Sirghie et al., 2005). Instruments that produce histograms of fibre distribution are particularly useful for observing the effects of retting and cottonisation treatments as the changes in the diameter distribution give a quantified picture of the process.

Although flax fibre cross-sections are irregular polygons they can be considered to be approximately circular. Changes in diameter distributions assist the quantification of cottonisation treatments. Attempts have been made to produce estimates of single flax fibre diameter and the proportion of multiple fibre bundles in a sample; Grishanov (2006) produced software to assist this process. Diameter distributions are 'deconvoluted' and

the output is an estimate of the numbers of single (ultimate) fibres and their mean diameter along with estimates of the number of fibres made up of two, three, four and five or more ultimate fibres. Although diameter values are output as microns, values are on a 'relative' diameter scale as fibre, and fibre bundle, cross-sections deviate significantly from circularity.

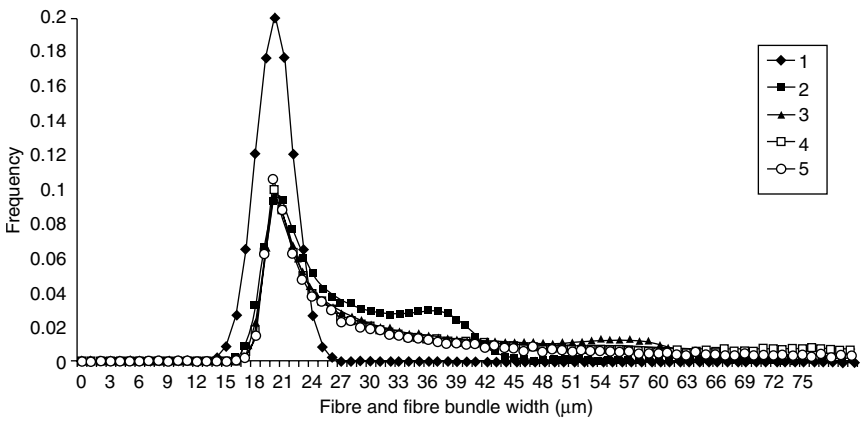
Workers at De Montfort University have led the way in using this approach for determining the effectiveness of the retting process (Harwood *et al.*, 2008a, 2008b; Horne *et al.*, 2008, 2010). Application of the deconvolution software to diameter distributions enables comparison of different fibre samples, irrespective of the degree of retting. Deconvolution software can provide the following additional data:

- Estimates of percentages of single fibres present in a sample, and fibre bundles containing two to five fibres.
- The average deviation of deconvoluted data from original data.
- The mean and standard deviation of ultimate fibre diameter.

Figure 12.8 shows the theoretical distributions of diameter for average fibre diameter of 20μ and standard deviation $\sigma = 2\mu$ for single fibres and fibre bundles consisting of two to five fibres. The distributions are labelled (i)–(v), respectively.

Airflow method for measuring fibre fineness

The ease with which a fluid will flow through a porous mass is dependent upon the surface area of the pores; the higher the surface area per unit volume, the greater is the resistance to fluid flow. The research which led to the



12.8 Theoretical distribution of fibre and fibre bundle width. (1) single fibre and (2–5) two to five fibres in the bundle (Grishanov *et al.*, 2006).

adoption of airflow methods for the textile industries was laid down in the late 1930s and 1940s with the first Micronaire instrument produced in 1947. Considerable evolution followed in the next decade.

It was shown that the flow of air through a standard plug of fibres is proportional to the difference in pressure across the plug (ΔP) and the diameter of the spaces between the fibres squared (d^2):

$$\text{flow} = K \cdot \Delta P \cdot d^2$$

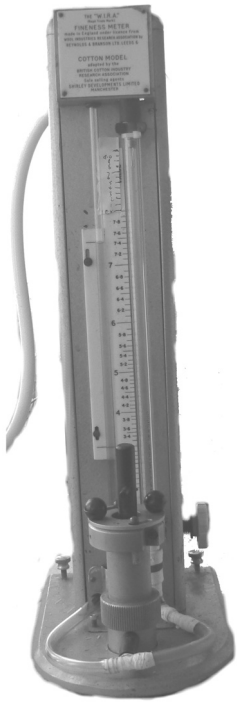
Using two principles – that at a given pressure difference the airflow through a uniformly distributed mass of fibres is determined by the total surface area of the fibres, and fine fibres have higher specific surface areas (surface area per unit mass) than coarse fibres – two instrument types evolved (Anderson, 1954). The first type of instrument relates the pressure drop at a standard rate of air flow to d^2 ($d^2 \propto \Delta P^{-1}$). The second instrument using a constant air pressure applied to the fibre mass determines the flow of air through the sample: K is a constant so we can either relate d^2 to the flow (by keeping ΔP constant) or to ΔP (by keeping the flow constant) or to the drop in pressure (keeping the applied pressure constant). $d^2 \propto$ flow. All modern instruments are based upon one of these scenarios.

The airflow concept was adapted to develop instruments for classifying wool and cotton fibre fineness and they are the standard quality control methods for assessing the fineness of these fibres (Fig. 12.9). Results for wool are expressed as the mean diameter in microns and for cotton as the mean fibre linear density in micrograms per inch (micronaire value). The advantages of airflow systems are their wide acceptance by industry, the low cost of equipment and ease of operation. However, the data are limited in their quality, the equipment available is generally specific to either wool or cotton and results are sensitive to sample preparation. Use of airflow methods with other fibres is commonplace but only as a guide to relative fineness (Faughey *et al.*, 2000; Sinha, 1970; Sinha and Bandyopadhyay, 1968). One of the ASTM standards, for measurement of flax fibre fineness, is based on the airflow principle (ASTM D7025-04a).

Operation

A fibre sample of known, standard, weight is compressed into a cylinder of a known, standard, volume and exposed to an air current at a known pressure. The rate of airflow through the fibre mass is measured using a rotameter which is calibrated to correlate with fibre fineness (using standard reference fibres whose fineness has been measured by a direct method), e.g. microns for wool, micrograms per inch for cotton.

The surface area of a fibre (length × circumference) is proportional to its diameter but for a given weight of sample the number of fibres will increase



12.9 WIRA wool fibre fineness meter. (WIRA Instrumentation Ltd, Bradford, UK.)

with fibre fineness so that the specific surface area is inversely proportional to fibre diameter. The fibre diameter will therefore determine the ratio of airflow to differential pressure. For fibres of approximately circular cross-section and constant overall density such as unmedullated wool (BS 3183:1968), the estimate of fineness obtained by the airflow method shows a good correspondence to the average fibre diameter as determined by projection microscopy.

The same method can be used for measuring cotton fineness, and is usually carried out on raw cotton opened and blended using a laboratory blender or Shirley analyser. However, results will be affected by the maturity of the cotton fibres as well as their fineness. For this reason the results are expressed in arbitrary Micronaire units (Micronaire is the trade mark of the Sheffield Corporation) (BS 3181-1:1987).

12.4.3 Fibre length and length distribution

The length of natural fibres varies considerably, even in samples of the same provenance, whether animal or plant. The fibre length is the second most important property of a fibre, fineness being the first. Wool is a relatively

long fibre and fibre length will vary between different breeds of sheep; fine wool sheep breeds (e.g. Merino and Charollais) typically have wool of 4–10 cm whereas long-wool breeds (e.g. Lincoln and Cotswold) have wool up to 30 cm long. Cotton is a much shorter fibre with Sea Island cotton fibres around 50 mm in length, whereas most are much shorter and some Indian cotton may be less than 12 mm in length.

The importance of fibre length lies in its processing, especially spinning. Generally the longer a fibre, the easier it is to process. However, fibre processing machines are designed to operate on a narrow range of staple lengths, and, even within a particular range of lengths, some machine adjustments must be made to optimise the process for the specific material being processed. Therefore it is important that the raw material should not show much variance from the established standard length.

This variation in fibre length of the raw material also needs to be controlled during the combing processes where short fibre is combed out (combing waste). The quantity of short fibre present will impact on the waste produced and hence on the economics of the manufacturing. Longer fibre also assists in the cohesion of rovings and slivers as longer fibres will overlap more for any given level of twist and, consequently, less twist is needed to produce satisfactory slivers from long fibre.

With long fibre, there will be fewer fibre ends in a given length of yarn and yarn evenness improves with increasing fibre length. A longer fibre will produce a higher strength yarn for the same level of twist. Fibre length and fineness are quite strongly correlated in the cases of cotton and wool: for wool, the longer the fibre the coarser the wool; for cotton the opposite is true as longer cotton fibres are generally finer than short cottons. Fibre length for wool and cotton is usually expressed as staple length: in simple terms the staple length of wool is the average overall length of the fibres in their naturally crimped condition. For cotton the staple length is between the mean length and the maximum length.

Fibre diagram machine

The fundamental principle of this machine is that it produces a fibre length diagram which is similar to that obtained from a traditional 'hand draw', when a fibre sample is hand laid on to a velvet board so that the length distribution can be observed. A fibre beard is produced in which all the ends of the fibres are aligned and the beard is then passed between two parallel plates that act as a capacitor. The change in capacitance is used to calculate the number of fibres present as a function of length, the distribution of fibre lengths is reported with the standard deviation and CV%. The maximum fibre length that can be detected using this type of instrument is approximately 300 mm.

Results are presented as Hauteur and Barbe fibre lengths. Hauteur (H) is the mean fibre length biased by the cross-section (linear density) of the

fibres; Barbe (B) is the mean length biased by fibre weight. Hauteur and Barbe are both measured in mm and have the relationship:

$$B = H\left(1+\left(\frac{CVH}{100}\right)^2\right)$$

where CVH is the coefficient of variation of Hauteur in %.

BS 5182:1975 describes the standard test for the measurement of wool fibre length.

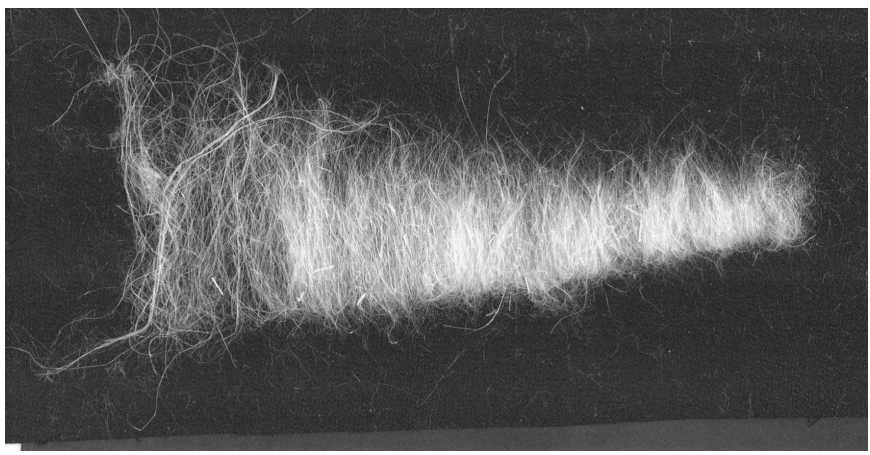
Fibre length distribution tester

Testing is carried out on a beard of fibre produced by drawing a comb over a sample of the fibre. A typical beard of flax fibres removed from the comb after testing is shown in Fig. 12.10.

The beard is drawn over an illuminated sensor where the changes in optical density enable the fibre length to be calculated. The length distribution data are generated at intervals defined by the operator. Length measurements determined using a fibre length distribution tester are limited to fibres up to 80 mm long.

12.4.4 Fibre strength

Because of the variability found in natural fibres, testing the strength of these fibres is time consuming and problematic. The strength of individual fibres shows a high degree of variability and for this reason a large number



12.10 A beard of flax fibres.

of fibres have to be measured to ensure statistical reliability. It is difficult to handle and mount single fibres in the jaws of a strength testing machine, especially if fibres are particularly fine. For this reason tests are often carried out on fibre bundles.

To measure single fibre strength a universal tensile tester is often used. Universal tensile testing equipment is widely available from many testing machinery suppliers, for example Uster, SDL Atlas, Instron. The principle is very simple: individual fibres are clamped vertically between two jaws and extended at constant rate of elongation. The applied force (stress) is measured continuously as the elongation (strain) increases; a complete stress strain diagram may be produced, alternatively the maximum force required to break the fibre may be displayed along with the elongation at maximum stress and/or at the breaking point. The strength of fibres is normally expressed as 'tenacity', i.e. the maximum force (Newtons) required to break the fibre divided by the linear density (tex) at the point of rupture.

There are instruments available that are specifically designed for single fibre strength measurement which are particularly useful for some fibre types (WIRA single fibre strength tester, the Lenzing Vibrodyn, Textechno Fafegraph HR, SDLATLAS Tinius Olsen H1KS). British standards BS EN ISO 5079:1996 and BS 4029:1978 describe the determination of breaking force and elongation at break of individual fibres, and tensile elastic recovery, respectively. The ASTM has published a standard test method for evaluation of the tensile properties of single textile fibres (ASTM D3822).

The testing of bundles of fibres takes less time and involves fewer test samples than testing individual fibres. The use of manual instruments requires training and experience to produce valid results but bundle strength has assumed greater importance than single fibre strength tests. The operating principle is to determine the breaking force required to rupture a carefully prepared thin ribbon of fibres which are clamped at very short gauge length; the weight of the fibres in the ribbon is determined and, so long as the fibre length is known, the tenacity of the fibres can be calculated (detailed instructions are always provided in the operating manuals). Careful sampling and multiple tests are needed to ensure that the results are representative of the whole batch of fibre. The 'Stelometer' (derived from STrength and ELOngation METER) is the instrument most commonly used for bundle strength testing of natural fibres and is available from a wide range of manufacturers. Semi-automated methods are commonly used and readily available, particularly for cotton testing; the operative is required to select and prepare the samples, usually with the aid of a 'fibrosampler' or other combing device to prepare a beard of fibres which is then inserted for automated testing (the USTER HVI System).

12.4.5 Fibre colour

Fibre colour will vary between different fibres and will be dependent on the processing to which it has been subjected. Colour can be measured instrumentally using a reflectance spectrophotometer. The technique requires controlled conditions of illumination, sample size and background. The principles of reflectance spectrometry are well established (McDonald and Rigg, 1980). As fibre samples exhibit variations in texture and colour, measurement using reflectance spectrophotometry requires careful sampling:

- Ensure samples are taken from different parts of the material and that duplicate measurements are taken in order to obtain a mean reflection spectrum.
- Ensure the sample is sufficiently thick to be opaque, thus preventing light transmitting through the sample and reflecting back from the sample holder.
- Rotate the sample 90° between measurements and take a mean value of 4 measurements to minimise any direction effects caused by the texture of the fibre sample.

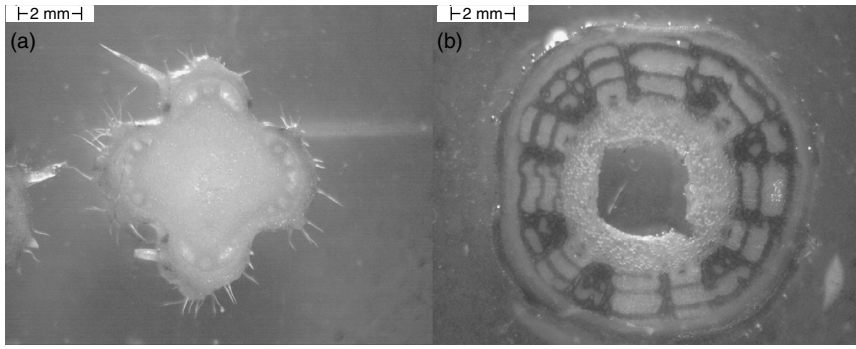
By far the most common system used for colour measurement is that developed by the CIE (Commission Internationale de l'Eclairage). The colour of a fibre sample is expressed in terms of three-dimensional colour coordinates, represented by the CIE L*a*b* colour space system, by which all surface colours can be represented.

Assessments of 'Whiteness' and 'Yellowness' of textile materials are often used to monitor changes during processing. Caution is needed when using measurements calculated from reflectance data with natural fibres, particularly bast fibres, as calculated rankings are often significantly different from visual rankings.

12.4.6 Optical microscopy

Optical microscopy can provide a direct image of plant structure and in conjunction with staining methods can be used to study the arrangement of fibres in the plant stem and the presence of non-cellulosic constituents. There are two types of optical microscopy relying on either reflected or transmitted light. Reflected light microscopy is used to study the surface of a sample. A sample can be simply placed under the microscope lens, or may be cut and polished into a flat surface to allow compositional and structural data to be collected.

Transmitted light microscopy requires samples that are optically transparent or translucent so that light can pass through them. Fibre samples



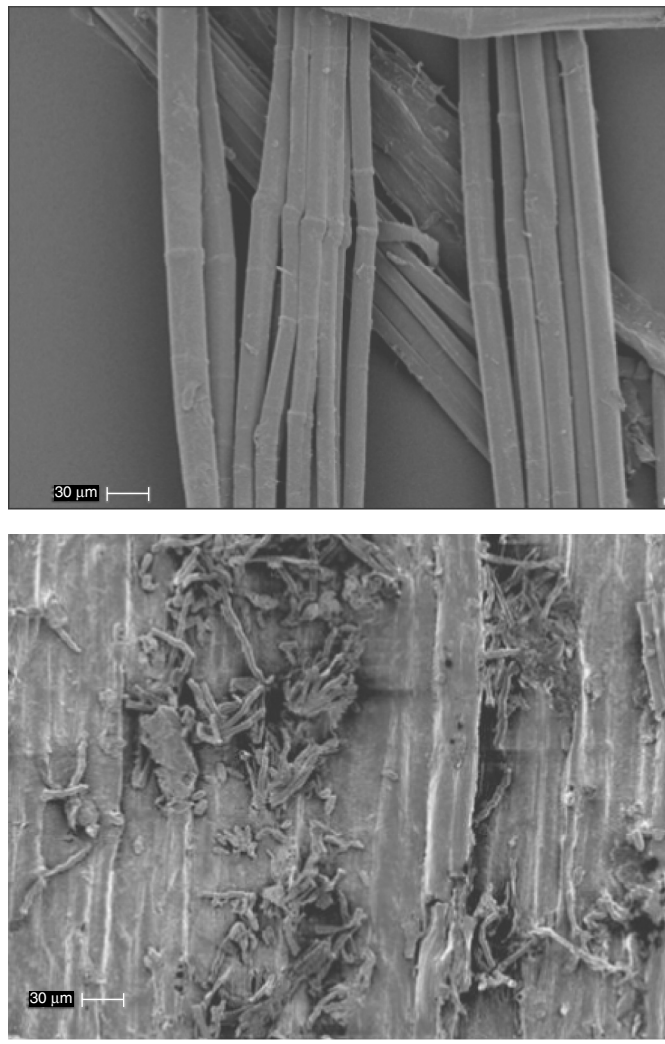
12.11 Cross-section of *Urtica dioica* stems (stained with phloroglucinol to highlight lignin: (a) upper stem; (b) lower stem.

are sufficiently thin for study without any preparation; however, other plant material will often require thin sections to be studied. Many techniques for preparing thin cross-sections are discussed in the literature (e.g. Greaves *et al.* 1995; Steedman 1960; Stoves 1957). To avoid distortion when cutting a cross-section a microtome can be used with the sample embedded in a supporting medium which may be a wax or polymer resin. A wide range of resins are available: the use of Spurr's resin has proved particularly successful for bast fibres, as it is an embedding medium that is low in viscosity and useful for plant materials which are difficult to infiltrate (Akin *et al*., 1996; Derue *et al*., 2002; Donaldson *et al*., 2001). Another resin that may be used for the same reasons is LR white (Fraser *et al*., 1982).

As with reflected light microscopy no direct chemical information is obtained from transmitted light microscopy. However, the use of specific staining techniques enables information on chemical composition to be gained. For example, the distribution of pectic substances in flax has been studied using a 0.02% aqueous solution of ruthenium red. Similar studies for lignin (Fig. 12.11) use acid phloroglucinol (Akin *et al*., 1996) and toluidine blue (Fraser *et al*., 1982). Such studies have shown that the concentration of lignin in the fibre bundles was significantly lower than in the xylem of the stem.

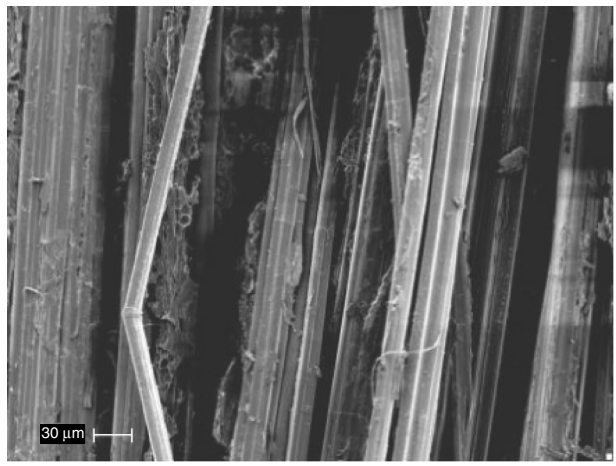
12.4.7 Electron microscopy

There are two forms of electron microscopy. Scanning electron spectroscopy (SEM) is a technique that has been available since the mid-1960s and is used for the study of surfaces. It can be used to study internal structures if a section through a sample is examined. Transmission electron microscopy (TEM) is an older technique dating from the 1930s, and is able to investigate internal structure. Although some elemental contrast can be obtained



12.12 SEM images: (a) chemically retted flax fibres; (b) outer surface of field retted flax straw.

from SEM arising from variations in atomic number throughout a material, it is not suitable for studying the distribution of lignin or pectic substances in plant structures. However, it can be used to study the surface of fibres and fibre bundles. Figure 12.12(a) shows an SEM image of chemically separated flax fibres and can be compared with Fig. 12.12(b) where there is clear evidence of fungal activity on the outer surface of flax straw which been retted in the field. Figure 12.13 shows the effect of retting on the inner surface of field retted flax straw. The separation of individual fibres as a result of field



12.13 SEM image showing inner surface of field retted flax straw.

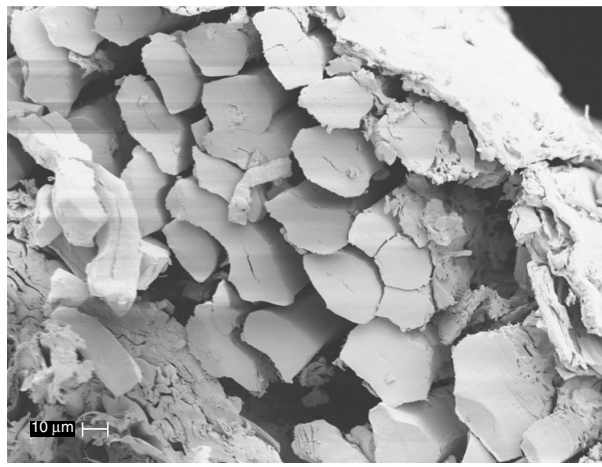
retting is well illustrated by the SEM image of a cross-section of the flax stem in Fig. 12.14. The technique can be used for a visual evaluation of differences between fibres and related materials.

Modern SEM instruments often include EDS (energy dispersive spectroscopy) analysis. SEM-EDS allows elemental analysis of the surface and immediate sub-surface of materials. It is particularly useful for metal analyses but can, theoretically, be used for all elements below sodium in the periodic table.

TEM is used for the study of internal structures and sample preparation is important as ultra-thin sections must be used. To permit good imaging, samples are stained with heavy metal salts, usually lead or uranium, in the same way as for other microscopic techniques. The very high resolution of the method enables detailed structural information to be obtained.

12.4.8 Cleanliness

Cleanliness affects the cost of fibre materials, low trash content materials normally commanding higher prices than materials with higher trash contents. Although there are manual test methods for trash and other non-fibre for cellulosic fibres automated methods are usually used. Instruments are available from a number of manufacturers (e.g. Uster and SDA Atlas) which allow the easy determination of trash, dust, microfibre and some other fibre contaminants. Dust is an important contaminant particularly with bast fibres as it can cause environmental health and processing problems in spinning and knitting mills. Test systems can also be used



12.14 SEM image showing fibre separation in field retted flax straw.

Table 12.2 Target specification for flax fibre on the cotton system

Property	Target specification
Fibre length	Mean length 25–30 mm
Short fibre	Not more than 15%
Strength	Not less than 28 g tex^{-1}
Trash	Total trash content less than 10%
Fibre fineness	Micronaire value less than 7
Colour grade	The whiter the better

to determine the 'cleanability' of the materials, a major consideration for the processing of fibres. The Sussen MDTA 3 and Uster instrument range were developed for cotton testing but are used for a range of other 'fine' fibre types. Output data includes the quantity of useful fibre and trash, dust, microfibre and nep content in fibre samples. The data are obtained by separation of the components by mechanical and airflow systems. Useful fibre is collected as a partially aligned sliver which can then be used for small-scale spinning trials.

As an example, MDTA data were used in the evaluation of different flax accessions grown for short fibre to be used in high value textile end uses (Harwood *et al.*, 2008a; Texflax, 2001). The aim of the work was to progressively select the 'best' accessions through evaluation of fibre obtained from straw from replicated trial crops harvested over three growing seasons. The selection criteria were initially based on the optimal properties for spinning on the cotton system (Table 12.2).

Five flax accessions were finally selected as having the optimum properties for spinning on the cotton system and were successfully blended with cotton to produce commercial 26 Nm (i.e. 38 tex) yarns with flax content up to 50% (Harwood *et al.*, 2008a, 2008b; Horne *et al.*, 2010).

For protein fibres the contaminants in raw materials are primarily grease and adhering soil which are effectively removed in raw fibre scouring prior to any further processing. Relatively simple laboratory test methods simulate commercial scouring processes and allow the clean fibre yield to be determined with supplementary standard tests for monitoring the quality of scouring processes (Wool Testing Authority, 2011).

12.4.9 Fibre length, strength, elongation and micronaire (HVI)

The Uster HVI (high volume instrument) provides data relating to average fibre length, percentage of short fibres, average fibre tenacity, average elongation at break and fibre fineness (micronaire value). The instrument was developed for cotton and is the standard measurement system used by the international cotton trade. The system can be used very effectively for cottonised flax and other fine bast fibres but results obtained will be 'relative' rather than absolute values (Harwood *et al.*, 2008a; Horne *et al.*, 2010).

12.4.10 Sensory factors

Following evaluation of the quality and performance characteristics of natural fibres it follows that their effectiveness in end-use products must be assessed. Whilst the uses of natural fibres encompass a hugely diverse range such as automotive, medical to fashion and clothing, it is worth mentioning here that the promotion of natural fibres in clothing has been addressed in studies of their comfort and physiological influence. Kawabata was a pioneer in the objective evaluation of fabric composed of both man-made and natural fibres. Fabric handle is clearly a subjective assessment, based on an individual perception of comfort values such as smoothness, stiffness, softness. Kawabata recognised that the stimuli causing a psychological response of handle are determined by the physical and mechanical properties of the fabric and developed the KES-F system whereby both subjective and objective measurements are correlated (Bishop, 1996; Kawabata and Niwa, 1989; Postle and Dhingra, 1989). Later research carried out by Zimniewska *et al.* (2004) investigated the physiological aspects of natural vs. man-made fibres. A review of the ultraviolet blocking properties of natural fibres is reviewed informatively in the *Handbook of Natural Fibres*, volume 2.

12.5 Chemical properties

Chemical analysis of bast fibres using traditional 'wet' methods are mostly based on work undertaken on flax by the Linen Industries Research Association (LIRA) and, later, the Lambeg Industrial Research Association, from 1919 until it closed in 1993. A special collection of LIRA publications can be viewed at the Irish Linen Centre at Lisburn museum. Chemical testing of flax and hemp is undertaken by CELC (Confédération Européenne du Lin et du Chanvre), and by members of the European Cooperative Research Network on Flax and other Bast Plants. An excellent review of the chemical testing of bast fibres is given in Chapter 3 of this volume. A comprehensive review of all chemical methods of testing of natural fibres is clearly beyond the scope of this chapter. However, key sources on chemical testing, analysis and properties are listed at the end of this chapter.

12.5.1 Chemical extraction of bast fibre

In order to evaluate various quality parameters of bast fibres it is often necessary to extract these fibres from the plant as cleanly as possible. Many methods have been employed by previous workers to release the fibres from bast plants. Pott (2003) suggested that treatment with water at 160–180°C would separate flax fibre from straw by removal of pectin, hemicellulose and lignin similar to the steam explosion process (Nebel, 1995). Van den Oever *et al.* (2003) developed an autoclave process for small-scale fibre extraction. Other researchers have investigated the use of chemicals such as sodium chlorite and sodium hydroxide (Cook, 1984) as well as pectolytic enzymes (Akin *et al.*, 2007; Militky *et al.*, 2000). Enzyme systems used in conjunction with emulsifiers and ultrasound have also been studied (Sirghie and Van Langenhove, 2004). However, on a laboratory scale the use of sodium hydroxide for fibre extraction has been found to produce the cleanest fibres and can be used for flax, hemp, ramie and nettle fibre (McCormick, 2006). The laboratory method described here can be applied to either decorticated fibre or plant stems where decortication has not been carried out or is impractical. The fundamental principle is that a known mass of dry sample is heated in sealed stainless steel beakers containing 0.5M NaOH and 0.025M EDTA solution at 130°C for a time dependent on the plant material. After neutralising with dilute acetic acid and rinsed with dilute ammonium hydroxide (1ml.880 ammonia/l) and finally rinsing with water, the sample is dried, reweighed and the percentage fibre content calculated.

12.5.2 Alkali solubility

The degree of retting is of general importance for bast fibres and is generally associated with removal of pectins and other non-cellulose materials.

Determination of alkali-soluble materials is a useful test when following removal from the parent plant bast fibres will contain varying amounts of non-cellulosic materials, typically pectins and hemi-celluloses. Some material is soluble in boiling water and further amounts are removed in normal wet processes. Alkali extraction can be used to gauge the effectiveness of a particular fibre processing method and when it is important to know how much soluble material is present. The method described would typically be used for bast fibres such as flax, hemp, ramie and nettle. Samples of fibre are boiled in 0.5M Na_2CO_3 solution for 2.5 hours and the loss of mass, compared to the loss when boiled in deionised water, is the 'alkali solubility'. The alkali solubility is calculated as follows:

$$\text{Alkali solubility } (\%) = \frac{100 \times (a - b)}{a} - \frac{100 \times (c - d)}{c}$$

where: a = initial mass of fibre, b = mass of fibre after extraction with 0.5M Na_2CO_3, c = initial mass fibre to be extracted with boiling deionised water, and d = final mass of deionised water specimen.

12.5.3 Cellulose content (cuprammonium fluidity)

Natural retting and the use of chemicals and enzymes for fibre extraction and processing are likely to cause some degradation to the cellulose molecules by hydrolysis or oxidation. This may be manifest in an undesirable decrease in fibre strength. It is a well known fact that pure cellulose dissolves in cuprammonium solution to give a viscous fluid and that any reduction in strength is accompanied by a nonlinear fall in the viscosity of the solution. If, however, the reciprocal of viscosity, which is termed 'cuprammonium fluidity', is used, this increases linearly with the percentage loss in strength over a wide range.

This method (BS 2610:1978) has been widely used for cotton and regenerated cellulosic fibres but is more difficult to use for other natural cellulosic fibres because of the high, and variable, concentrations of non-cellulosic components. However, the method has been adapted for flax by pre-treating to remove the non-cellulose materials (BS 3090:1978) and has been used on some other fibres.

12.5.4 Lignin content

Lignin is a complex 3D branched copolymer comprising, primarily, a mixture of three aromatic alcohols in a network of phenyl propene basic units. Lignin is a very diverse polymer and is hard to study which accounts for the large number of different analytical methods used. Lignin is responsible for

strength and rigidity in plants; it also controls diffusion processes and provides protection from microbial attack. The quantitative estimation of lignin is very dependent upon the method used; furthermore, the distribution of lignin within plant tissues is localised and is mainly associated with cell walls. The nature of the lignin present in plants also varies with growth stage; the degree of polymerisation and cross-linking tend increase with maturity (Blackburn, 2006). Lignin is important as it affects many aspects of fibre production, processing and properties. The situation is very complex. Lignin is important during plant growth, e.g. it influences the recovery of flax from lodging (Sharma, 1986), mature plants are more difficult to ret than crops harvested earlier and so lignin has a negative effect on fibre extraction. The presence of lignin has a negative effect on the quality and the commercial value of flax fibre (Sharma, 1986). As a result, investigation of the concentration, distribution and properties of lignin can be important at all stages of production and processing.

There are various methods for the determination of lignin. The choice of method will depend on whether quantitative or qualitative information is being sought and the type of plant material being investigated. Chemical methods include the Klason method (Theander and Westerland, 1986) and the acetyl bromide assay (Hatfield *et al*., 1999). Instrumental methods include infrared spectroscopy (Himmelsbach *et al*., 1999), nuclear magnetic spectroscopy (Ralph *et al*., 2001), gas chromatography (Sonada, 2001) and Raman spectroscopy. With over 40 different methods to be found in recent literature, it is easy to see why the International Lignin Institute (ILI) has included lignin characterisation and analysis in recent activities (ILI, 2011).

The distribution of lignin in plant tissues is most easily appreciated by simple staining of cross-sections. Figure 12.11 shows an example of the sort of variation that is found when sections are stained with phloroglucinol and clearly demonstrates that the concentration of lignin in plant tissues varies dramatically across and along plant stems. Micro spectroscopic methods, particularly infrared and Raman spectroscopy, have been successfully used to explore the distribution and nature of lignin and other components in plant cross-sections (e.g. Himmelsbach *et al*., 1999; Stewart *et al.*, 1995).

Klason and acetyl bromide determination of lignin

The Klason method is described here because it has been extensively used for measurement of lignin concentrations in bast fibres. It is based on the observation that carbohydrates can be hydrolysed by sulphuric acid to leave an acid-insoluble lignin residue (acid-insoluble lignin) which can then be determined gravimetrically. The procedure is carried out using a two-step hydrolysis of the carbohydrate components in which a sample of homogenised plant material is treated with a 72% solution of sulphuric acid at

room temperature, and then a secondary hydrolysis is performed by diluting the mixture and refluxing (Browning, 1967). Due to this harsh chemical treatment the solid residue remaining is unlikely to be representative of the lignin as it occurs naturally in the plant but attempts have been made to characterise Klason lignin (Hatfield and Fukushima, 2005; Reeves, 1993; Reeves and Galletti, 1993).

The main criticism of the Klason method is that the lignin residue can be contaminated by non-lignin components. Inclusion of a pre-extraction step using an isotropic mixture of ethanol and benzene will remove waxes and cutaneous matter (Monties, 1989). Other sources of contamination are degradation products from proteins and carbohydrates. A pre-extraction step with 'acid detergent' to remove proteins results in a lower estimate for lignin concentration – acid detergent lignin (ADL). Hatfield *et al.* (1994) compared the insoluble residues produced by the Klason lignin (KL) and the ADL methods and concluded that KL is a better estimate of the total lignin concentration in forages than ADL. Other workers have made similar observations (Fukushima and Hatfield, 2004; Jung *et al.*, 1999).

The quantitative determination of acid-insoluble lignin by either the KL or ADL method is commonly used in lignin chemistry but its use for bast fibres is questionable. Many researchers have found the results to be unreliable and not reproducible for samples with low lignin content (Day, 2005); flax fibre falls into this category. Love *et al.* (1994) found the KL content of flax fibre to be 3.2 ±0.6% which they believed overestimated the true lignin content of 0.9% as determined by nuclear magnetic resonance (NMR) spectroscopy.

The estimated concentrations of lignin in natural cellulosic fibres reported in the literature vary widely within each fibre and between fibres.

The acetyl bromide assay of lignin is widely used as a rapid and sensitive method for determining lignin concentration in small sample sizes (Johnson *et al.*, 1961) and the sensitivity of the method makes it suitable for analysing materials in which the lignin concentration is low, e.g. flax fibres. Acetyl bromide acetylates the free hydroxyl groups on the alkyl side chains and substitutes a bromine atom on the alpha carbon (Hatfield and Fukushima, 2005). The absorbance of the resulting solution is measured at 280 nm and the concentration of lignin assessed from a calibration curve using a suitably prepared lignin standard (Fukushima and Hatfield, 2001). Results from acetyl bromide assays are higher than obtained from the Klason or the ADL methods.

The wood pulp industry has a well-established method for measuring the bleaching required for wood pulp. For pulp bleaching the severity needed is directly related to the lignin content of the pulp. The method provides a 'Kappa' number which is approximately proportional to the lignin content. Kappa number is determined by ISO 302:2004 and is based on the

back-titration of an excess of potassium permanganate used to bleach pulped material; the results are expressed as the Kappa number in the range of 1–100. Kappa number has been used for investigating lignin concentrations in bast plant materials used in papermaking (e.g. Danielewicz and Surma-Slusarska, 2010) and for bast fibres Müssig and Fischer (2011) have used the Kappa number method extensively for their work on bast fibres.

A useful summary table which illustrates the lignin contents measured for a wide range of natural cellulosic fibres has been produced by Müssig *et al*. (2010). No one method is ideal for all cases, and the limitations of each method have to be borne in mind when results are interpreted. A succinct review of methods for chemical analysis is included in the work of Blackburn (2006).

12.6 Instrumental methods

Standard laboratory instrumental methods are used for the determination of a host of fibre characteristics, including study of the chemical composition of fibres, their surface morphology, thermal and flammability behaviour, amongst many others. Such methods enable fibre quality and end use behaviour to be assessed. This section provides examples of the methods used by various workers and serves as a source of information for further reading.

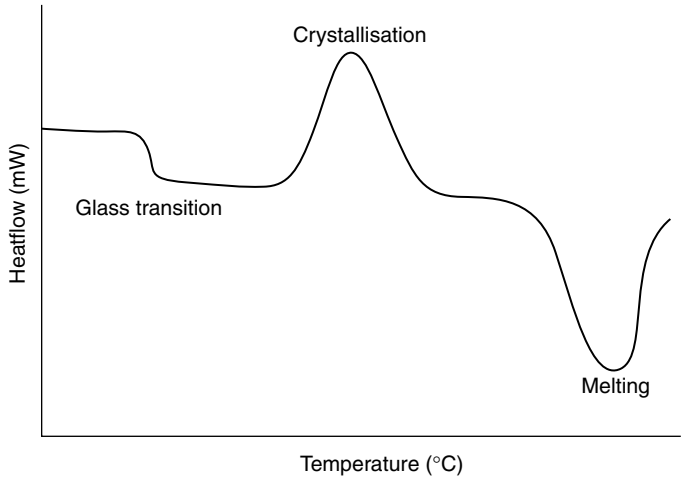
12.6.1 Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) is a method used to study the thermal transitions that occur in materials as they are heated. The principle is based on measuring the energy required to steadily heat a sample at a steady rate (or emitted during cooling at a steady rate) relative to a reference material. A sample of the material under study is placed in a cell and an empty cell serves as a reference. As each cell is heated, the difference in heat input for the two cells is measured as the temperature increases (or the difference in heat emitted during cooling is measured). In this way the heat capacity of the material can be calculated:

$$\text{Heat flow} = \frac{q}{t}$$

$$\text{Heating rate} = \frac{\Delta T}{t}$$

$$\text{Heat capacity} = \frac{(q/t)}{(\Delta T/t)} = \frac{q}{\Delta T} = Cp$$



12.15 Features of a DSC curve.

Fibres are physically complex and contain both crystalline and amorphous regions. Heating such a sample will cause a glass transition to occur in the amorphous region which is the reversible transition from the brittle state into a molten state. The temperature at which this occurs (Tg, the glass transition temperature) can be observed from the DSC curve (Fig. 12.15).

When the temperature rises to above the glass transition temperature, a polymer will become more mobile until it reaches the optimal temperature for crystallisation to take place. The molecules will arrange themselves in an extremely ordered fashion, and the sample will give off heat. This is observed as a reduction in heat flow. The area under the peak represents the latent heat of crystallisation. Application of more heat will cause the crystals to fall apart and the material to melt.

DSC is therefore a method whereby significant information relating to the crystalline and amorphous components of a polymer can be gained.

There are many examples of the use of DSC in the study of natural fibres. DSC normally forms part of a suite of analytical methods and has proved especially useful for studying natural fibre composites. Again there are many examples of such work but the work of Sgriccia and Hawley (2007) where DSC is in the characterisation of such composites is typical. Other illustrations are Islam *et al*. (2010) and Baiardom *et al.* (2004). Moran *et al*. (2008) utilised the technique in the extraction of cellulose from sisal fibre for use as a nanofiller for biodegradable matrices.

12.6.2 Thermal gravimetric analysis (TGA)

For the evaluation of the thermal and flammability behaviour of natural fibres and products made therefrom, a cone calorimeter may be used.

This enables testing of small samples of various materials in the condensed phase to be studied, Ignition time, mass loss rate, combustion products, heat release rate and other parameters associated with burning properties can be determined. This method has been widely used for example in the study of such properties of flax/polypropylene composites, e.g. Schartel *et al.* (2003); Manfredi *et al.* (2006). Many workers have found thermogravimetric analysis (TGA) a useful tool for the study of thermal degradation behaviour of natural fibres. The principle of operation of the technique is the measurement of mass change with temperature. For example compositional and structural information, as well as thermal degradation behaviour, of chemically treated hemp cellulose was studied by Ouajai and Shanks (2005). Titok *et al.* (2006) describe a thermogravimetric analysis of flax fibre bundles. Another example of how TGA may be used is a study of the pyrolysis products of flame retardant cotton fabrics carried out by Zhu *et al.* (2004). Majdanac and Teodorovic (1987) reported on the kinetics of the thermal decomposition of cotton cellulose. TGA enabled the thermal degradation and fire resistance of natural fibre composites to be investigated (Manfredi *et al.*, 2006). The cationic modification and dyeing of ramie fibre is another area where TGA has been employed (Zhao-Tie *et al.*, 2007).

12.6.3 Spectroscopic methods

Spectroscopy in various forms has become a routine tool for investigating the structures and properties of natural fibres. The general principles and techniques are described in a wide range of sources. All forms of spectroscopy are based on the principle of measuring the quantity of energy absorbed or emitted when a sample is exposed to electromagnetic radiation or high-energy particles. Common forms of spectroscopy include the usual ultraviolet (UV), visible (Vis), infrared (IR), Raman and fluorescence methods and their Fourier transform (FT) variants; flame ionisation methods (for metal analyses) including atomic adsorption (AA), atomic emission (AE), atomic fluorescence (AF) and later variants such as plasma emission (PE); and onwards into NMR, mass spectrometry (MS), matrix-assisted laser-de-sorption ionization–time of flight (MALDI-TOF) MS and X-ray spectroscopy.

The literature available on the use of spectroscopic methods is very extensive and cannot possibly be adequately reviewed here. For this reason the following is a very brief selection which illustrates some of the applications of these methods to natural fibres during recent years, and will provide a starting point for further literature research.

Work by Lang *et al.* (1986), Howell and Davies (1991), Edwards *et al.* (1997) and Garside and Wyeth (2003) are examples of investigations into the identification of natural fibres using IR transmission and reflectance techniques, FT-IR and Raman spectroscopy for the identification of a wide

range of natural fibres. Meijer *et al*. (1995) used visible spectroscopy for the determination of pectin contents of flax.

A paper by Akin *et al*. (1996) describes the analysis of fibre and core fractions of flax by gas–liquid chromatographic methods, ^{13}C CPMAS NMR spectrometry, electron microscopy and UV absorption microspectrophotometry. Results give an insight into the structure and composition of the cell walls in relation to quality and end uses. Mwaikambo and Ansell (1999, 2002) used wide angle X-ray diffraction, DSC, FT-IR and SEM methods to investigate the chemical and surface morphology of chemically treated hemp, sisal, jute and kapok fibres for improvement of fibre to resin bonding in natural composite materials.

It is known that the degree and method of retting flax stems will have a direct effect on the fibre quality. Morrison *et al*. (2000) evaluated the composition of dew-retted and water-retted flax fibres by chemical and mass spectral analyses to determine their chemical differences. Phenolics, waxes, cutin and carbohydrates were determined by gas liquid chromatography. Pyrolysis mass spectrometric analysis differentiated water- and dew-retted fibres. A later study by Himmelsbach *et al*. (2002) investigated changes in a range of chemical components of flax during retting using FT-IR microspectroscopic analysis of cross-sections. Other workers (Mooney *et al*., 2001) used High Performance Anion Exchange Chromatography (HPAEC) and MALDI-TOF mass spectrometry to study the sugars and oligomers extracted from flax during chemical and enzymatic retting processes.

Fibre surface properties and their interface with resin matrices are clearly important for the natural fibres composites industry. Modifying the fibre surface can improve hydrophilicity, wetting and adhesion properties. Singh *et al*. (2000) used diffuse reflectance FT-IR and other techniques to study the surfaces of modified sisal.

Sharma and Reinard (2004) explored the potential of visible and near IR spectroscopy as a tool for the rapid quality assessment of flax fibres during the spinning process. They successfully focused on assessment of the quality of input fibre and changes in fineness at all stages from the doubling (blending) through to the roving stage. It was concluded that assessment using visible and near IR methods could allow fineness and spinning quality of flax to be predicted.

12.7 Future trends

The nature of international trade and the trend towards high levels of process automation are causing fibre quality to be described by results of objective testing methods. The grading of quality against specifications has been well established for many years for cotton and wool fibres but for most other fibres the production volume has been too low and qualities too varied for grading systems to be established.

The move away from qualities being defined relative to reference samples is relatively slow and very dependent upon the fibre type and the end uses. The increased use of natural fibres in composites is an example of an end use which is influencing moves towards improved specifications; as the applications become more refined, raw material input to meet performance or processing criteria is becoming more commonplace.

The USA has led the trend for standardisation of test methods with the work on flax and there is a growing trend for suppliers to provide test data, albeit restricted in diversity at present, alongside physical samples. It can be expected that measurements of fibre parameters such as fineness, length, strength, trash content, colour and useful fibre will be expected by consumers of a wider range of fibres in the near future.

In the research areas, many of the established methods for evaluating fibre characteristics and properties were very labour-intensive. For the major fibres, cotton and wool-type fibres, many test methods have become highly automated. For the majority of natural cellulosic fibres there has been little advance in automating methods for the evaluation of physical properties except for fibre diameter and length.

12.8 Sources of further information and advice

For further information and advice the reader is directed to the following publications:

Advances in Wool Technology (2009). Ed. N. A. G. Johnson and I. M. Russell. Cambridge: Woodhead Publishing.

Bast and Other Plant Fibres (2005). R. R. Franck. Cambridge: Woodhead Publishing.

Handbook of Natural Fibres. Vol. 1: *Types, Properties and Factors Affecting Breeding and Cultivation* (2012). Ed. R. Kozłowski. Cambridge: Woodhead Publishing.

Handbook of Natural Fibres. Vol. 2: *Processing and Applications* (2012). Ed. R. Kozłowski. Cambridge: Woodhead Publishing.

Industrial Applications of Natural Fibres, Structure, Properties and Technical Applications (2010). Ed. Jorg Mussig. Chichester: John Wiley.

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ASTM (2004), ASTM D-7025-04 *Standard Test Method for Assessing Clean Flax Fibre Fineness*. West Conshohocken, PA: ASTM.

ASTM (2005), ASTM D-7076-05 *Standard Test Method for the Measurement of Shives in Retted Flax*. West Conshohocken, PA: ASTM.

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12.10 Appendix: abbreviations

AA	atomic absorption spectroscopy
AE	atomic emission spectroscopy
AF	atomic fluorescence spectroscopy
ASTM	American Society for Testing and Materials
BSI	British Standards Institution
CIE	Commission Internationale de l'Eclairage
CPMAS	cross polarization/magic angle spinning
DIN	German Institute for Standards
DSC	differential scanning calorimetry
EDS	energy dispersive spectroscopy
EN	European standards
FT	Fourier transform
FT-IR	Fourier transform infrared spectroscopy
HPAEC	high performance anion exchange chromatography
HVI	high volume instrument
IR	infrared spectroscopy
ISO	International Organization for Standardization
LIRA	Linen Industries Research Association
MALDI-TOF	matrix-assisted laser-de-sorption ionization–time of flight
MDTA	microfibre dust and trash analysis
MS	mass spectroscopy
NMR	nuclear magnetic resonance spectroscopy
OFDA	optical fibre diameter analyser
SEM	scanning electron microscopy
TEM	transmission electron microscopy
TGA	thermal gravimetric analysis
UV	ultraviolet spectroscopy
VIS	visible spectroscopy

13

Developments in fibrous flax breeding and cultivation

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Abstract: This chapter looks at methods of flax and linseed breeding (*Linum usitatissimum* L.), beginning with a survey of flax growing in Europe and the rest of the world. The chapter goes on to describe the ongoing work with flax and linseed genetic resources before discussing the International Flax Database and finally conventional and unconventional methods of breeding.

Key words: *Linum usitatissimum* L., flax, linseed, breeding methods, conventional, unconventional, anther culture, haploid, diploid plants, transformation, genetically modified organism (GMO).

13.1 Introduction

During the last few years, the amount of flax grown in Europe and the rest of the world has decreased dramatically. The following sections discuss the reasons for this decrease, and the current situation in flax producing areas is outlined.

13.1.1 Flax growing in Europe and in the rest of the world

There has been a decrease in the number of flax producing areas all over the world in recent years. In traditional Western European countries like France, Belgium and the Netherlands, flax areas have been decreasing since 2006 (*Situační a Výhledová Zpráva*, 2009). In Egypt, however, there has been a small increase in the number of flax production areas. The first flax reports from China in 2006 confirmed a flax area of approximately 130 000 ha, of which production from only 78 000 ha was processed.

The world flax areas cover approximately 400 000 ha, of which about 25% are located in France, Belgium and the Netherlands. Fibre production from Western European countries represents approximately 60% of the world market. The available statistical data show that worldwide fibre production is mainly concentrated in Western European countries between the rivers Escaut and Seina (*Situační a Výhledová Zpráva*, 2009).

At the beginning of the 1990s the flax areas in EU countries reached 50–80 000 ha, but decreased massively between 1991 and 1992 to 44 000 ha during the peak of the flax crisis. Thanks to an endowment from the EU, flax areas stabilized and interest in flax growing rose. Flax areas again increased and reached more than 100 000 ha in 1995. However, the main reason for this unexpected increase was speculative flax growing in non-traditional flax growing countries with the aim of receiving subsidies. That is to say, the financial support was provided to these countries without any view to following processes and utilization. Flax growing areas and their development during the last 10 years are presented in Table 13.1.

Since 2000, rules for providing subsidies have been adopted and many non-traditional flax growing countries (Great Britain, Spain, Portugal) lost interest in flax growing. For this reason the flax areas again decreased from almost 214 000 ha in 1999 to 87 000 in 2002. The admittance of new countries to the EU (Latvia, Lithuania, Poland, Czech Republic) meant that the flax areas again increased in 2004, but in the long term a lasting decrease in flax areas has been observed (*Situační a Výhledová Zpráva*, 2009).

13.2 Key issues of fibre flax breeding and cultivating

In this section, the basic sources and steps of breeding work are discussed, starting with European flax genetic resources followed by the breeding methods that are generally used.

13.2.1 Flax genetic resources – national inventories

Successful breeding work depends on the range of genetic diversity kept in the respective flax national collections (national inventories) of individual European countries followed by the breeding methods used. The first step to make an inventory of the European flax genepool was made at the ad hoc meeting of flax collection holders within the newly established Industrial Crops and Potato Network at the last European System of Cooperative Research Networks in Agriculture (ESCORENA) network by European Cooperative Programme for Crop Genetic Resources (ECP/GR) held in Prague, on 7–8 December 2001 (Maggioni *et al.*, 2001). The structure of the respective national inventories – Bulgaria, Czech Republic, France, Germany, Hungary, the Netherlands, Poland, Romania, Russia, Ukraine – as well as the methods of the work with flax genetic resources were presented (Maggioni *et al.*, 2001).

The stocktaking of European flax genetic resources in 2001 showed the following status. Bulgarian flax collection is kept in two institutions. In Sadovo 945 accessions were maintained in 2001. The evaluated 450 accessions showed that 23% are linseed, 29% flax and 46% of intermediate type. Landraces and old cultivars create 58%, advanced cultivars 15% and breeder's lines 14%. Except

Table 13.1 Extent of flax growing (areas in hectares) in EU countries

Country	1999	2000	2001	2002	2003	2004	2005	2006	2007	2008
France	49 129	53 680	66 561	66 772	74 439	78 281	81 843	76 278	74 500	67 688
Belgium	12 176	13 320	16 860	15 315	19 306	19 823	18 761	15 919	14 740	12 230
Netherlands	3 570	4 016	4 415	4 062	4 615	4 517	4 691	4 366	3 500	2 525
Total	**64 875**	**71 016**	**87 836**	**86 153**	**98 360**	**102 621**	**105 289**	**96 563**	**92 740**	**82 490**
Great Britain	15 000	12 089	4 860	177	1 976	1 820	21	0	0	0
Finland	863	1 067	365	202	97	67	57	0	0	0
Germany	569	402	200	200	224	180	38	30	51	42
Spain	126 226	13 895	457	55	0	0	0	0	0	0
Austria	336	450	132	171	142	110	133	129	0	0
Sweden	1 327	21	0	25	0	30	0	0	34	
Denmark	11	45	19	2	0	0	0		0	0
Italy			0	6	20	80	18		0	0
Ireland									0	0
Portugal	4 678	3 810	0	0						
Latvia						2 400	2 072	1 057	0	356
Lithuania						5 600	3 599	1 420	424	247
Poland						5 745	1 507	788	1 044	779
Czech Republic						5 499	4 311	2 736	824	156
Total EU	**213 885**	**102 183**	**93 869**	**86 991**	**100 819**	**124 152**	**117 043**	**102 723**	**95 117**	**84 070**

Source: DG VI – C4 European Commission, Vlas Berichten 8/02, 12/03, 22/03, 22/04, 23/05, Czech Flax Union.

for *L. usitatissimum*, there are 12 wild species in the collection: *L. altaicum*, *L. austriacum*, *L. bienne*, *L. flavum*, *L. grandiflorium*, *L.humile*, *L. perenne*, *L. punctatum*, *L. setaceum*, *L. strictum*, *L. trigynum* and *L. viscosum* (Shamov, 2001). In addition 283 accessions are maintained at the AgroBioInstitut (ABI) in Kostinbrod. Of these, 178 were received from IGR–Sadovo, 29 from other Bulgarian institutions, 71 from foreign institutions and 5 are own breeding materials. These accessions were originated from Europe, Asia, Africa and America. Only 6.4% are of Bulgarian origin. Cultivars represent 54% of the collection, landraces and primitive cultivars 27.5%; breeding lines 15%; wild forms 1%; genetic stocks 2.5% According to the plant type, fibre flax represented 31% of the collection; linseed 35%; combined type 32%; other types 2%. 259 accessions were described for 15 morphological traits, 5 biological and 4 yielding characters, according to International Union for the Protection of New Varieties of Plants (UPOV) (1991, 1995) and to the International Flax Database (IFDB) descriptors (Pavelek, 1994, 1995) and other descriptors for the species *L. usitatissimum* L. (Rykova *et al.*, 1987, 1989). Linseed and combined type accessions were also analysed with biochemical methods for oil and proteins content, Cd accumulation and fatty acid composition of oil according to standards of the International Standard Organization (ISO). Fibre quality evaluation of flax varieties was also made according to Bulgarian standards (Balabanova and Atanasov, 2001).

The Czech flax national collection has been managed since the 1960s by Agritec Ltd., Sumperk. It is one of the largest European collections, with 2011 accessions of flax, linseed, intermediate and wild types in 2001. Based on geographical representation, the main part of the collection is of European origin, followed by a number of accessions from Africa, the USA and Australia. The flax national collection is described according to the Descriptor List (Pavelek, 2001a) by 22 passport as well as 55 special descriptors covering morphological traits, biological and yielding characteristics (Pavelek, 2001b).

The French flax and linseed germplasm collection contains 1650 accessions with about 50 Institut National De La Recherche Agronomigue ((INRA)-lines. It is maintained by the INRA flax breeding laboratory. The genetic resources were collected from 42 different countries, but mainly from the USA (360 accessions), Argentina (327), Russia (192) and France (108). The collection is composed of 426 accessions of linseed, 273 of flax and 207 of intermediate *Linum*. Other genotypes seem not to have an agricultural interest but could be used as parents for breeding. Characterization is completed and impurities or heterogeneous material are detected. A catalogue of all the accessions, including their passport data is available on Internet at http://www.inra.fr/Internet/Produits/Lin/index.htm. About 800 genotypes have almost fully been described for 17 qualitative and 14 quantitative traits (Fouilloux *et al.*, 2001).

The German flax collection was previously kept in the two German genebanks (Federal Centre for Breeding Research on Cultivated Plants, Braunschweig, Germany (BAZ) Gene Bank/Braunschweig; Institute of Plant Genetics and

Crop Research, Gatersleben, Germany (IPK) Genebank/Gatersleben) in 2001. A total of 2304 accessions of *Linum* plant genetic resources were maintained as follows: BAZ: 621; IPK: 1683. Almost 95% of these accessions (2181) belong to *L. usitatissimum* as the only crop species of the genus. This species actually constitutes (with the exception of four as yet undetermined accessions) the entire BAZ collection, while in the IPK collection, 85 entries from at least 25 other species of the genus *Linum* are also maintained. With respect to the origin of the material, Russia/the former Soviet Union and Germany with 184 and 177 accessions, respectively, are the major donor countries, followed by Italy (87), Hungary (75), France (72), Iran (67) and Portugal (58). In total, 67 countries are listed as donors (BAZ: 32, IPK: 67). Regarding the continents, Europe with 1147 accessions is the origin of almost 50% of the entries, while Asia (179), America (154), Africa (82) and Australia (2) donated the second half, together with 745 accessions of unknown origin. In the active collection of the BAZ, modern varieties (72%) and breeding lines (21%) prevailed, whereas only few landraces (6%) were represented. In the IPK collection, modern varieties and breeding lines form a major part as well (total 44%). Less than 1% landraces and 5% wild material are present, while the sample status of 50% of the accessions is unknown. About equal amounts of oil (37%) and fibre (45%) types were maintained at Braunschweig, only 3% were classified as combination types. In Gatersleben, however, combination types dominate with 49%, followed by 27% fibre and 7% oil types; the usage of 17% of the entries is unknown. Concerning characterization and evaluation, the respective BAZ activities were focused on inflorescence, agronomic and seed trait. The evaluation activities for the genus *Linum* include 46 descriptors. In the IPK genebank, *Linum* accessions were also characterized for a majority of the IFDB descriptors, the respective data not yet being available in an electronic format (Dehmer *et al.*, 2001). Nowadays all accessions from BAZ have been moved to IPK which is now the only institution responsible for the whole German germplasm stock (Dehmer *et al.*, 2001).

The Hungarian National *Linum* Collection, which is over 45 years old, is part of the national collection and is composed of 409 accessions. 94% of the *Linum* collection belongs to the species *L. usitatissimum*. The analysis of *Linum usitatissimum* by subtaxa showed a wide range of variation. 80% of the collection derives from Hungary (40.3%), former Czech and Slovakia (15.9%), Germany (13.2%), Great Britain (3.2%), Romania (2.7%), France (2.4%) and Poland (2.2%). 71% of the collection is composed of advanced cultivars. Characterization and evaluation have already been carried out on 307 accessions, equivalent to 75% of the *Linum* collection (Simon, 2001).

The Dutch *Linum* collection was adopted by the Centre for Genetic Resources (CGN), the Netherlands in 1995, after a group of four private Dutch breeding companies encouraged the institute to take action for the proper conservation of this valuable collection. In 1996, an arrangement for cooperation in the field of maintenance and evaluation was established between the

breeding companies and CGN. The collection included 974 accessions in 2001 and is divided into fibre flax, linseed, intermediate flax and wild species.

A minimal descriptor list was developed based on the descriptors for *Linum* of the IFDB of the FAO European Cooperative Network on Flax. The present CGN descriptor list includes 16 descriptors that can be divided in two groups:

- 10 mandatory descriptors. These traits should be always recorded during the regeneration.
- 6 optional descriptors. These traits can be recorded when sufficient time is available or at the occurrence of diseases for which the character can be optimally scored in the field.

In the framework of a multidisciplinary fibre research programme, a core collection of fibre flax was developed. The core was generated over the period from 1998 to 2001 and the development was conducted after different stages, resulting in the final core collection of 84 accessions (Soest, 2001).

The Polish Flax Collection managed in former Institute of Natural Fibres (INF), nowadays Institute of Natural Fibres and Medicinal Plants (INF&MP) Poznan comprises 864 accessions: 48 accessions of wild species, 29 landraces or primitive cultivars, 588 advanced cultivars, 102 breeder's resources and 97 accessions of unknown status. The INF collection holds also unique Poland-originating accessions. All genotypes present at the INF genebank have been characterized as far as genetic and economic qualities are concerned. The flax collection is also described according to resistance to diseases, especially the most dangerous one – Fusarium wilt. The most important task is the improvement of the protection and preservation of flax and hemp genetic resources. The main aim of the INF genebank is to help breeders in their work by increasing availability of genetic resources for breeding and research, and by screening the core collection for properties important for breeders (Rutkowska-Krause, 2001).

The Romanian *Linum* national collection is represented by 3845 accessions, mainly of *L. usitatissimum* (98%), with another 20 species. Only 220 accessions are considered as duplicates. 2161 accessions are oil varieties, 1068 are fibre type, 71 are intermediate, and 545 unknown. The institutions holding flax collections in Romania are the Research Institute for Cereals and Industrial Crops of Fundulea (2880 accessions), the Suceava genebank (520), the Agricultural Research Station of Livada (420) and the Agricultural Research Station of Simnic (25). The National Genebank of Suceava has the responsibility to preserve '*ex situ*' the Romanian agrobiodiversity. About a third of the collection has Romanian origin, from which a small part is represented by old cultivars (Strajeru, 2001; Vasile, 2001).

The Russian flax national collection located in the N. I. Vavilov Research Institute of Plant Industry (VIR) in St Petersburg consists of 5521 accessions

and represents the biggest European collection. It covers a wide range of diversity. The collection includes all three flax types: fibre, oil, usually called linseed, and intermediate type, which can be used for both purposes. Some wild species are also included in the collection. The collection covers the whole area where flax is cultivated. More than half of the collection is represented by local folk bred varieties. Also several commercial varieties, lines and other breeding material are conserved. The main part of the collection is composed of accessions originating from the former Soviet Union Republics. The main donors of genetic material are Russia, Uzbekistan, Ukraine and Tajikistan. The majority of these accessions are landraces, which now can be found in this collection only. Practically all flax commercial varieties ever bred on the territory of the USSR are maintained. Breeding lines and donors of different agronomic characters are widely represented too. Apart from the former Soviet Union, flax genetic material from 58 foreign countries is included in the collection. Some countries, such as India and Ethiopia, are represented mainly by local accessions. For some other countries such as, for example, Germany and the USA, the collection is represented mainly by commercial varieties and breeding material. A genetic collection consisting of about 250 inbred lines and lines of lower generations are conserved at the VIR. These lines carry different morphological and agronomic characters. This collection includes different phenotypes of traits such as colour and shape of flowers and seeds, resistance to rust, duration of vegetative period, plant height and others. For many of them, genetic control of the character has been identified (Brutch, 2001).

The Ukrainian flax collection contained 1042 samples in 2001. The collection was only established after 1992, since all material necessary for carrying out scientific research and breeding work were previously obtained from the VIR. The collection, based in Glukhiv, is currently composed of samples from 45 countries, the largest amount from Russia (256); 117 from Sweden, 54 from the Netherlands, 75 from Czech Republic and Slovakia, 52 from Ukraine, 52 from the USA, 34 from Argentina, 30 from France, 23 from Poland, 23 from Hungary and 61 from Germany. So far, 870 accessions have been characterized and a number of samples have been singled out for their useful properties, such as 132 for fast-ripening, 154 for seed yield, 81 for straw yield, 76 for fibre content in the stalks, 42 for plant height, 114 for good spinning capacity of fibre, and 41 for resistance to lodging. All the materials have also been evaluated for their resistance to diseases, and five carriers of resistance to Fusarium wilt have been identified. Characterization of the collection is carried out and includes information about country of origin, originating institute, authors, botanical species, intra-specific taxon, life cycle, type of development, ploidy, value of the sample, availability, etc. Unfortunately, this work is still far from completion (Virovets *et al.*, 2001).

The Italian flax collection is maintained by CRA-Instituto Sperimentale per le Colture Industriali (CRA-ISCI). It comprises 380 accessions, mainly

originating from European countries. A database (MS Access) for both passport and descriptive data on the collection, according to the IFDB structure is available for internal use (Mandolino, 2006).

Part of the present Latvian flax collection has been built by repatriation of Latvian material from Russian and German institutes. The Latvian flax genetic resources are maintained by two institutes: the Latvian Genebank of Cultivated Plants and the Latgale Agricultural Science Centre. The former maintains 26 accessions from Latvian origin in both base and active collections (Grauda, 2006).

The Portugal flax collection maintained by Banco Português de Germoplasma Vegetal/Direcção Regional de Agricultura de Entre Douro e Minho (BPGV/DREM) consists of 147 accessions. Estação Agronómica Nacional (EAN Genebank) maintains 8 accessions. All accessions originate from Portugal (de Sousa, 2006).

The Slovakian flax genetic resources are stored at the Genebank of Slovak Republic, which is the part of the Research Institute of Plant Production in Piestany. It consists of 170 accessions, of which 145 accessions are both in base and active collections and are safety duplicated at RICP Praha-Ruzyne. The passport data of 136 accessions are computerized and ready to be submitted into the IFDB (Nožková, 2006).

The Lithuanian collection and storage of flax genetic resources was started in 1994. The collection of *Linum usitatissimum* L. in Lithuania consists of 922 accessions in the active collection of which 51 accessions are stored under long-term storage conditions. At the Field Crops Coordinating Centre in Lithuanian Institute of Agriculture in Akademija, Kėdainiai district, 51 accessions of flax are stored in long-term storage facilities. The active flax and linseed collection is stored and investigated at the Upytė Research Station of the Lithuanian Institute of Agriculture (Jankauskiene, 2006).

Nowadays the work with flax collections is in progress, the traditional methods of evaluation based on passport and special descriptors analysis are still replenished by molecular methods of characterization in order to rationalize the work with collections, to create core collections as well as to identify and to distinguish the respective accessions. Various DNA markers have been widely used for diversity analysis in plants, including random amplified (RAPD) molecular markers, inter-simple repeat (ISSR), amplified fragment length polymorphism (AFLP) and simple sequence repeat (SSR). All these methods have also been applied to study flax germplasm diversity (Cloutier *et al*., 2009; Diederichsen and Fu, 2006; Everaert *et al*., 2001; Fu, 2002, 2005; Fu *et al*., 2002a, 2002b; Vromans, 2006; Wiesnerova and Wiesner, 2004). One of the last developed molecular method is Inter retrotransposon amplified polymorphism (IRAP) being also often used for germplasm collections analysis (Antonius-Klemola *et al*., 2006; Kalendar and Schulman, 2006; Smýkal *et al*., 2008; Vukich *et al*., 2009).

13.2.2 International Flax Database (IFDB)

Conservation and maintenance of plant genetic resources (PGR) and biodiversity utilization have an international character and via submitted documents – 'Convention on biological diversity' (UNCED, 1992) and 'Global Plan of Action' (FAO 1996) – are coordinated by FAO and International Plant Genetic Resources Institute (IPGRI) (currently Bioversity International) in Rome. The contemporary strategy of PGR monitoring, study, conservation and utilizing is determined by these documents. The Global Plan of Action is focused on worldwide networks creation resulting in PGR protection, conservation and utilization. Via IPGRI the ECP/GR has been operating since 1980 and nearly all European countries are involved (Maggioni *et al.*, 2001). The activities on PGR are carried out through ten networks in the respective working groups in the framework of action VII (2004–2008) where also the creation of the international crop databases is considered to be one of the FAO and IPGRI networks' activities. These central crop databases are accessible via the European Internet Search Catalogue (EURISCO (European Plant Genetic Resources Search Catalogue with Passport Data on Ex Situ Collections Maintained in Europe)) (Maggioni *et al.*, 2001). The EURISCO web catalogue automatically receives data from the National Inventories (NI). It effectively provides access to all *ex situ* PGR information in Europe and thus facilitates locating and accessing PGR. EURISCO is hosted at and maintained by the IPGRI on behalf of the Secretariat of the ECP/GR. EURISCO and the list of FAO/IPGRI Multicrop Passport Descriptors (MCPDs) agreed for data exchange by the European database managers in 1996 (Lipman *et al.*, 1997) have also been used as a starting point for the IFDB development.

The system of flax germplasm evaluation, description and characterization is not uniform either in Europe or in the USA and Canada and differs according to the breeders' requests in individual countries. Regarding the different evaluation and characterization systems used in the individual European genebanks as well as overseas ones and in order to exploit the richness of the flax genetic diversity for breeding activities it is necessary to have a good, unique system of evaluation, description and characterization which should be uniform in European countries at least. Therefore the IFDB can be considered as an initial input for this aim (Pavelek, 1995).

The IFDB has been managed and coordinated by the Agritec Ltd. since 1993 in the framework of FAO ESCORENA Flax and other Bast Plants Network (FAO FOBPN) (Pavelek, 1995, 1997, 1998a, 1998b; Pavelek *et al.*, 2001) and then since 1999 in the framework of IPGRI Coordination Group Network for Sugar, Starch and Fibre Crops (CGN-SSFC), nowadays Sugar, Starch, Fibre Crops & Aromatic Plants Network (CGN SSFC&APN) at Bioversity International. It presently includes passport data of 8385 accessions of 11 collections from 10 countries in an ACCESS IFDB structure. From

an analysis of the delivered data it can be concluded that approximately 37% of the accessions are unique. It also revealed large differences in the percentage of descriptors filled in among the collections. The total European genepool is estimated at 27 437 accessions, maintained at 16 genebanks. The majority of these accessions are maintained in Russia, Romania, Germany, Czech Republic and France. In EURISCO approximately 17 175 *Linum* ssp. accessions are recorded of 31 institutes from 21 countries (Pavelek, 2009).

A new IFDB passport and E/CH structure after the meeting of Flax and Hemp Group by Sugar, Starch and Fibre Crops Network in Wageningen 2006 (Bas *et al*., 2006) has been adopted and demonstrated in the database managers meeting in Quedlinburg 2009 (Pavelek, 2009). It is based on MultiCrop Passport Descriptors (MCPDs) and consists of 17 passport descriptors, 6 additional passport descriptors and 7 recommended E/CH descriptors (Pavelek, 2009). Based on 7934 entries (except 451 entries from Bulgaria) contemporary analysis of IFDB MCPDs showed the results in Table 13.2.

Contemporary status of IFDB concerning the entries of the individual European genebanks and the structure according to the type of use is presented in Tables 13.3 and 13.4.

Table 13.2 Survey of IFDB MCPDs filled in

MCPDs	Filled in (%)
ACCENUMB	40.34
ACCENAME	72.37
INSTCODE	100.00
ACQDATE	36.36
DONORCODE	68.39
DONORCODECTY	30.19
DONORNUMB	47.32
ORGCTY	83.80
GENUS	100.00
SPECIES	100.00
USETYPE	64.19
PLOIDY	53.38
ORIGIN	57.60
CHARMATER	51.94
PEDIGREE	33.97
GROWTHHAB	59.97
LIFETYPE	60.81
COLLYEAR	56.40
COLLSITE	38.76
LONGSITE	44.23
LATITSITE	44.59
ELEVSITE	31.62
COLLNUMB	33.10
COLLDATE	27.83
COLLSRS	42.05
AVAILAB	56.78
ALTERDATE	53.49

Table 13.3 Contemporary status of IFDB at the beginning of 2004

European genebanks – databases included	Number of accessions
Previous IFDB	1385
IPGR Saadovo – Bulgaria	(453†)
CGN Wageningen – the Netherlands (from Internet)	747 (974*)
CAS Latgale region – Latvia	15
IPK Gatersleben – Germany (from Internet)	1680 (2304*)
ENMP Elvas – Portugal	87
Suceava, Livada – Romania	499 (247*)
VIR St. Petersburg – Russia	1486
VNIIL Torzhok – Russia	243
INF Poznan – Poland	200
ABI Tapioszele – Hungary	410
IBC Glukhiv – Ukraine	1182
Total	7934 (8986*)

* All entries are described by passport data and partly by describing data.
† All entries are described by passport and describing data.

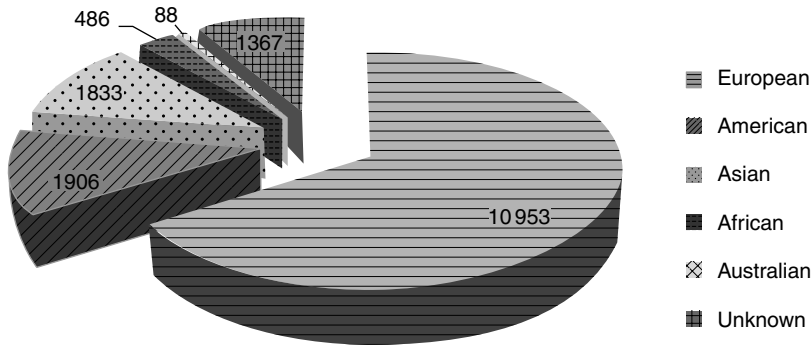
Table 13.4 Structure of contemporary status of IFDB according to the type of use

Use type	Number and % of accessions
Fibre	3700 (46.63%)
Linseed	825 (10.40%)
Combined type	509 (6.42%)
Others	77 (0.97%)
Not specified	2823 (35.58%)
TOTAL	7934

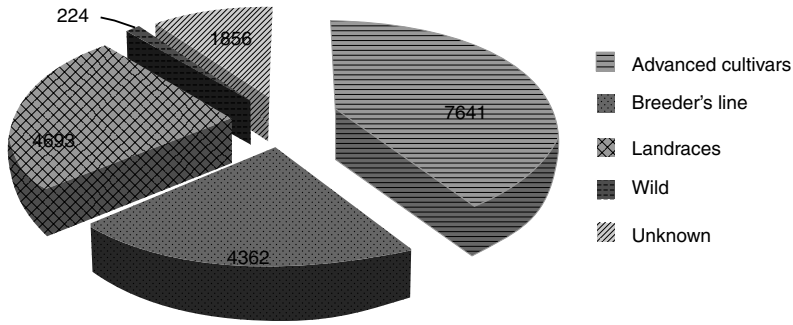
From Table 13.4 especially it is obvious that nearly one-half of IFDB is created with fibre flax, followed with linseed. Unfortunately more than one-third is not specified because this descriptor was not filled in.

Based on geographical analysis it is understandable that most maintained accessions are of European origin (Fig. 13.1). The respective part of Asia's accessions origin is kept in the VIR St Petersburg collection and also a not insignificant part of accessions belongs to the category 'unknown' where the geographical representation is not identified – for example in genebanks IPK Gatersleben – Germany, CGN Wageningen – the Netherlands.

The more significant variability was identified in the category 'origin of the maintained material' (Fig. 13.2). The biggest part in most European genebanks is created by the advanced cultivars, followed by the breeding lines. This trend is marked in the genebanks which are often located in the research or breeding institutes especially interested in breeding activities and the development of their own origin varieties. The category 'landraces' is maintained in the genebanks with long history like in St Petersburg – Russia, Sadovo – Bulgaria, and Sumperk – Czech Republic where the base



13.1 Structure of IFDB according to the geographical origin.



13.2 Structure of IFDB according to the origin.

of collection was just established on old flax and linseed materials at the beginning. Categories 'breeder's lines' and 'landraces' are in IFDB practically comparable. The category 'wild material' is registered in individual genebanks only in Sadovo – Bulgaria, IPK Gatersleben – Germany, and INF Poznan – Poland and it depends on the genebank manager's decision whether this category becomes the part of *Linum usitatissimum* collection or not. The respective part belongs also to the category 'unknown', which is nearly 57% in ISCI Bologna – Italy, 37% in IPK Gatersleben – Germany, and 30% in IBC Glukhiv – Ukraine.

Because in IFDB there are now 8000 incomplete records and the total European genepool consists of approximately more than 27 000 accessions it is necessary to adopt another 19 000 records into the IFDB structure in the future (Pavelek, 2004).

13.3 Methods of flax and linseed breeding

Linum usitatissimum L. species is a self-pollinated crop and its genetic improvement can be carried out through conventional breeding methods

of hybridization and selection on the one hand or utilizing new techniques such as haploidy, interspecific hybridization, mutation, tissue culture and transformation on the other. Commercial breeding of flax started at the end of the nineteenth century (Vromans, 2006). Nowadays, several breeding methods are available, but the pedigree method is the most common one used in flax breeding (Salas and Friedt, 1995).

An indispensable precondition for genetic and breeding research is good knowledge and targeted work with genetic resources collections as mentioned above. This includes both intraspecies variability of certain crops (old and modern cultivars, landraces and experimental exactly defined lines – mutants, transloci lines) and variability of wild forms or forms of a certain genus often crossed with cultural forms. Genetic resources collections demonstrate genepools of all spectrum traits and characters which can be utilized based on hybridization. A very close genetic link between requested and non-requested characters can be considered a large problem by incorporating some abilities from the wild species into cultural forms resulting often in the loss of cultural phenotype and its cultural characteristics. It is often very difficult based on backcrossing to renew the former phenotype. Very important for breeding work (choice of parental genotypes) is the fact that these parental genotypes are equipped with the requested genes on the genetic background of the yielding line.

Hybridization followed by progeny selection is still the main breeding method in flax and linseed breeding programmes. Intraspecies breeding is predominantly used.

13.3.1 Conventional methods of breeding

Conventional methods include intraspecies intervarietal crossing, pedigree method of breeding, and single seed descent (SSD) method:

basic cross: A×B
topcross: A×B, A×C, A×D, A×E
saturating cross: (A×B)×C
backcross: (A×B)×B, (A×B)×A
convergent cross: (A×B)×(C×D)×(E×F)
different types of diallel crosses: A×B, A×C, A×D, B×C, B×D, C×D

Breeding in European countries

Content of fibre in the stem and yield of fibre per hectare are the main characteristics of flax and the subject of improvement by breeding methods. Long and short fibres are used in textile industry, waste products such

as flax shive and tow are used in building, car and air industries for furniture, paper and fuel production, chaff and seeds in the food industry, and flax oil for technical use – varnish, colours and paint production. Genetic resources for fibre content and fibre yield improvement are known and the breeding work is based mainly on traditional methods. The choice of initial useful parental patterns is frequently discussed. Tadesse *et al*. (1997) studied crosses between high × high as well as low × high yielding flax components resulting in the recommendation that hybridization of low and high yielding flax lines may be useful for increasing genetic variability and obtaining high yielding flax lines. Based on results (Pavelek, 2001c) also crosses of high × high yielding flax components can be successful when the genetic diversity of parental lines is large. In case of Venica variety (SL-988 × Viking) registered for growing in the Czech Republic in 2001 (Pavelek, 2001c) parental lines were based on completely different varieties as follows: SL-988 (Regina × Toržokskij), Regina (Reina × /Reina × Fibra), Toržokskij (M-34 × Tomskij-10), Viking (Natasja × Silva), Natasja (Wiera × Mapun). Fibre content in the stem and its quality represented by thinness and divisibility can be considered main criteria for all European countries focused on flax breeding. Selection for fibre content should be effective (Fouilloux, 1988) and fibre content is positively influenced by the recessive genes cumulation (Fouilloux *et al*., 1991). Nevertheless the evaluation of the correlation relationship between fibre content and qualitative characters of the highest breeding stages is very problematic (Kate, 1991).

Based on these traditional methods all fifteen – 11 flax and 4 linseed – cultivars registered for growing in the Czech Republic were bred. The situation is similar in other European countries such as France, the Netherlands, Poland, Romania, former countries of Soviet Union, Ukraine, Lithuania, Latvia and Russia. There are often simple or sophisticated cross combinations derived from the West European and East European cultivars (A × B; /A×B/×C; /A×B/×/B×C/). Also interspecies hybridization was used with the species *Linum crepitans* L. in order to include earliness into the *Linum usitatissimum* L. Success in this direction was achieved, although these early maturing types were not used in further breeding. The effectiveness of interspecies crossing is usually low but it could be increased using the method of embryo tissue culture – cultivation of unmatured isolated embryos in *in vitro* conditions. Breeding of resistant lines is generally carried out on provocation fields infected by pathogens sometimes followed by tests at the stage of seedlings. Also Trouvé (1996) indicated that flax breeding over the past century has used a few high performing genotypes resulting in a significant loss of genetic variability. Recurrent selection breeding is suggested for flax in order to avoid genetic stagnation and to reach genetic population variability (Trouvé, 1996). Good yield of the stem and fibre is positively correlated with the vegetation period. Vegetation period length, course of vegetative

and generative phases as well as fibre content are good inherited characters (Pavelek, 1985, 1991a, 1991b). Beckmann and Kromer (1995) consider fibre content, tensile strength, percentage elongation and Young's modulus as selection criteria for breeding of flax varieties. Khotyleva *et al.* (1997) in agreement with Fouilloux *et al.* (1991) and results (Pavelek, 1980, 1982) reported content of fibre influenced by additive effects and also incomplete dominance and superdominance.

In the second half of the last century the breeding work was focused on increase of yielding potential. A lot of new fibre flax and also linseed varieties were bred in traditional European countries like France, the Netherlands, Czech Republic, Poland and Russia (Ivanova and Shamov, 1996; Pavelek, 2001c; Pavelek and Tejklová, 2005; Scheer-Triebel *et al.*, 1997; Todorov and Lukipudis, 1997) but also in other countries like India and the USA (Foster *et al.*, 1997, 1998; Geleta, 1999; Pradhan *et al.*, 1999; Stephens, 1996; Yadav *et al.*, 2000).

Nowadays the breeding work is predominantly directed on the characters stabilizing the yield, for example resistance to fungi diseases, unfriendly outside factors, qualitatively new content of matters, etc. New methods of breeding work are looked for based on mutagenesis (Rakouský *et al.*, 1998; Tejklová, 1995, 2002, 2008), on molecular approaches (method of isoenzyme spectra – Krulíčková *et al.*, 2002; Pošvec and Krulíčková, 1999; Yurenkova *et al.*, 1992); anther culture, mutation breeding, RAPD analysis (Fu *et al.*, 2003; Mansby *et al.*, 2000); and methods of transformation making it possible to solve the problem when the possibilities of genetic variability are limited and exhausted. Fibre content, and resistance to *Fusarium* and scorch are the most important breeding goals for fibre flax (Fouilloux, 1988), whereas seed yield, fatty acid composition and resistance to rust (*Melampsora lini*) and *Fusarium* are important for linseed breeding (Rowland, 1998).

Alternaria linicola, *Fusarium oxysporum* f.sp. *lini*, *Septoria linicola*, *Melampsora lini*, *Phoma exigua* var. *Linicola* and *Botrytis cinerea* can be considered the most harmful for both flax and linseed throughout the world (Ondřej *et al.*, 2008) and the resistance breeding work is carried out in most European countries. Intensity of occurrence and harmfulness of the respective diseases are strongly influenced and modified by geographical conditions. Complex Alternaria + Oidium + Melampsora occur in subtropical conditions especially, while Fusarium + Septoria + Melampsora + Oidium are located in dry arid areas. In the northwest European countries there are Alternaria + Botrytis; in southwest areas and south countries complex Fusarium + Oidium predominates.

Alternaria linicola is a serious disease of linseed in England, Ireland, Denmark, the Netherlands, Belgium and Normandy. The loss of the yield reaches 10–60% (Chauhan and Srivastava, 1975). This disease is spread by

the seeds or the rest of damaged plants. Information concerning the genetics of resistance is disunited. The resistance influenced by one major gene was demonstrated by Kalia *et al.* (1965) and Evans *et al.* (1995, 1997) while polygenic resistance was found by Klose *et al*. (1993).

Occurrence of *Oidium lini* also known as powdery mildew was detected in 50–60% of cases in the last century in England and Germany especially connected with the decrease of yield in the range 5–20%. In India (Pavgi and Singh, 1965) and China the loss reached 30–60%. Generally it occurs in Europe, Australia, Asia and America. Taxonomy and origin are still not completely investigated. It is supposed to result from the existence of two or three species: *Erysiphe polygoni*, *Erysiphe cichoraceum* and *Sphaeroteca lini.* Heritability of resistance is being studied in India only. Formerly one gene of resistance (R1) was detected (Badwal, 1975), in 2000 two genes and in 2002 three genes of resistance (R1, R2, R3) were demonstrated (Rashid and Duguit, 2002). Based on the results reached in the Czech Republic it was stated that the genetic background of the resistance to *Oidium lini* is unclear, is valid for the set of tested varieties and could be different depending on the choice of varieties (Ondřej *et al*. 2008).

Fusarium oxysporum f. sp. *lini*, *Fusarium avenaceum* and *Fusarium equiseti* are considered to be the most serious diseases of flax and linseed together called Fusarium wilt. It is spread over the world in semi-arid or arid areas especially. This disease is one of the limiting factors of flax and linseed growing in the USA and Canada (Kommendahl *et al*., 1970). Thanks to the programme of resistant breeding in North America all commercial flax and linseed cultivars are characterized as resistant or moderate resistant (Kenaschuk and Rashid, 1993; Kenaschuk *et al*., 1996). Resistant flax and linseed cultivars were also developed in Europe (Kroes *et al*., 1998, 1999). Wide pathogenic variability in *Fusarium oxysporum* f. sp. *lini* has been reported. Many different biotypes and pathogenic types of *Fusarium oxysporum* f. sp. *lini* have been detected and isolated (Armstrong and Armstrong, 1968; Kommedahl *et al*., 1970) from different regions in the world: Argentina, the USA, Canada, Australia, India, Japan (Borlaug, 1945; Kroes *et al*., 1999, 2002, cited in Muir and Westcont, 2003; Houston and Knowles, 1949; Milikan, 1945, 1948; Sharma and Mathur, 1971; Tochinai and Takee, 1950). Some varieties are more susceptible than others. Resistance is probably influenced by the minor genes and it has a polygenic character. Investigations carried out in eight countries on nine localities during 1995–1996 showed that population virulence in the USA and Canada was very strong, medium in France, Germany, Czech Republic and Russia, and very weak in Belgium and the Netherlands. The most resistant varieties were AC Linora, AC Emerson and Atalante. The most susceptible were Ocean, Barbara, Liflora and McGregor varieties (Kroes, 1997). In the Czech Republic 80 linseed breeding lines were tested in field conditions in 2005. 47.9% of the lines showed medium

resistance, while 52.1% were medium susceptible. In 2007 55 breeding lines were evaluated. 41.8% of the lines were resistant including standard varieties Atalante and Jantar. Medium resistance was detected in 25.6% lines also linseed varieties including Bajkal and Lola. 20% lines showed medium susceptibility with linseed varieties Flanders, Lindor, Recital and Kaolin. Susceptibility was detected in 7.2% of the lines of varieties Niagara, Baladin and Alaska and high susceptibility was found in 5.4% of the lines including Astral, Jupiter and Ocean varieties (Ondřej *et al.*, 2008). The varying levels of resistance or susceptibility are evidence of the polygenic character of the resistance and it can be used by the breeding work on the provocation fields (Ondřej *et al.*, 2008).

Breeding work is carried out according to breeding aims. For flax and linseed these are presented in Table 13.5.

Through the breeding aims the following target parameters should be reached:

Flax (*Linum usitatissimum* L.)
Flax ideotype:

- resistance to lodging: 9 p;
- resistance to pathogen complex: 8 p;
- middle vegetation period;
- yielding potential of unretted stem reached in trials: 7–8 t.ha^{-1};
- yielding potential of the seeds reached in trials: 1.10–1.30 t.ha^{-1};
- long fibre content potential reached in trials: 22–25%;
- total fibre content potential reached in trials: 39–41%;
- long fibre yielding potential reached in trials: 1.25–1.40 t.ha^{-1};
- total fibre yielding potential reached in trials: 2.50–3.0 t.ha^{-1}.

Table 13.5 Breeding aims for flax and linseed

Flax	Linseed
• High resistance to lodging • High resistance to pathogen complex • Middle vegetation period • Average unretted stem yield • High content of fibre in the stem • High yield of fibre per hectare • New quality: low linolenic acid content	• High resistance to lodging • High resistance to pathogen complex • Middle vegetation period • Low stem yield • High seed yield • High content of fat in the seeds • High fat yield per hectare • New quality: low linolenic acid content

Cultivars will be enhanced by:

- low linolenic acid content: less than 5%;
- increasing absorption and accumulation of Cd and Pb, increased toleration to Cd and Pb;
- resistance to herbicide Basta.

Linseed ideotype:

- resistance to lodging: 9 p;
- resistance to pathogen complex: 8 p;
- middle vegetation period;
- seed yielding potential reached in trials: 2.30–2.40 t.ha.$^{-1}$;
- fat content potential reached in trials: 42–46%;
- fat yielding potential reached in trials: 980–1000 kg.ha^{-1}.

Cultivars will be enhanced by:

- low linolenic acid content: less than 5%;
- increasing absorption and accumulation of Cd and Pb, increased toleration to Cd and Pb;
- resistance to herbicide Basta.

Breeding in India, the USA and Canada

India, the USA and Canada are the biggest growers and producers of linseed in the world (*Situační a Výhledová Zpráva*, 2009). A list of linseed areas and production figures in 2003–7 are presented in Table 13.6.

It is obvious from the table that the production in India has an extensive character in comparison with the USA and Canada and according to Gill (1987) the seed yield is very low. Therefore the main breeding goals in India especially but also in other overseas countries (Gill, 1987) are:

- improvement of the seed yield;
- improvement of content and quality of oil;
- resistance to diseases and pests;
- early maturing cultivars development.

The breeding approaches are similar to those in Europe including conventional breeding methods of selection and hybridization and also new techniques such as male sterility, haploidy, interspecific hybridization, mutation and tissue culture (Gill, 1980).

Table 13.6 Survey of linseed areas (ha) as well as the production (mill. t) in India, the USA and Canada, 2003–2007

	2003		2004		2005		2006		2007	
Country	Area	Production	Area	Production	Area	Production	Area	Production	Area	Production
India	450 100	176 700	476 500	196 500	448 700	169 700	436 800	172 500	426 000	167 000
USA	235 900	264 830	206 800	263 360	386 480	500 280	310 400	279 00	141 235	149 963
Canada	728 400	754 400	518 000	516 900	732 600	990 600	785 200	988 800	524 000	633 500

Breeding in India

Pure line selection

This method was used at earlier stages of linseed improvement (Gill, 1987) and many of the old linseed varieties originating from India were pure lines, e.g. NP 12, NP 121, NP 124.

Hybridization

Hybridization has been used to combine different characters in one material since the beginning of the last century (Gill, 1987). This method was often used for incorporating the resistance from different sources into the hybrids. The pedigree method was the most used method at the time (Gill, 1987).

Pedigree method of breeding

The pedigree method of breeding, commonly used from the 1930s onwards (Culbertson, 1954; Grahan and Roy, 1924; Hayes *et al*., 1955; Kenaschuk, 1975), includes three main steps:

- crossing between parental varieties possessing the characters to be combined;
- growing the progenies of the selected types;
- keeping accurate records in order that the individual plants can be traced from one generation to the next.

A detailed description is presented by Gill (1987). The pedigree method of breeding has been used for developing the improved linseed varieties in India. In India there are different linseed breeding centres located in different states focused on various characters. Eight states in India – Bihar, Delhi, Gujarat, Madhya Pradesh, Punjab, Uttar Pradesh, Rajasthan and West Bengal – are involved in linseed breeding (Gill, 1987). Linseed breeding in India is very well organized and it has an old history. The research programmes on linseed were strengthened in 1947 by the Indian Central Oilseeds Committee (ICOC), later under the project 'Intensification of Regional Research on Cotton, Oilseeds and Millets' (PIRCOM) in 1958. Because the results of breeding work were not as good as expected the Indian Council of Agricultural Research started the All India Coordinated Research Project on Oilseeds (AICRPO) in 1968 (Gill, 1987) using international approaches. As a result of selection and hybridization several different linseed varieties were developed from time to time as reviewed below.

Bihar

Breeding work carried out in Bihar resulted in linseed varieties Sabour 6, BR1, BR2, BR9, BR12, BR29, BR35, BR40 bred before 1956. The other

varieties covered the period between 1964 and 1969 represented by NP 5 and NP 142 especially compared to 3/2, T 397, 17/34, K 2, B 67 and B 37. Since 1969 the breeding work has again intensified and testing continued with varieties N 2, LC 36, BS 11, BS 44, NP (RR) 9, B 67, NP (RR) 5, T 397, L 52, LS 3 and FRW 9.

Delhi

Linseed breeding in Delhi started in 1915 with 123 distinct types isolated from the collection of seed samples from all over India (Howard and Khan, 1924). For cultivation in North India three varieties were recommended: NP 12, NP 121 and NP 124. Breeding work was carried out by the Indian Agricultural Research Institute (IARI). In 1933 work on breeding for disease resistance was started for the first time in India. Resistant genes were incorporated from the American varieties in 1930–1 and the initial materials were used as parents in crosses combining resistance to rust with economically important characters (Desphande, 1950). Nine varieties were recommended for cultivation in different states: NP (RR) 5 (Bihar), NP (RR) 9, 38, 45 (Delhi), NP (RR) 204 (Gujarat), NP (RR) 9, 204, 272 (Madhya Pradesh), NP (RR) 9, 10, 37, 45 (Punjab), NP (RR) 9 (Utar Pradesh), NP (RR) 38, 45 (Rajasthan), NP (RR) 38, 40 (West Bengal). All these varieties are marked RR – rust resistant. The present campus of IARI is a self-contained aesthetically laid out sylvan complex spread over an area of about 500 ha. The beautiful clock-towered building of the Central Library of the Institute constitutes the focal point of the campus around which stand the laboratory buildings of various divisions, staff quarters, students, hostels, guest houses, a medical dispensary and schools for boys and girls. The experimental fields, which form an integral part of the IARI campus, cover an area of about 340 ha, of which about 300 ha are irrigated by an interlinked chain of tubewells and water storage tanks, while the remainder is used for dry land farming research experiments.

Madhya Pradesh

The breeding work on linseed varieties development was conducted at the College of Agriculture, Indore. Resistance breeding was started in the 1950s and 21 varieties showed resistance to both rust and wilt. The main breeding aims are: resistance to rust, wilt and powdery mildew, high seed yield, high oil content. The contemporary breeding work is focused on dual purpose varieties and late date of sowing after harvesting paddy. Three varieties were bred and recommended for cultivation: Jawahar 17, Jawahar 522 and R 7. These cultivars are now overtaken by the varieties R 936, R 948, R 987, R 1001, R 1004 and R 1017.

Punjab

The breeding work began at Lyalpur in the area of today's Pakistan. Three types were selected: Type 5, Type 23 and Type 31. Type 5 was commercially successful and exported also to the USA resulting in the variety K2 registration in 1954 suitable for Punjabi conditions. In order to discover other germplasm useful for the Punjab plains 69 exotic linseed lines were studied in 1957–8 (Gill and Sigh, 1960). Three of them – lines LI 5743, 5465 and 56104 – were detected as resistant against rust and wilt and line LI 5465 was also immune to powdery mildew and was used in further breeding work. Yellow seeded strains were tested in order to select high yielding and resistant strains (Gill and Singh, 1959, cited in Gill, 1987) and this work resulted in the development and releasing of the high yielding variety LC 185 in 1970 derived from the cross between NP (RR) 37 × Kangra Local (Gill, 1975). Better than K 2 were the strains LC 45 and LC 54. Strain LC 45, high yielding and also resistant to rust and wilt, has been released as the variety Himalini in Himachal Pradesh.

Uttar Pradesh

The breeding work started in 1923 in Kanpur resulting in three varieties named Type 477, Type 486 and Type 50 being released. Unfortunately their genetic variability was rather pure so hybridization was undertaken in order to combine rust resistance with other agronomic characters (Mehta and Mital, 1951, cited in Gill, 1987). Systematic breeding work in the 1950s led to the development of different types H 397, H 603-2, T 397 and T 603. Their mutual crossing resulted in the lines 485, 491, 1193-1, 1193-2 and based on hybridization of the lines 485 and 1193-1 the selection T 126-2 was finally originated in 1958 followed by the three new varieties Hira, Mukta and Neelum (Gill, 1987).

West Bengal

Breeding work in the West Bengal area was focused on dual purpose flax development for both oil and fibre. These activities have been carried out since 1936 resulting in hybrid No. 37 development. Two varieties – B 67 (Neda) and B 96 – were released.

The pedigree method of breeding can be modified (*Modified pedigree method*) and different approaches to crossing (*Backcross method*, *Recurrent selection*) can be used according to the breeding aims.

Modified pedigree method

This method is based on the single seed descent after hybridization followed by the homozygosity process for several generations. Usually in F_5

generation each progeny is maintained in bulk (Brim, 1966). Choice of the individual plants can be applied for the traits with high level of heritability (rust resistance for example), while the characters of complex disposition (like yield or other less heritable characters) are easier to detect after the homozygous lines establishment. This method can be carried out when the parental varieties are very well adapted to the outside conditions.

Backcross method

This method is recommended when desirable genes need to be incorporated into the genotype, for example resistance to diseases into the susceptible varieties. The method was described by Kenaschuk (1975) and it was used not only for resistance incorporation but also for yellow seed incorporation into the brown seeded variety (Gill, 1964).

Recurrent selection

Recurrent selection by linseed can be applied when undesirable linkages need to be overcome. This method was successfully used in cotton (El-Adl and Miller, 1971; Miller and Rowlings, 1967), in soybean (Hanson *et al.,* 1967), in wheat and barley (Redden and Jensen, 1974) and in tobacco (Matzinger and Wernsmann, 1968).

Mutation breeding

Mutation breeding has a quite long history in flax and linseed and chemicals or rays were often used to improve requested characters. Favourable mutations are reported for many different traits by various authors. Improvement of oil content was demonstrated by Larter *et al*. (1965), Rath and Scharf (1968), Srinivasachar and Malík (1971), Seetharam (1972) and Srinivasachar *et al*. (1972). Other improvements include: seed yield (Bari, 1971); iodine value (Harpstead, 1961; Rath and Scharf, 1968; Srinivasachar and Malík, 1971; Srinivasachar *et al*., 1972); seed weight (Seetharam, 1972); and height and maturity (George and Nayer, 1973).

Interspecific hybridization

All the above-mentioned linseed breeding centres in India predominantly used the pedigree method of breeding. Nevertheless interspecific hybridization has also been used in order to improve and extend the genetic variability of fatty acid composition (Gill, 1966; Yermanos, 1966). Interspecific hybridization, however, is difficult due to the different numbers of chromosomes reaching 8, 9, 10, 12, 14, 15, 16, 18, 30 and also more than 30 haploid chromosomes. The method was only successful when crossing the species

with the same number of chromosomes as in the case of *Linum usitatissimum* L., for example, which could incorporate resistance to the rust *Melampsora lini.* Some species like *Linum grandiflorum*, *Linum perenne* and *Linum austriacum* are self-incompatible and crossing between them and *Linum usitatissimum* species was not successful (Ghai, 1966; Gill, 1966).

Hybrid linseed

Production of hybrid varieties accompanied by vigour or heterosis effect is connected with knowledge about floral morphology and the mode of pollination. Although heterosis by flax ranged from 40% (Carnahan, 1947) to 231% (Dubey and Singh, 1969) flax and linseed hybrid production is not so usual as in maize, sugarbeet, sorghum, onion or pearmillet. The main reason is the flower of the male sterile flax has a small corolla which fails to open to allow cross-pollination. The problem of male sterile flax was investigated by Dubey and Singh (1965, 1966), Kumar and Singh (1970, 1972) and Kumar (1971) . In India, especially, lot of studies were devoted to the investigation of combining ability of flax or linseed varieties in diallel crosses permitting also the evaluation of heterosis effect. Positive and significant heterosis for seed yield per plant at 40 crosses and fibre yield at 13 crosses was confirmed by Pant and Mishra (2008). This effect is often derived from the cumulative effect of the additive genes, dominance, or non-allelic interaction. Hence the success of any selection or breeding programme depends on precise estimation of various components (Mohammadi *et al.*, 2010). The mutual relations of the genes expressed by the general combining ability (GCA) or specific combining ability (SCA) influence manifestation of the respective characters. GCA by flax and linseed is connected with traits like plant height, number of capsules per plant, weight of capsules per plant, seed yield (Popescu *et al.*, 1999) while non-additive effects expressed by SCA were confirmed by Sood *et al.* (2007) for number of the seeds per capsule, 1000 – seed weight. Sometimes both additive and non-additive gene actions were found (Bhateria *et al.*, 2006).

Breeding in Canada

Canada is the world's largest producer of oilseed flax, with Saskatchewan producing about 80% of the flax grown in western Canada with a current production of 1.041mt (Flax Council of Canadaweb sites, production in 2005/2006). There are three breeding programmes in Canada focused on the development of brownseeded flax types and yellowseeded low linolenic linseed types under the common name 'Solin'. The first two programmes called 'Agriculture' and 'Agro-Food' Canada are carried out in Morden and Manitoba. The third one focused on the development and breeding of agricultural crops is undertaken at Saskatchewan's university in Saskatoon (Flax Council

of Canada, Saskatchewan Flax Development Commission). The first two programmes started in 1900 and based on them the first flax fibre varieties were developed: Diadem, Ottawa 770 B, Ottawa 829 C and Novelty. In the 1950s varieties Linott, Raja and Rocket were bred and in the 1960s varieties Dufferin, McGregor, Norlin, NorMan, AC Linora, AC McDuff, AC Emerson, AC Carnduff and AC Lighting were developed. Breeding work at the university in Saskatchewan started in the 1920s and from this period the varieties Royal and Redwood 65 originated. In 1974 the breeding programme was prolonged and updated and based on it the varieties Vimy, Somme, Flanders, CDC Normandy and CDC Valour have been bred.

Mutation breeding was the main breeding method used by both the Australian and Canadian breeding programmes which started development of linseed varieties with completely different fatty acid composition in comparison with traditional composition standard in the species *Linum usitatissimum* L. Using X-ray irradiation the linseed variety Redwood 65 was bred and from it the linseed variety Dufferin registered in 1979 was derived (Micke *et al*., 1985). Via chemomutagenesis new fibre genotypes resistant to wilt (*Fusarium oxysporum*) were developed and in Australia the genotypes with various fatty acid composition completely unique to *Linum usitatissimum* L. species with lower linolenic acid content (from 60% to ca. 30%) were created. Based on mutual crossing of these types with lower linolenic acid content quite new types have been bred with unique composition of fatty acids not known in *Linum usitatissimum* L. species till this time (Green, 1986a, 1986b). The linolenic acid content is less than 3% while linoleic content was raised from approximately 15–70% (Dribnenki and Green, 1995). From these types many other low linolenic linseed varieties were derived under the name 'Linola' and commercialized at the beginning of the 1990s (Dribnenki and Green, 1995; Green, 1992). The first linseed variety originated in the framework of this programme was Linola TM947, a late maturing yellow seeded variety with very high oil content in the seeds and good resistance to lodging (Dribnenki *et al*., 1999). The other one is Linola TM2047 (Solin 2047) (Dribnenki *et al*., 2003). This variety is immune against strains of rust (*Melampsora lini*) of North American origin and oil content is higher than in Linola TM947. The third one is Linola TM2090 (Solin 2090) (Dribnenki *et al*., 2004) which is good in oil content, seed yield and it is also immune to North American strains of *Melampsora lini* and relatively resistant to Fusarium wilt *Fusarium oxysporum* f. sp. *lini*. The last two varieties were produced by the Canadian company Agricole United.

Nowadays the breeding work in Canada is focused on dual purpose flax *Linum usitatissimum* L., to determine whether the plant of linseed can be used for both oil and fibre (Nandy and Rowland, 2008). For dual purposes (seed/oil, straw/fibre) it is recommended to use crosses linseed × linseed or linseed × flax lines rather than flax × flax ones (Foster *et al*., 1997, 1998).

Recombinant inbred lines (RILs) based on crosses of linseed × linseed or linseed × flax lines showed positive genotypic correlations resulting in increasing the chances of extracting RILs producing both high seed/oil and straw/fibre yields (Foster *et al.*, 2000).

Current studies have shown no tight linkage between stem fibre content and oil traits indicating the possibility of developing dual purpose varieties (Foster, 1997). It is known that product quality and productivity can be improved by a better understanding of the material structure. To increase the fibre yield and identify superior varieties for fibre production it is very important to know in detail the ultrastucture of the fibre bearing stem tissues (Roland and Vian, 1991). In order to combine both fibre and oil in one plant RILs were studied using light microscopy and molecular markers to screen the parents and the RIL population to identify polymorphic markers for the identification of possible dual purpose varieties (Nandy and Rowland, 2008).

Breeding in the USA

Almost all the activities connected with flax and linseed are concentrated in North Dakota. North Dakota produces over 90% of the flaxseed in the USA. The value of the flaxseed crop in North Dakota is estimated at $45 million per year. During recent years, the USA has been a net importer of flaxseed. At present, the only flax breeding and genetics programme in the USA is situated at the North Dakota Agricultural Experiment Station. In fact, the programme at NDSU is one of only three in North America. The value and market for flaxseed as a healthy food continues to develop. A recent news release indicated that a major baby food manufacturer will be adding an enriched Omega-3 product. This could require a significant quantity of flaxseed. Research continues to support flaxseed as a healthy choice for heart health as well as reduced cancer risk.

The primary objective is to develop and evaluate genetic material to improve yield potential while maintaining resistance to pests, maintaining oil content and oil quality and maintaining other agronomic characteristics for potential cultivars. Since producers have historically planted later than would be expected to produce greatest yields, a part of the breeding effort will be devoted to evaluation of a delayed seeding date. With the interest in flax as a human food, a minor effort will continue to evaluate material with a yellow seed coat colour which is preferred for 'eye appeal'.

Main breeding goals are as follows:

- to develop flax cultivars with desirable agronomic characteristics; seed yielding ability; quantity and quality of oil;
- to reach tolerance to wilt and pasmo;
- resistance to known North American races of rust.

The breeding program is divided into two major parts: (1) rust resistance for two genes conditioning resistance (M^3 P^3), and (2) use of other genes for resistance (either as one gene or multiple). The two parts would require different groups of crosses. In addition, a minor effort will be to continue to evaluate golden (yellow-seeded) flax as preferred in the human food market. Most of the breeding programmes, as in India or Canada (Kenaschuk, 1975), will follow the pedigree breeding scheme. The details of the planned breeding methods to be used are as follows:

- Cross and F_1 generation: crosses between selected parents are made in the greenhouse and the F_1 plants are grown in a greenhouse.
- F_2 generation: from 500 to 3000 F_2 plants are grown the following year in the greenhouse and evaluated for rust resistance. Resistant plants are grown to maturity.
- F_3 generation: progenies of rust resistant F_2 plants are grown in the field and selection is made on agronomic appearance for the best plants within the best family. Normally, four plants are selected from each selected family.
- F_4 generation: plants are evaluated for rust resistance in the greenhouse and only lines homozygous for resistance will be planted in the field the following year. Four to six plants will be selected from selected families.
- F_5 generation: seed from selected F_4 plants are planted as hills. Selected hills will be advanced.
- F_6 generation: lines are grown in field trials for yield evaluation. In addition, data on other characters such as flowering date, plant height, wilt tolerance, etc. are collected. In later generations, yield testing will be conducted at multiple locations on selected lines. The most promising lines will be evaluated in 'tri-state' trials. All lines in the 'tri-state' nurseries will be evaluated for oil percentage, iodine value, height, maturity, lodging and pasmo as well as yield in both early and late seeded nurseries. Presently, the 'tri-state' nurseries are grown in ND, SD, and Manitoba, Canada. Each cooperating station grows two trials per season. Selected lines from 'tri-state' nurseries are grown in regional trials in the flax growing area. As a final stage of evaluation, lines will be evaluated in Research Extension Centre trials and increased, named and released. Selected material of high priority will be rapidly advanced in a modified single seed descent method to 'pure' lines for yield evaluation.

Population improvement

The secondary objective to develop and maintain populations with useful genetic variability is necessary to support the development of improved cultivars. Much of this effort will centre on the health food aspect of flax. Yellow seed coat is

preferred in human food products. In addition, concern has been expressed concerning the level of Cd in the seed of several plants including flax.

Several potential sources of yellow seed colour have been identified in the US collection of flax (Miller and Hammond, 1988). Several lines have been selected for crossing with low accumulating Cd lines (additional breeding is planned after evaluation of Cd content in the crossed material). In addition, crosses have been initiated with selected advanced yellow seeded experimental lines and adapted brown seeded lines. The segregation of advancing generations will be followed to determine mode of inheritance of yellow seed coat colour and potential as varieties for the health food market. As the material is developed it may be important to evaluate other quality characteristics for health benefits (lignan content, etc.). Populations need to be available in the event of market change. Diverse populations will be maintained to attempt to meet changes in demand.

Breeding in China

Very obvious progress has been also made in China during last several years both in traditional and biotechnological methods of breeding resulting in registration of many fibre and linseed varieties (Li, 1994; Li *et al.*, 1997; Quiang-HeShun *et al.*, 1996; Song *et al.*, 1996; Zhang *et al.*, 1996). Also gamma radiation was used for breeding of cultivar Heiya 4 (1978) (Mutant variety database, FAO IAEA) which is resistant to lodging, tolerant to wet soil and higher pH. The other flax Chinese varieties were derived from this cultivar.

Conclusions

Requested characters can be found in another species; however, it is difficult to transmit them due to hybridization barriers. Also induced mutagenesis is nearly at the top of its utilization (mutagenesis is a causal phenomenon of statistically less effective requested large populations with high working demands). However, the technologies used in modern industry require new varieties equipped with new characteristics. Sustainable agriculture requires more ecologically friendly ways of growing and for systems of phytoremediation of contaminated soils. A revolutionary development might view the plants as 'factories' for the synthesis of specific compounds. Thus the traditional methods are likely to be augmented or overtaken by methods of cellular and genetic engineering and molecular biology.

13.3.2 Unconventional methods of breeding

Haploid production in flax

Crop improvement is based on the extension of genetic variability followed by selection of suitable phenotypes with desired combination of features,

and then reduction to eliminate genetic variability to meet the conditions of distinctness, uniformity and stability in newly developed varieties. Extension of the variability for desirable properties is induced by intra- or intergeneric hybridization, mutation induction, somaclonal variation utilization or somatic hybridization. Genotype stabilization is achieved either by the classical breeding methods (pedigree selection, bulk breeding) or by using haploids. The second method is very effective: completely homozygous genotypes, non-segregating in any trait, are obtained in one generation. For comparison, it took even than two decades from the mutation induction to the official registration of the first low-linolenic variety Linola 947 using the classical breeding method (Dribnenki and Green, 1995).

The oldest method of haploid production is the polyembryonic method (Rosenberg, 1974). Some genotypes are able to produce twins in one seed, from which mostly one is diploid and one is haploid, but in a very low frequency and, moreover, induction of twinning into breeding materials is a very lengthy procedure.

Some biotechnological methods were studied for haploids and subsequently double-haploids (dihaploids, DH) production in flax (Poliakov *et al*., 1994): pollination of emasculated flowers with pollen grains chemically or physically treated, treatment of plants with biologically active substances, pollinating diploid flax cultivars with tetraploid ones, interspecies crossing (the so-called Grandiflorum method described in Poliakov *et al*., 2001), ovary culture, microspore culture or anther culture.

Microspore culture

The first information about flax microspore culture establishment and androgenic regenerated plants was published in Nichterlein *et al*. (1991a). Nichterlein and Friedt (1993) reported detailed methodology of shoot regeneration from microspore-derived embryoids and microcalli in microspore culture. The highest frequency of regenerated plants was obtained in microspore cultures of the hybrid Atalante × Szegedi 62 (F1) at 30°C. From 61 microspore calli 65.6% produced shoots. Shoots were rooted on MS medium containing indolylacetic acid (IAA) (0.1 $mg.L^{-1}$), silver nitrate (1 $mg.L^{-1}$) and Gelrite (6 $mg.L^{-1}$). From 113 inoculated shoots, 44 surviving plants were obtained from the more responsive hybrid. The majority of the plants were haploid. After colchicine treatment, 24% of the initially haploid plants produced seeds.

However, the overall efficiency of plant regeneration is still not suitable for practical breeding (Obert *et al.*, 2009).

Ovary culture

The first results of experiments with ovary culture and callogenesis in ovary culture were reported by Bartošová and Preťová (2003). Bartošová *et al.*

(2003a, 2003b) presented callogenesis and shoot regeneration in culture of isolated ovaries. After that several other works followed. Obert *et al*. (2005) reported the highest frequency of callusing ovaries: 64% in genotype AC Emerson on N6 medium with growth regulators as for the anther culture. Ploidy of callus cells was 2*n*, 4*n* up to 16*n*. Regeneration from ovary calli was realized via bud and shoot formation.

Bartošová *et al*. (2005) reported shoots obtained in ovary culture which were diploid. Callus-induction and regeneration rates in ovary culture were genotype-dependent (Bartošová *et al*., 2006). Flow cytometry analysis showed 2*n* in yellow-green calli of genotypes Viking and AC Emerson and 4*n*, 6*n*, 8*n*, 16*n* in dark green calli.

Burbulis *et al*. (2007b) reported effect of genotype, growth regulators and level of sucrose on callus induction in ovary culture.

Ovary culture is at the beginning of its development and it is not yet sufficiently effective to be used in flax and linseed breeding.

Anther culture

Anther culture is the most promising, most studied and most successful method of haploid and DH production in flax. Microspores developing in anthers have a reduced number of chromosomes (*n*). They develop into pollen grain coming through the several developmental stages. In the uninuclear stage, they are able to change their direction of development in specific *in vitro* conditions and haploid plants can grow up from them. Such plants are not fertile, they must be treated with some mitotic active substance to double their chromosome set. In many cases, however, spontaneous diploids are caused by endomitosis. Progenies of DH plants are completely uniform in genotype and they cannot segregate in any character. The absence of a dominant effect in complete homozygotes makes selection of desired genotypes easier (Poliakov *et al.*, 1994). That is why using of anther culture can reduce the time needed for obtaining pure lines.

Anther culture seems to be an excellent method for breeding interval abbreviation commonly. It has been developed progressively since the 1960s in different plant species. Guha and Maheshwari (1964) reported production of Datura haploid plants by culturing anthers. Anther culture in flax has been studied since the 1970s.

The first experiments on flax anther culture were described in Sun (1979). Soon afterwards, Sun Hong Tao and Fu Wei Dong obtained complete plants from F1 hybrids through the callogenesis in isolated anthers, bud regeneration in calli, shoot development from buds, and shoot rooting (Sun and Fu, 1981). This method is called indirect regeneration in contrast to embryogenesis, which is named direct regeneration. Cytological tests confirmed

microspore origin of regenerated plants because haploid number of chromosomes was detected.

Anther culture as an object of research

A system of indirect plant regeneration from the microspores in flax anther culture was assumed and further improved by other researchers in different laboratories around the world. Experiments were conducted on flax and linseed as well. There are no differences among those two cultural types, for fibre (flax) and for seeds (linseed) production, in *in vitro* cultures. The first results of experiments with flax anther culture from individual laboratories were reported: Nichterlein *et al.* (1989a) (Institute of Agronomy and Plant Breeding, Justuts-Liebig-University, Giesen, Germany), Poliakov (1991) (All Russian Flax Research Institute, Torzhok, Russia), Tejklová (1992) (Agritec, Research, Breeding and Services Ltd Šumperk, Czech Republic), Rogalska and Rutkowska-Krause (1994) (Institute of Natural Fibres, Poznan, Poland), Preťová *et al.* (1995) (Institute of Plant Genetics and Biotechnology SAS, Nitra, Slovak Republic), Lassaga and Bretón (1997) (Estacion Experimental INTA, Paraná, Argentina, Fac. de Ciencias Agropecuarias, Universidad Nacional de Entre Ríos, Paraná, Argentina), Spielmeyer *et al.* (1998) (CSIRO Plant Industry, Canberra, Australia), Chen *et al.* (1998a, 1998b, 1998c, 1998d) (Agriculture and Agri-Food Canada, Morden Research Centre, Morden, Canada), Kurt and Evans (1998) (Department of Agronomy, Faculty of Agriculture, University of Ondokuz Mayis, Samsun, Turkey), Soroka (2004) (Institute of Oilseed Crops, Ukrainian Academy of Agricultural Sciences, Zaporozhye, Ukraine), Song *et al.* (2004) (Raw Material and Industry Institute of Flax, Heilongjiang Province, Shuangcheng 150111, China), Burbulis *et al.* (2005a, 2005b) (Laboratory of Genetics and Biotechnology, Faculty of Agronomy, Lithuanian University of Agriculture, Kaunas, Lithuania), Grauda *et al.* (2005) (Institute of Biology, University of Latvia, Salaspils, Latvia) and Sun (2009) (Agronomy College of Inner Mongolia Agricultural University, Huhhot 010019, China).

Method optimization – study of factors influencing efficiency of method

Researchers have solved the problems associated with each step of anther culture: callus induction, determination of ploidy in callus, bud regeneration, shoot elongation, shoot rooting, determination of ploidy in plants, colchicine treatment and transfer of plants into soil (Fig. 13.3). The aim of their effort was to develop a universal highly effective method of DH lines production from microspores via anther culture applicable to large flax and linseed breeding material.

Over the years many experiments have been undertaken for the identification of factors affecting individual steps of anther culture.

Genotype

Anther culture is developed for linseed and flax varieties (Nichterlein *et al*., 1989b). Production of anther calli was generally higher in flax genotypes (Tejklová, 1996), and conversely, bud regeneration was higher in linseed genotypes (Tejklová, 1998). Bud regeneration was lower in hybrids having flax genotypes as one parent, compared to linseed ones (Rakouský *et al*., 2001). Genotype of donor plants is shown to be an important factor influencing regeneration capacity of anthers (Nichterlein *et al*., 1991a). Nichterlein *et al*. (1991b) noted the various responses of linseed varieties to cultivation conditions at all phases of plant regeneration (callogenesis, bud and shoot regeneration, shoot rooting).

Callogenesis and organogenesis were higher in F1 than in parental varieties (Poliakov *et al.*, 1994; Rogalska and Rutkowska-Krause, 1994); the highest frequencies of both characteristics were reached in variety Orshanski-2. Chen *et al*. (1998b) described the highest callus induction (63.5%) and overall efficiency of regeneration (12%) in one F1 linseed hybrid. Higher frequency of bud regeneration per isolated anthers in F3 hybrids compared to pure lines was demonstrated in Rakouský *et al*. (2001), as well, but in Tejklová (1996), differences among genotypes in callus induction were more significant than differences between linseed and flax varieties or between varieties and hybrid populations.

Differences among genotypes in anther callus production were described by Vigier and Bretón (2005), Gonzales and Bretón (2005) and Burbulis *et al*. (2005) (response namely on growth regulator combinations). Variation for anther culture response was significant both within and between the hybrids and their parents (Burbulis and Blinstrubiene, 2006). On the other hand, Tejklová (1992) reported that from 15 flax and linseed genotypes, only linseed Duferin was without any callus induction. Frequencies of callogenesis were similar in all genotypes.

Wielgus and Mankowska (2007) and Wielgus *et al.* (2008, 2009) reported differences in callus production in anthers of F1 from different combination crossing of linseed. Anthers from some crossings were not able to form callus while anthers from combinations Linola with Szafir, Opal and Bionda were characterized by higher than average capacity to form callus.

Genotype had a significant effect on callus induction and shoot regeneration from anther-derived calli of linseed and flax (Chen *et al*., 1998b; Poliakov *et al*., 1994). Cultivars significantly affected callus induction rate both on solid and liquid medium (Kurt and Evans, 1998). Chen *et al.* (1999) demonstrated a strong genotype effect on callus induction and shoot regeneration in anther culture in their study on 44 flax genotypes with diverse genotypic

background. Some genotypes responded well to media with different levels of growth regulators (Chen and Dribnenki, 2002; Chen *et al.*, 2003).

Significant differences in callus induction in anthers and plant regeneration among genotypes were observed by Rutkowska-Krause *et al.* (2003, 2004) and Obert *et al.* (2003, 2004). Only one of four varieties produced shoots in the anther culture reported by Burbulis *et al.* (2005).

Areco was a more responsive genotype compared to Marina (Tejklová, 1998). Callus induction and bud regeneration, as well, were higher in Areco. Sun (2009) and Lassaga *et al.* (2010) compared regeneration capacity in different linseed genotypes, and Burbulis *et al.* (2009) selected four responsible genotypes from nine studied linseed varieties in their experiment. Results illustrated that genetic background is important in callus induction and shoot production in the anther culture.

A strong genotype effect on callus induction and shoot regeneration was described in Burbulis *et al.* (2009). They suggested determining specific combinations of growth regulators for each genotype. On the other hand, the callus induction and plant regeneration in anther culture from both types of flax (for fibre and for seed production), highly homozygous varieties, and hybrid populations, as well, were obtained on the same culture media.

Donor plants

The genotype and the physiological condition of donor plants as well as the developmental stage are crucial for haploid induction (Obert *et al.*, 2004).

Physiological condition of donor plants

Influence of donor plant ontogenetic stage on anther culture was demonstrated in Bretón and Lassaga (1999). Better responsivity of anthers collected from plants in the ontogenic phase bud development compared with anthers from plants in the late flowering phase was observed by Tejklová (1996), when callogenesis in anthers from the first harvest reached 9% and from the second one, two weeks later, only 0.7%. Callus induction was higher in anthers harvested from donor plants at the beginning of the flowering period than from anthers harvested later.

Lassaga *et al.* (2010) used anthers from plants in ontogenetic stage fully flowering for high anther callus production, but very low bud regeneration from calli was obtained. Anthers harvested from plants at early flowering stage were more responsive compared to anthers from plants at a later flowering stage (Chen *et al.*, 1998b).

Lower sowing density and additional fertilizing of donor plants caused higher callogenesis in anthers cultured *in vitro* (Tejklová, 1998).

Temperature of donor plants cultivation

Lower temperature in cultivation room for donor plants (14°C in daytime, 8°C at night) supported callogenesis and organogenesis in the anthers (Nichterlein *et al.*, 1991b).

Significant differences in callus induction were noticed when anthers were collected from plants cultivated in the field after cold days in comparison with anthers collected after hot days (Tejklová, 1996). Low temperature probably delays pollen-grain development so that more microspores are in the right development stage for callusing or endogenous conditions in anthers are more convenient for microspore callusing in low temperature.

Lowered cultivation temperature of donor plants for about 4 days before anther collection improved anther responsiveness in experiments in which donor plants were grown on the medium-long days (Tejklová, 1996).

Donor plants were grown in the same light conditions but either in lower (7–13/13–25°C day/night) or in higher (13–20/25–31°C day/night) temperature. Callogenesis in anthers harvested from donor plants from lower temperature was 7 times higher (Tejklová, 1998).

Precondition of donor plants influenced callogenesis in subsequently isolated anthers (Burbulis *et al.*, 2005). Anthers from donor plants grown at 18/14°C were more responsive than those grown at 22/18° (day/night).

Day length of donor plants cultivation

Nichterlein *et al.* (1991b) cultivated donor plants in a 16-h photoperiod. Tejklová (1992) presented the significant influence of donor-plant cultivation condition on callus induction in the anther culture. The highest callogenesis was obtained in anthers from plants cultivated during the early spring in a greenhouse, the lower frequencies were in anthers from plants cultivated from the end of March to the first half of June in vegetation halls and the lowest frequencies were in anthers from plants cultivated from the second half of April to the beginning of July in the field. In the next experiment, callogenesis in anthers harvested in the field was higher than in anthers from the greenhouse (Tejklová, 1996). Both experiments have in common that the conditions for growing donor plants differed in temperature and day-length. High temperature and long photoperiod of donor plants growing both decreased anther responsiveness in the anther culture. Including a low-temperature period in culture system for donor plants prior to anther collection did not increase the callogenesis in anthers probably because the plants were grown on a long day (Tejklová, 1996). Day-length of donor plants notably influenced callogenesis and organogenesis in cultured anthers. Anthers harvested from plants grown in short day (8 h) were more responsive compared to anthers from plants grown on a 16-h day (Tejklová, 1998).

Study on the influence of photoperiod shortening for donor plants on flax anther culture confirmed the importance of this factor. Anthers from plants grown on the short day (8-h photoperiod) were significantly more responsive than anthers from the long day (16-h photoperiod) (Rakouský *et al*., 2001).

Rutkowska-Krause *et al.* (2004) cultivated donor plants either in the greenhouse (winter vegetation period; 16-h day) or in the vegetation hall (summer period; about 16-h day). Temperature was not defined. Statistical analysis showed that differences in callogenesis and organogenesis between those two periods were not significant.

Colchicine treatment

In some experiments, colchicine treatment applied on donor plants before anther collection improved organogenesis in anther calli (Rakouský *et al*., 2001).

Bud size

Mononuclear microspores were observed in the flax flower buds with a diameter of 1.32–1.78 mm and with a length of 4.08–4.78 mm, depending on genotype, testing place and year (Poliakov *et al*., 1995), and with diameter of 1.3–1.8 mm and length of 4.0–4.5 mm (Rutkowska-Krause *et al*., 2003, 2004). Lassaga *et al*. (1998) found the higher response in linseed anthers having about 1.5 mm in length. Bud regeneration was achieved only from flower buds with mononuclear microspores but anthers with microspore tetrads or with dinuclei pollen grains produced only calluses in low frequency (Rutkowska-Krause *et al*., 2004). Uninuclear microspores were observed in floral buds of 1.3–2.3 mm diameter in European linseed genotypes with larger flowers (Nichterlein, 2003).

Anther pretreatment

Temperature

Low-temperature (4°C) pretreatment on inflorescences and isolated anthers of flax did not seem to lead to the increase in percentage of callusing and plant regenerating anthers (Poliakov *et al*., 1995; Rogalska and Rutkowska-Krause, 1994). Later Rutkowska-Krause *et al*. (1995) reported that refrigerator-temperature pretreatment on inflorescences before anther extraction reduced callogenesis induction in the anthers. Cold pretreatment on extracted anthers cultured on medium improved responses of anthers only when 12-h pretreatment was used on anthers of variety Belinka.

Cold pretreatment of isolated anthers (4°C, 1–5 days) inhibited anther callogenesis and storage of flower buds (4°C, 2–8 days) did not increase anther callogenesis (Tejklová, 1996). Three days' pretreatment of flower buds in the dark at 4°C significantly lowered the number of callus inducing anthers (Kurt and Evans, 1998). Also Tejklová (1998) detected that pretreatment on isolated anthers 1–5 days/4°C decreased callogenesis in anthers. Pretreatment 16–48 h at 38°C increased callus induction in anthers insignificantly. Temperature 41°C applied for any tested period decreased callus induction in anthers compared to control without any temperature pretreatment. Fourteen days' low-temperature (4°C) pretreatment decreased callogenesis and organogenesis in anther culture of cultivar Nike compared to non-treated anthers (Rutkowska-Krause *et al*., 2003).

Chen *et al*. (1998b) found out that 1-day culture in 35°C in the dark improved overall efficiency of regeneration in one F1 linseed hybrid but not in the second tested hybrid. Three days' pretreatment in different temperatures (6, 28 and 36°C) influenced irregularly anther callus induction in four genotypes and their hybrids (Bretón, 2001). Obert *et al.* (2003, 2004) presented callus induction in the anthers improved by cold pretreatment (7 days/8°C) but only in some genotypes. Dramatic increase of induction rate in flax anther culture when anthers were pretreated for 3 days at 4°C and afterwards kept for 1 day in 35°C was declared in Preťová *et al.* (2006).

Position of cultured anthers on culture medium

Anthers immersed into agar culture medium showed lower callus production than anthers placed on medium surface in all variants (Tejklová, 1996). Anther orientation on the culture medium surface showed no significant effect on callogenesis and regeneration (Chen *et al.*, 1998b).

Light condition

Callogenesis in anthers cultivated four weeks after isolation in the dark in the same culture temperature was higher than in anthers cultivated in the 16-h photoperiod (Tejklová, 1996) but organogenesis in those calli was low. One-week culture of anthers in the dark was enough for better callus induction (Tejklová, 1998) and did not decrease the regeneration from anther calli in next subculture.

Two-weeks' culture of anthers in darkness after isolation significantly increased callus induction and bud regeneration in the anther culture (Rutkowska-Krause *et al*., 2004). Cultivation of anthers in the dark during the first month of culture improved callus induction in anthers (Rutkowska-Krause *et al.*, 1995).

Osmotic pressure

Preculture of anthers on medium containing 15% sucrose for 2–7 days before transfer to the same medium with 6% sucrose increased the frequency of microspore-derived shoot regeneration and reduced frequency of shoot regeneration from somatic tissues in the anther culture (Chen and Dribnenki, 2004). The same results were obtained if 6% sucrose and 9% non-metabolize osmoticum polyethylene glycol were used instead of 15% sucrose.

Culture medium

Culture medium consistency

Overall lower frequency of callogenesis in anthers was recorded on a liquid culture medium compared to a solidified one (Kurt and Evans, 1998).

Basal medium – macro-micronutrients

Nichterlein *et al.* (1989a) tested two basal media. MS with reduced ammonium nitrate to 10% was more convenient for the flax anther culture.

Nichterlein *et al.* (1991a, 1991b) tested different basal media with different levels of amino acids, growth regulators and carbohydrates, and with different pH as well. Effect of culture media combination on bud induction in anthers calli was obvious. Medium A22 with lowered level of ammonium nitrate, completed with naphthalene acetic acid (NAA), 6-benzylaminopurine (BAP) and sucrose for callus induction, and P20 with standard ammonium nitrate supplemented with zeatin (1 $mg.L^{-1}$) and maltose for bud regeneration were the best medium combination. Anthers from Atalante gives 31% bud regeneration in such culture conditions.

Sun *et al.* (1991) presented large experiments in which influence of macronutrient levels in different combinations on anther callus induction and shoot regeneration was studied. An optimal combination for callogenesis and following organogenesis was B5 medium with 2 $mg.L^{-1}$ (2 mg/L) kinetin and 2 $mg.L^{-1}$ IAA modified by double dose of potassium nitrate and calcium chloride, but although it was the best combination, frequency of organogenesis in isolated anthers was only 0.24%.

Significant influence of culture medium on callus induction in cultured anthers was also noticed by Tejklová (1992). The highest callogenesis (up to 18%) was reached in anthers on modified MS medium (10% NH_4NO_3) supplemented with 1.11 $mg.L^{-1}$ 2,4-D and 0.21 $mg.L^{-1}$ kinetin and nil callogenesis was obtained on the medium N6 with 1 $mg.L^{-1}$ BAP. Besides the influence of nitrogen level and form, influence of growth regulators, however, must be considered in this experiment. Effect of ammonium nitrate concentration on

the flax anther culture was studied in Tejklová (1998). In the experiment with two levels of ammonium nitrate in callus-induction medium (10 or 100% of NH_4NO_3 in basal MS medium) and the same two levels of ammonium nitrate in bud-regeneration medium, combination of 10% in callus-induction medium with 100% in bud-regeneration medium was the most successful. Bud regeneration was 4% per isolated anthers and 66.67% per anther calli.

Preťová *et al.* (2001) presented sporadic callogenesis on media N6 and N&N. Obert *et al.* (2004) induced higher callus formation on media Mo, N6, MS and N&N supplemented with various combinations of growth regulators. Callus formation was the highest according to genotype.

Influence of $CuSO_4$ on callus induction in anthers was studied in Vilamonte and Bretón (2004) and Bretón *et al.* (2004). Higher concentration (10 mM) of this salt in culture medium improved callogenesis in anthers compared to 1 mM $CuSO_4$.

Grauda *et al.* (2005) compared two basal media for flax anthers of four genotypes. Medium NLN 82 supplemented with 1 $mg.L^{-1}$ BAP and 0.05 $mg.L^{-1}$ NAA and 6% sucrose was better than MS medium with 1 $mg.L^{-1}$ BAP and 2 $mg.L^{-1}$ 2,4-D for embryo formation. Embryos formed green regeneration zones after transferring them on the regeneration medium.

Burbulis and Blinstrubiene (2006) obtained callogenesis higher than 30% on medium with reduced ammonium nitrate to 10% of MS basal medium. Anthers were cultured in the dark at 25°C. Bud (or shoot) regeneration was not presented.

Overall efficiency of regeneration was reduced on 2× salts medium while it was increased on 1/4 salts medium, compared to 1× salts medium (international Patent PTC/CA2002/001722, Chen *et al.*, 2003).

Growth regulators – auxins, cytokinins

Influence of four growth regulators on flax anther culture was described in Sun *et al.* (1980). From eight auxins which were added into callus-induction medium, 2,4-D, NAA and Picloram were more favourable for anther callus induction while callus was induced at lowest frequencies when IAA, IBA and TIBA were added into induction medium (Tejklová, 1996). Zeatin was more suitable for callogenesis in anthers and for bud regeneration in anther calli, as well, than BAP (Tejklová, 1998).

MT in callus-induction medium resulted in higher frequency of callogenesis in anthers compared to BAP in the same concentration. When calli from medium with MT were transferred on regeneration medium with either MT or BAP in the same concentrations, bud regeneration was significantly higher on medium with BAP (Tejklová, 1998).

The type of auxin appeared to determine the organogenetic capacity of anther-derived calli. The combination of 2 $mg.L^{-1}$ of 2,4-D with 1 $mg.L^{-1}$ of

BAP significantly increased the overall efficiency of regeneration by 42% (Chen *et al.*, 1998a, 1998c). Regeneration medium with 4.5 μM zeatin had significantly higher percentage of calli forming shoots than the same basal medium with 0.01 thidiazuron (TDZ) (Chen *et al.*, 1998b). Percentage of calli forming shoots increased as the TDZ concentration increased; however, shoots from higher TDZ concentrations were abnormal in phenotype and often died during the transfer into soil.

Soroka (2004) claimed that callus grew and developed better at BAP concentration of 2 $mg.L^{-1}$, compared with 4 and 6 $mg.L^{-1}$ in culture medium. Shoot and root regeneration was low and was not dependent on BAP level. Transfer of explants onto fresh medium stimulated de-differentiation of regenerated structures.

Gonzales and Bretón (2005) studied the influence of different levels of kinetin on callus induction in anthers. 2 $mg.L^{-1}$ of Kin was the best. Substitution of zeatin with kinetin in higher concentrations did not improve organogenesis in the anther calli (Gonzales *et al.*, 2005).

Vitamins

Medium containing 10 $mg.L^{-1}$ of thiamine had a significantly higher overall efficiency of shoot regeneration than the medium with 0.1 $mg.L^{-1}$ (Chen *et al.*, 1998a, 1998c). Similarly 1 $mg.L^{-1}$ of thiamine in callus induction medium slightly decreased callus induction and significantly improved bud regeneration compared to standard (0.1 $mg.L^{-1}$) level of thiamine (Tejklová *et al.*, 2008).

Carbohydrates

There were no significant differences in callus induction and bud regeneration between variants of 6 or 10% sucrose in medium for callus induction (Tejklová, 1996). Replacement of half amount (30 g) of sucrose in callus-induction medium with the same amount of glucose decreased the callogenesis in anthers while replacement of half amount of sucrose with the same amount of maltose increased anther calli production (Tejklová, 1998).

Carbohydrates in the induction medium had a significant effect on both callus induction and shoot regeneration. Medium for callus induction containing 6% or 9% maltose showed the most overall efficiency of regeneration among five levels of maltose evaluated (Chen *et al.*, 1998a). Level of sucrose in culture medium affected proportion of microspore-originated and somatic cell-originated regenerants. Frequency of spontaneous chromosome doubling appeared to depend on the sucrose concentration in the induction medium. In comparison with sucrose, lactose was found to increase callus induction from anthers; however, the effect of lactose

on shoot regeneration appeared to be genotype-dependent (Chen and Dribnenki, 2002).

Rutkowska-Krause *et al.* (2004) described the highest callus induction on medium with lower level of sucrose (3%) or sucrose combined with glucose (5.5% of each); maltose was the worst carbohydrate. An increasing level of sucrose from 6 to 9% increased callus induction in linseed varieties Lirina and Barbara (Burbulis *et al.*, 2005) but significantly reduced frequency of responding anthers in their hybrids (Burbulis and Blinstrubiene, 2006). Bud (or shoot) regeneration was not presented.

Temperature and light conditions of anthers

Culture of anthers at 35°C for one day prior to transfer to 25°C in darkness significantly increased the overall efficiency of regeneration. Improved efficiency was attributed to the increased organogenetic capacity of anther-derived calli (Chen *et al.*, 1998b).

Callogenesis was higher in anthers cultured in the dark but organogenesis was higher in anther calli developed on a 16-h photoperiod (Tejklová, 1998). A dark period for 1 week was sufficient for callogenesis improvement (Tejklová, 1998).

Lassaga *et al.* (2010) incubated anthers for 10 days in darkness at 27°C and then in 16-h photoperiod and 25°C. After 25–30 days, frequency of anthers producing green calli was 17–78%, according to genotype. Frequency of anthers with green callus was 41% in cultivar Atalante. However, frequency of shoot regeneration was very low.

Conditions for higher callus induction decreased shoot regeneration

Conditions for higher callus induction were often connected with lower shoot regeneration as demonstrated in the previous item.

The highest shoot regeneration rate was found in cross 2 (27.5%), which also showed the lowest callus-formation rate (Friedt *et al.*, 1995).

Higher bud regeneration related to calli was detected in variant with lower callus induction (Tejklová, 1996, 1998).

High content of auxins (NAA, IAA) in initial medium induced higher callus production but there was no bud regeneration in the calli (Rutkowska-Krause *et al.*, 2004).

Shoot production and rooting

Lower growth regulator levels (0.02 $mg.L^{-1}$ BAP plus 0.001 $mg.L^{-1}$ BAP) were better for shoot production (Rutkowska-Krause *et al.*, 2003). Culture medium for shoot-production in the callus explants producing buds with lower growth regulator levels (0.02 $mg.L^{-1}$ BAP, 0.001 $mg.L^{-1}$ NAA) was

more productive than medium with higher levels both of them (1 mg.L^{-1} BAP, 0.05 mg.L^{-1} NAA) (Rutkowska-Krause *et al.*, 2004).

The highest shoot rooting was detected on half-strength basal SH2 medium (Rutkowska-Krause *et al.*, 2003). Lower concentrations of sucrose and glucose were more suitable for rooting of cut shoots. Optimal medium for shoot rooting was half-strength SH2 medium containing 10 g sucrose without or with 10 g glucose (Rutkowska-Krause *et al.*, 2003, 2004). Chen *et al.* (2003) noted that medium for shoot regeneration and elongation containing 1% sucrose produced longer and more vigorous shoots than the same medium containing lower or higher concentrations of sucrose.

Tejklová and Rakouský (2005) improved rooting frequency and percentage of more viable rooted plantlets by Na_2MoO_4 enhancement in rooting medium.

Cytological analysis

Flow-cytometric analysis of calli developed in the anther culture demonstrated haploid, diploid, tetraploid, mixoploid (di- and tetraploid or tetra- and octoploid) cells after 3 months of culture (Rutkowska-Krause *et al.*, 1996).

Rutkowska-Krause *et al.* (2001b) described ploidy in callus cells 2*n* and 4*n* after 3-months culture, and 2*n*, 4*n* and 8*n* after 6-months culture *in vitro*.

Regenerated plants were diploid (mostly) or haploid (Nichterlein *et al.*, 1991b). Microspore origin of plants regenerated from F1 was proved cytologically (haploid plants) or by monitoring of the homogeneity of progeny (in spontaneous DH lines) (Friedt *et al.*, 1995). Up to 50% of anther culture derived diploid plants originated from the somatic tissues of anthers which was demonstrated by segregation in their offspring.

Chen *et al.* (1998c, 1998d, 2001) determined the microspore origin of anther culture derived plants using inter-simple sequence repeat (ISSR) and randomly amplified polymorphic DNA (RAPD) markers. These molecular methods can differentiate plants originated from different microspores in one anther. About 80% of the regenerated plants originated from microspores and 60% of them were doubled haploids (Chen *et al.*, 1998b).

Segregation in studied traits (colour of corolla, filaments and seeds) was found in 5 offspring from 13 diploid anther regenerants tested (Rakouský *et al.*, 2001). That means 65.5% of regenerants were derived from microspores and 35.5% from the anther somatic cells.

Bartošová *et al.* (2005) demonstrated homozygosity, somaclonal variation and epigenetic modifications on the level of isozyme spectra of acid phosphatase and peroxidase in plants regenerated from microspores.

Anther culture applications in flax breeding

The importance of this method for flax breeding is highlighted in all contributions on anther culture of flax. The main benefit is to shorten the breeding period. Another application is outlined in the selection at the haploid level and mutation induction systems.

Sun *et al*. (1980) and Nichterlein *et al.* (1989b) outlined the first use of anther culture in flax breeding. Marshall (1992) described the anther culture as an adjunct to classical flax breeding techniques for haploid production.

Friedt *et al.* (1995) regenerated doubled-haploid lines from hybrids from intraspecific crossing with the aim of raising alpha-linolenic content in linseed. They used 6679 anthers of the F1 donor plants of the three crosses. Callogenesis reached on average 20.6% and shoot regeneration 4.6% per isolated anthers. Finally 39 doubled-haploid lines were tested from which 4 lines produced more than 60% alpha-linolenic acid in fat, 50% lines showed a fat content superior to that of their cross-parents. Crosses were carried out in 1991, evaluation of doubled-haploid lines in field tests were carried out in 1994. Classical breeding (pedigree selections or bulk breeding) of autogamic crop takes about 15 years. These numbers clearly demonstrate a benefit of anther culture in flax.

Analysis of the progenies of plants regenerated from anthers showed that the doubled haploids (anther lines) are worse in agronomical traits than the initial variety but progenies in following generations in some cases exceeded the initial variety (Poliakov *et al.*, 1994). Some anther lines were more resistant to Fusarium sp. or completely resistant to *Melampsora lini* (Poliakov *et al.*, 1994). The number of regenerants obtained in the anther culture significantly surpassed initial cultivars in the complex traits (Poliakov *et al*., 1998). Anther line BTL-10 was selected as the best genotype available in new flax variety establishment. On the other hand, Steiss *et al.* (1998) described pedigree lines showing higher performance than simultaneously developed DH linseed lines.

Straathof (1998) highlighted the importance of the anther culture for *in vitro* selection system establishment. The main advantage of using haploids in linseed breeding is the rapid development of completely homozygous lines within one generation and efficient means of genotypic selection (Chen *et al.*, 1998c).

Rutkowska-Krause *et al*. (1998) evaluated anther lines derived from F2 generation. Some regenerated lines segregated plants with white and/or blue petals. This could be due to mutation induction during the period of plant regeneration (Rutkowska-Krause *et al.*, 1998).

Anther lines showed significantly ($P = 0.01$) lower variability of total plant length, technical length, plant weight and seed weight of individual

plants compared to initial material pedigree line Su 45/85, and flax variety Texa, as well (Tejklová and Pavelek, 2000). The anther culture is available for stabilization of breeding materials in flax.

Two rust resistance genes were inherited in expected Mendelian ratios in microspore derived populations developed via anther culture (Chen *et al.*, 2001). Rutkowska-Krause *et al.* (2001a) observed gametoclonal variation in plants regenerated from anthers. Changes of morphological traits and resistance to Fusarium wilt were described. Some perspective lines with higher resistance to Fusarium wilt were obtained. Rutkowska-Krause *et al.* (2003) described method of flax Alba 2 development using the anther culture techniques. Tejklová *et al.* (2003, 2008) and Tejklová (2004) presented linseed anther lines overcoming the control varieties in seed yield and resistance to complex diseases in field trials. One of them, AGT 997/05, is going to be registered in CR under the name Raciol.

The first results with radiation of isolated flax anthers by gamma ray for mutations induction were published in Song *et al.* (2004). Suitable gamma ray dosage was determined.

Conclusions

Isolation of parts of the flax plant (hypocotyl, cotyledon, part of stem, leaf, anther) breaks the plant integrity causing plant tissue de-differentiation and growth of callus tissue in controlled conditions. Growth regulators are known to be the main factors controlling callogenesis and organogenesis in plants. Balance of growth regulators is apparently very fragile in flax. Markedly lower levels of growth regulators (auxins and cytokinins, as well) compared, for example, to levels used in multiple-shoot culture in pea, are able to induce tissue de-differentiation and intensive callogenesis in explants. Differentiation of meristems in callus tissue and bud development from them is very hard to obtain. Probably the very high sensitivity of flax tissues to culture conditions is the reason why direct regeneration in flax has not been obtained yet. Burbulis and Blinstrubiene (2007a) assumed that differences in morphogenetic reaction of different linseed flax genotypes could be determined by the balance of endogenous hormones. Tejklová (1996) described flax variety Duferin as a non-responsive genotype in one experiment but it was responsive in the next experiment. Balance of endogenous hormones could be influenced by many factors; however, there has not yet been any study in this field of research. It could be interesting to detect, for example, growth regulator levels in shooting and non-shooting calli in flax, correlation between culture temperature of donor plants and endogenous hormones in buds or in anthers, and many other factors.

For obtaining plants originated from microspores, the interplay of multiple factors is needed: genotype, physiological condition of donor plant, developmental stage of microspores, desired sequence of changes in culture conditions, etc. However, it is possible that there are other factors influencing the outcome of anther culture, which have not been discovered yet. It is necessary to continue in experimental research for optimal factor combination allowing plant regeneration from microspores in the anther culture in the large breeding material at this time. Nevertheless, the method of anther culture for haploid production corresponding to the current level of knowledge has already been drafted and may be possible in breeding of flax.

Literature

In the following subsections, published articles of scientists and researchers working in European as well as world scientific teams dealing with flax and linseed are cited.

Summarizing publications (reviews)

Summarizing publications with chapters on flax anther culture include: Bergmann and Friedt (1997), Nichterlein and Horn (2005), Millam *et al*. (2005), Song (2007), Burbulis *et al.* (2007a), Preťová *et al.* (2006, 2007), Obert *et al*. (2009) and Song (2009).

Patents

Doubled haploid production is considered a very remarkable method for flax breeding and it has been patented (US Patent 7297838, international Patent PTC/CA2002/001722).

Method descriptions

Though plant regeneration was obtained from different flax and linseed varieties, the frequency of callogenesis in anthers and bud regeneration in anther calli was irregular and usually too low to be used as a routine breeding method. However, the work of Chen *et al*. (1998c) presented 30% overall efficiency of plant regeneration from the anther culture under optimal conditions and this result is very promising for routine use of this method in flax and linseed breeding.

The complete protocol for anther culture in linseed (and for flax, too) is described in Nichterlein (2003), Lassaga *et al.* (2004) and Rutkowska-Krause *et al*. (2004).

The optimized method of anther culture in flax is detailed in Tejklová (2008). The English version is summarized in Tables 13.7 and 13.8. Photographs of the individual steps in the method are shown in Figs 13.3 to 13.14.

Table 13.7 Protocol of anther culture in flax and linseed according to Tejklova (2008)

Donor plant growing	Donor plants for anther collection are grown, if possible, at lower temperatures (daily minimum temperature of 5–10°C, maximum daily temperature of 15–20°C). If the plants are grown in vegetation containers, it is recommended to use basic fertilizer before planting: 0.857 g NH_4NO_3 + 1.717 g $Ca(H_2PO_4)_2.H_2O$ + 1.665 g K_2SO_4 per container with a capacity of 5 L. The sowing density is 3 × 3 cm. During the phase of rapid growth – additional fertilizing should occur once every 14 days with full fertilizer, such as Super Wuxal (AGROBIO, Opava) according to instructions. Microspore regeneration ability is increased by growing donor plants on short days, so it is recommended for the cultivation of flax in field conditions, when plants begin to bloom in mid-June, to shorten the light period about a week before bud collection for about 8–9 h by covering plants with black canvas cover (Fig. 13.3).
Flower bud collection	Cut the buds with a length of 5–6 mm and a width of about 1.8 mm in linseed and a length of about 4–5 mm and a width of about 1.5 mm in flax (that is: cut the buds of maximal size, when it does not have coloured petals), remove any adjacent leaves (Fig. 13.4a). The most successful regeneration is in the anthers from the first few buds on the plant at the bud development stage.
Bud sterilization	Buds are briefly rinsed with 70% ethanol, briefly rinsed with distilled water, pour 1% B (Bochemie as Bohumin), shake for 3 min., pour chloramine B off, pour fresh solution of chloramine B, again for 3 min on a shaker and finally (from now on in sterile conditions – in a sterile box, with sterile instruments). Pour off chloramine B and rinse buds thoroughly three times with sterile distilled water (distilled water, sterilized in an autoclave for 15 min at 121°C).
Extirpation of anthers	Grasp the flower bud with tweezers, cut off about 1 mm segment of the calyx from the stem-side of the bud with a scalpel (so as not to damage the anthers), push aside sepals and petals and take out the anthers without filaments (Fig. 13.4b). The colour blooming genotypes should have whitish petals. Anthers are put on the surface of agar medium AC (for preparation see Appendix 1) in Petri dishes for callogenesis induction (maximum of 50 anthers in a Petri dish with diameter of 10 cm, with 20 mL of culture medium), close the dish with parafilm (parafilm M, Pechiney, Chicago). Sterilization of buds and isolation of the anthers must be carried out quickly, as anther tissues are negatively influenced by lack of nutrition and the presence of sterilizing agents, which is difficult to completely rinse out of the flower buds.
Callus induction	Extirpated anthers are cultured on AC medium (Table 13.8), one week in the light (16 h photoperiod 22/20°C day/night), then transferred for 1 week in darkness at 21°C and then back to light. In pollen sacs, calli from microspores start to develop, anthers are cracked and calli continue to increase in volume (Fig. 13.5). Callus induction is the highest in the third to fifth week after the anthers extirpation.
Bud regeneration	Transfer green callus 2–5 mm in diameter (Fig. 13.6) to regeneration medium AB (Table 13.8; 5 pcs per 100 mL; Erlenmayer flasks with 25 mL medium) and cultivate them on 16 h photoperiod (22/20°C day/night). Every 3 weeks transfer callus to fresh medium of the same composition, remove necrotic (browning) parts, divide a large callus into pieces with a diameter of 5 mm. A callus may also arise from somatic tissue – from pollen sacs or from filament (Fig. 13.8). Regenerants from callus of such origin are of donor plant genotype and the production of doubled haploids is not therefore possible. Regeneration of buds in the callus (Fig. 13.7) occurs mainly during the first three subcultures on regeneration medium.

(*Continued*)

Table 13.7 Continued

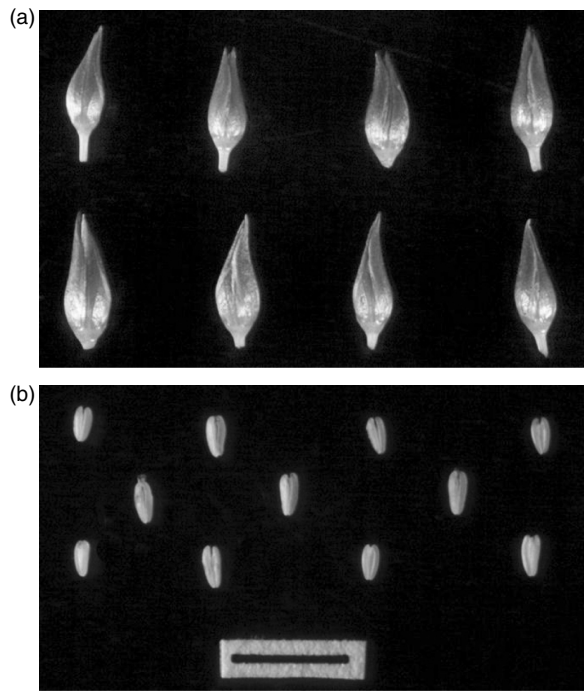
Long-term shoot-tip culture	Developing clusters of buds the size of about 3 mm are maintained on medium L (Table 13.8). Periodically after 2–4 weeks transfer explants to fresh medium, while removing necrotic parts and callus. Use developing shoots for rooting. Cultivation on 16 h day, 22/20°C day/night. Higher temperatures caused stronger callogenesis. In these conditions, new buds are constantly set up and shoots develop (Fig. 13.9).
Culture activation	If culture is maintained so long that the production of new buds and shoots is reduced, it is appropriate to include either 1-week subculture on medium L with BAP increased to 1 μM, or 2 times 3-weeks subcultures on medium L with BAP increased to 0.1 μM, and then again to keep the culture on L medium.
Shoot rooting	2–3 cm long shoots of normal appearance (not vitrified) are successively picked from the long-term multiple shoot culture (Fig. 13.10a), cut off with a sharp scalpel and injected vertically (not diagonally) into rooting R medium (Table 13.8). Often shoots are rooting on MS medium without growth regulators. Shoots usually develop enough roots within 2 weeks (Fig. 13.10b).
Transfer into non-sterile conditions	Soil for regenerated plants should be fine, sandy, with a small proportion of humus: mix of sandy soil, sand and peat in a ratio of 6:2:1, pH around 5.5. Rooted shoots without callus on the bases must be thoroughly rinsed to remove rest of agar from roots, then immersed for 1 minute into 0.15% Previcur and planted into pots with sterilized soil (1 h in autoclave at 121°C). Transferred plantlets have to be covered with a transparent cover (glass, plastic). Plantlets are cultivated in conditioned chamber (fluorescent tubes, 16 h day, 8 h night, 12 000 lux, 22°C). After enrooting plants (about 7th day after planting), the cover is partly lifted and the next day, when the plants do not wither, it is completely removed (Fig. 13.11).
Growing of plants to maturity	In order to flower, flax and linseed plants need to be grown on long days (15–16 h) and with sufficient light intensity (above 10 000 lux), and adequately watered and fertilized.
Colchicine treatment	Plants regenerated from anther culture, in which haploid chromosome number was determined using flow cytometry or which show signs of haploid (a subtle plant, stomata reaching only two-thirds of the size of control diploid genotype, plant is not setting bolls) are decapitated, the stem is bent, and the cutting end of the stems is immersed in a 1% colchicine solution in 1.5 mL test-tube with about 1 mL of solution. The test-tube is attached to the stem with the foil (Fig. 13.12). After the upper leaves get faded (it takes about 16 h), the test-tube is removed. Branches growing from auxiliary buds near the decapitated stem top are usually dihaploid and set seeds (Fig. 13.13). Treatment with colchicine is not carried out in spontaneous diploids (Fig. 13.14).

Table 13.8 Composition of culture media

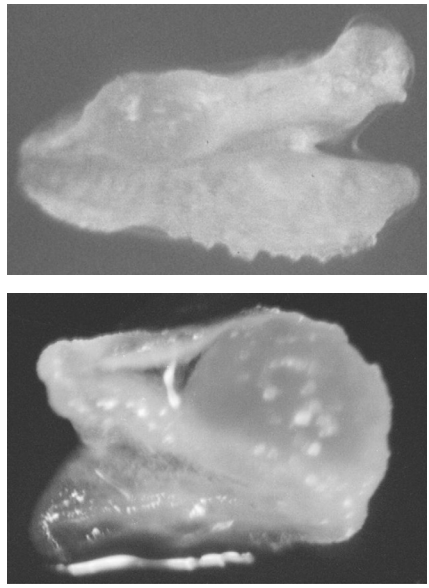
Medium	Macro–microelements	Vitamins ($mg.L^{-1}$)	Growth regulators ($mg.L^{-1}$)	Carbohydrates ($mg.L^{-1}$)	Others ($mg.L^{-1}$)	pH
AC	MS but only 165 $mg.L^{-1}$ NH_4NO_3	Nicotinic acid, 0.5 Pyridoxin HCl, 0.5 Thiamin HCl, 1	NAA, 1 Meta-topolin, 2	Maltose, 30 000 Sucrose, 30 000	myo-Inositol, 100 Glycin, 2 L-glutamin, 750 Agar Difco, 5500	5.5
AB	MS	Nicotinic acid, 0.5 Pyridoxin HCl, 0.5 Thiamin HCl, 1	BAP, 1	Sucrose, 30 000	myo-Inositol, 100 Glycin, 2 L-glutamin, 375 Agar Difco, 5500	5.8
L	MS	MS	NAA, 0.005 µM BAP, 0.050 µM	Sucrose, 40 000	myo-Inositol, 100 Glycin, 2 Agar Difco, 5500	5.5
R	MS	MS	NAA, 0.005 µM	Sucrose, 40 000	myo-Inositol, 100 Glycin, 2 Agar Difco, 5000	5.5

AC, medium for callogenesis in anthers; AB, medium for bud regeneration in anther calli; L, long-term shoot tip culture; R, rhizogenesis; MS, medium according to Murashige and Skoog (1962); NAA, naphthyl acetic acid; BAP, 6-benzylaminopurine.

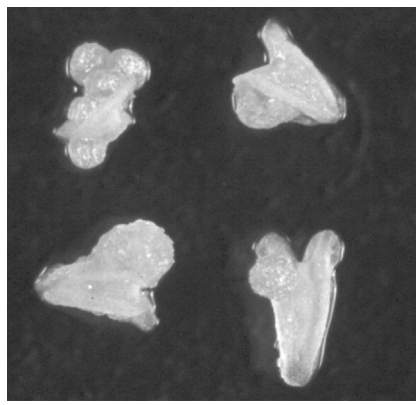
13.3 Day-shortening for donor plants with black nonwoven textile.

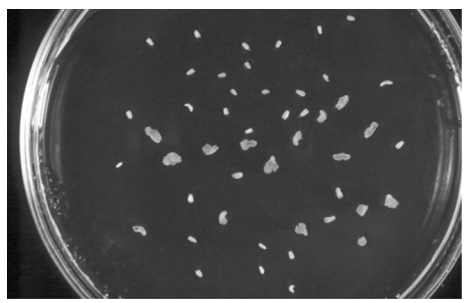


13.4 Buds ready for sterilization (a) and anthers isolated from flower buds (b). Bar = 5 mm.

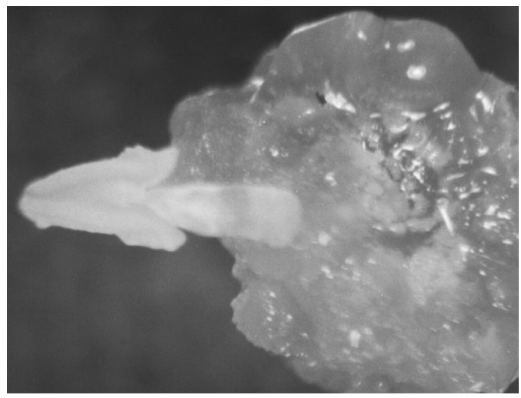


13.5 Callus development from microspore on induction medium.

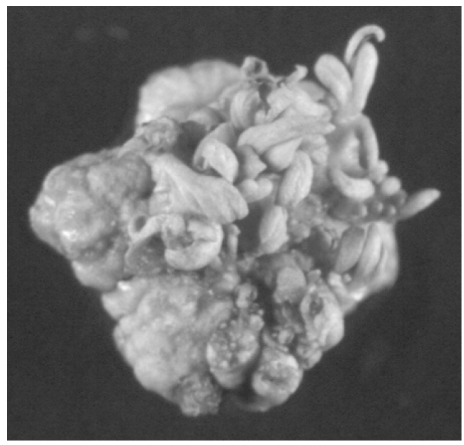




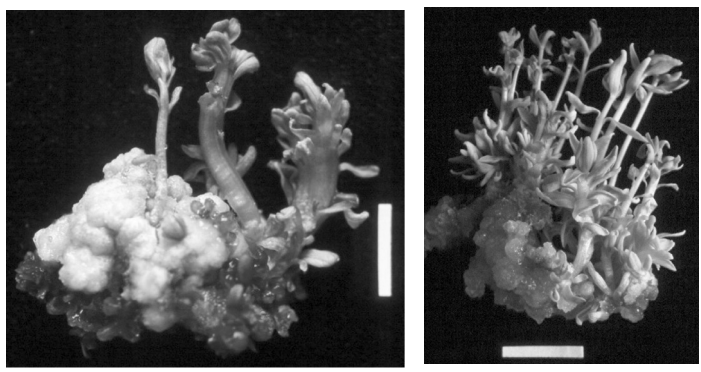
13.6 Calli suitable for transfer to regeneration medium.



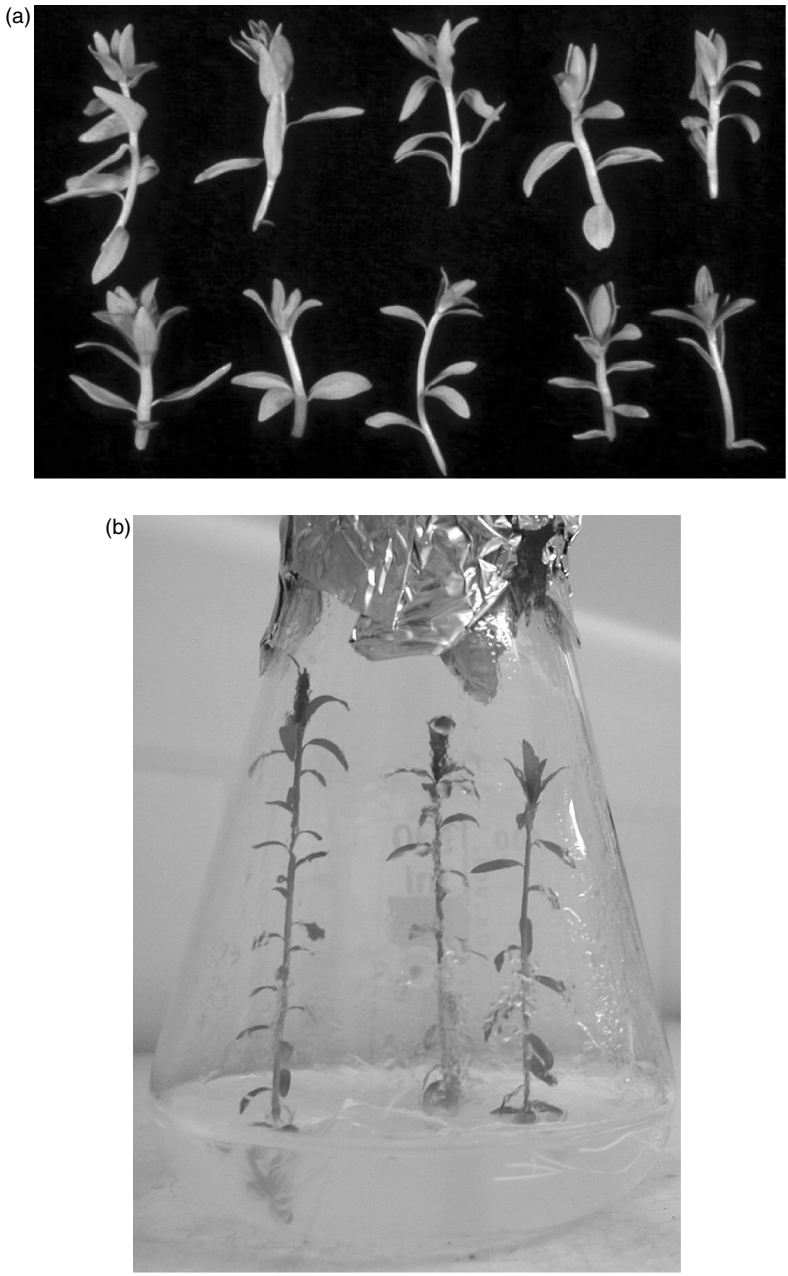
13.7 Callogenesis on filament. Callus originated in somatic cell.



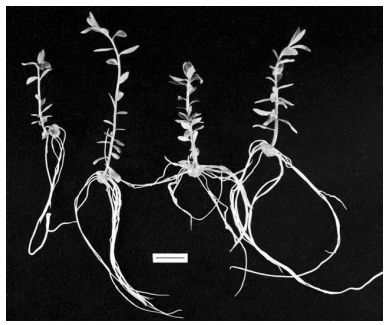
13.8 Bud regeneration in anther callus.

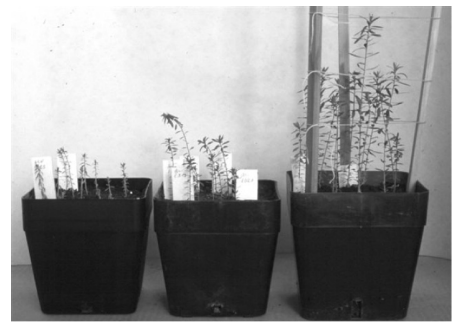


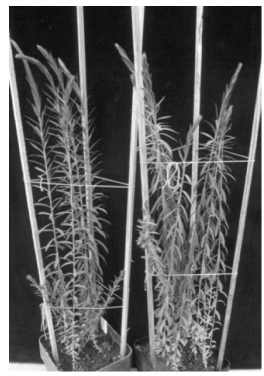
13.9 Shoot development from regenerated buds. Bar = 1 cm.



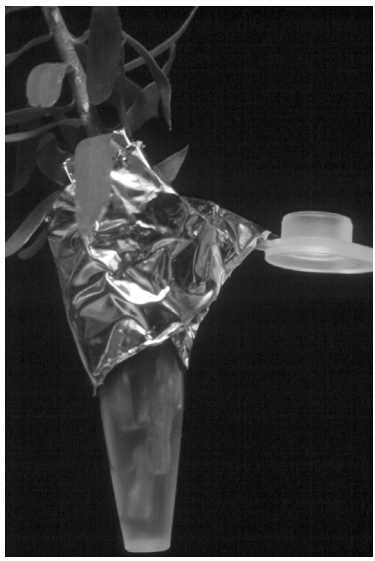
13.10 Cut shoots ready for rooting (a) and rooted shoot after 2 weeks on medium R (b).

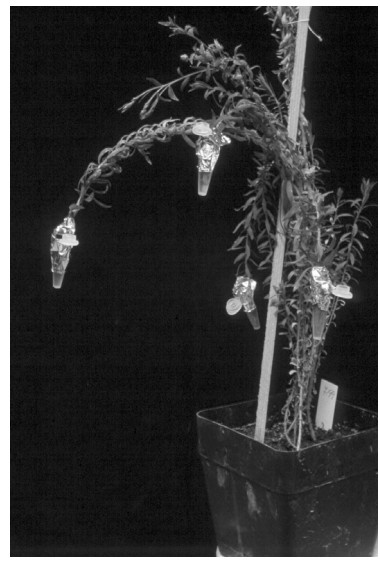






13.11 Rooted shoots are transplanted into soil to grow into flowering and set the seeds.





13.12 Colchicine treatment.

13.13 Dihaploid branches developed after colchicine treatment.

13.14 Haploid (left) and spontaneously diploid (right) regenerant.

13.4 Modern methods in flax and linseed breeding

13.4.1 DNA molecular methods

Exploitation and characterization of flax genetic resources and evaluation of flax genetic variability are very important for flax germplasm management and breeding. There are different ways to evaluate flax genetic variation, such as morphological characteristics, isozymes and molecular markers. Morphological characters tend to be more quantitative and environmentally dependent while isozymes are limited in numbers. DNA markers' abundance, environmental intensivity and non-tissue specific characteristics

are some of their advantages. They are useful for variety identification and evaluation of DNA variation. Different molecular markers including random amplified polymorphic DNA (RAPD), restriction fragment length polymorphism (RFLP), AFLP and SSR have been developed to analyse flax genetic diversity. SSR markers are still very limited in flax: only 11 and 28 SSR markers have been reported in two independent variety identification studies. The first comprehensive study on the development and analysis of a large set of SSR markers in flax is from Cloutier (2009). The 635 alleles detected by the 275 polymorphic EST-SSRs were used to study the genetic relationship of 23 flax accessions. Four major clusters and two singletons were observed. Subclusters within the main clusters correlated with the pedigree relationship amongst accessions. The expressed sequence tags - signature sequence tags (EST-SSTs) developed herein represent the first large-scale development of SSR markers in flax. They have potential to be used for the development of genetic and physical maps, quantitative trait loci mapping, genetic diversity studies, association mapping and fingerprinting cultivars.

Fu (2005) assessed the geographic patterns of flax variability in a world collection of cultivated flax by random amplified polymorphic DNA (RAPD) markers. Sixteen RAPD primers were applied to screen 2727 flax accessions representing 63 countries and one group of unknown origin, and 149 RAPD bends were scored for each accession. Grouping the accessions into 12 major regions explained 8.2% of RAPD variation. Accessions from East Asia and European regions were most diverse, but accessions from the regions of the Indian subcontinent and Africa were most distinct. Accessions from the West Asia region were genetically more related to those from the Africa region and less to those from the Indian subcontinent region. These findings are significant for understanding flax domestication and also are useful in classifying intraspecific diversity of cultivated flax, establishing a core subset of the flax collection, and exploring new sources of genes for flax improvement.

To characterize germplasm at the DNA level, a variety of molecular techniques are currently available. Compared to alternative genetic marker systems, AFLPs are generally considered relatively powerful in germplasm analysis because of the high number of markers that can be generated per analysis. In the study from van Treuren (2004), AFLPs have been used to identify redundant germplasm in the flax collection of the Centre for Genetic Resources, the Netherlands (CGN). Pairwise comparison of accessions was performed by ANOVA in order to identify redundant germplasm. Stepwise bulking of accessions until all remaining accessions were significantly different showed that the 29 accessions of breeder's lines could be reduced to 14. Only a small negative effect of this bulking approach on the among-population component of variance was observed, showing a reduction of 2.6%. This result is discussed in relation to improving the efficiency of collection management.

13.4.2 Flax transformation

Different methods to introduce genes into flax have been developed. *Agrobacterium*-mediated transformation is the most used procedure in flax transformation for its simplicity and smoothness. Flax, like most dicotyledonous crop species, is amenable to gene transfer via *Agrobacterium* (Hepburn *et al*., 1983). Flax cells are easily transformed with *Agrobacterium tumefaciens*, and easily grown and require an elaborate inoculation/selection/regeneration procedure (Jordan and McHughen, 1988a).

Most regenerable tissue of flax seems to be the hypocotyl. The efficiency of recovery of transgenic plants is very low: most shoots regenerating from the inoculated hypocotyls are not transgenic. Inoculating hypocotyls without removing the epidermis or providing a preculture period results in effective cellular transformation and shoot regeneration, but the shoots are all non-transgenic, presumably arising from non-transformed cells protected in the callus by nearby transformed ones (Jordan and McHughen, 1988b; McHughen *et al*., 1989).

To enhance transformation efficiency, an improved procedure for production of flax plant was developed (Dong and McHughen, 1992). An efficient and reproducible transformation procedure for flax was developed through optimization of precondition treatment of the hypocotyl, co-cultivation duration and selection scheme. Hypocotyls from 5-day-old seedlings were precultured for 6 days, followed by peeling (epidermis removal) and 3-day postpeeling preculture prior to inoculation with *Agrobacterium*. However, Mlynárová *et al*. (1994) published other results. In this procedure, neither the preculturing period nor the epidermis removal was used, suggesting these procedures are not essential for high transformation efficiencies. The data show that the differences in transformation efficiencies may be due to the particular flax cultivar used for transformation or to the *Agrobacterium* strain used.

To enhance transformation efficiency, Beranová *et al*. (2008) used the SAAT method (sonication assisted *Agrobacterium*-mediated transformation). The results showed that treatment with ultrasound facilitates an enhanced uptake of plasmid DNA into the cells of flax hypocotyls and cotyledons and that its efficiency depends on the duration of the treatment and frequency used.

As an alternative to the *Agrobacterium*-mediated transformation, particle bombardment can be used as a method for plant cell transformation. This transformation procedure involves the delivery of gold or tungsten microprojectiles coated with plasmid DNA which are then shot into plant cells. Wijayanto and McHughen (1999) documented a successful biolistic process to regenerate transgenic linseed flax, where hypocotyls were bombarded and cultured on a standard selection medium. The transformation

efficiency of particle bombardment was lower than in agrobacterial mediated transformation.

Selection of transformed flax plants is based often on resistance to antibiotic kanamycin. The disadvantage of kanamycin as selective agent in flax transformation is a great number of escapes. An alternative in flax transformant selection can be hydromycin B. Work aimed to develop an alternative protocol for selection of transgenic flax is useful for routine evaluation of broad sample numbers (Rakouský *et al*., 1999) and another antibiotic spectinomycin was successfully applied in selection (Bretagne-Sagnard and Chupeau, 1996). In order to use non-antibiotic resistance genes for the production of transgenic plants, the phosphomannose isomerase gene as selectable marker was used in flax transformation by Lambklin *et al*. (2007). They described that the mean transformation efficiency of 3.6% was comparable to that obtained routinely using the nptII selectable marker.

For monitoring gene expression in transgenic tissues, markers such β-glucuronidase (GUS), luciferase (LUC) or β-galactosidase (LazC) are used. The GUS assay is the most useful assay in flax transformation (Dong and McHughen, 1992); however, this assay is destructive. For this purpose (Hraška *et al*., 2006) described in more detail the contribution of GFP as a visual marker to the establishment, evaluation and improvement of transformation procedure for flax plants, which can continue in growth and development without damage of transgenic tissues.

Flax is amenable to transformation by *Agrobacterium tumefaciens* as well as *Agrobacterium rhizogenes*. The first report of the regeneration of flax transformed by *A. rhizogenes* was described by Zhan *et al*. (1988). Transformed plants were obtained from hairy roots induced by *A. rhizogenes*. These results show that the transformation by *A. rhizogenes* can be an alternative to transformation of *A. tumefaciens*.

Linum sp. was among the first crop species to benefit from herbicide resistant construct, as glyphosate (Roundup) resistance, sulfonylurea and glufosinate resistance all were quickly inserted and field tested in commercial linseed flax genotypes. Lorenc-Kukula *et al*. (2007) transformed flax with the aim to improve resistance to Fusarium. The idea was that the increase in the flavonoid content in the transgenic flax plants might be the reason for observed, enhanced antioxidant capacity of those plants. The increased antioxidative properties of transgenic plants may lead to improved resistance to Fusarium. Wróbel *et al.* (2004) report successful transformation of bacterial genes involved in polyhydroxybutyrate synthesis to *L.usitatissimum* plants. This offers a new perspective for environmentally safe production of basic components for modern biodegradable composites. Flax as an industrial crop can be utilized for phytoremediation as well. Flax transformation with heavy metal binding proteins is reviewed in Vrbová *et al*. (2009). However, to date, no transgenic linseed/flax is

permitted to grow for commercial utilization. Only one transgenic linseed has reached registered cultivar status, 'CDC Triffid' (a flax (L. usitatissimum L.) variety tolerant to soil residues of triasulfuron and metsulfuron-methyl), but authorization of the variety was rescinded in Canada in 2001 and cultivation of CDC Triffid flax has since been banned.

13.4.3 GMO flax

Since the commercialization of GM crops 13 years ago, the global area of GM crops has increased from 1.7 to 125 million hectares. Over 13 million farmers grew GM crops in 2008 compared to 12 million in 2007. Three new countries, Burkina Faso (cotton), Egypt (corn) and Bolivia (soy), planted GM crops for the first time, bringing the total number of countries growing GM crops to 25. The top eight GM countries are the USA 62.5 mil. ha/2008 (alfalfa, canola, cotton, corn, soybean, squash, sugar beet, papaya), Argentina 21 mil.ha (cotton, corn, soybean), Brazil 15.8 mil.ha (cotton, corn, soybean), India 7.6 mil.ha (cotton), Canada 7.6 mil.ha (canola, corn, soybean, sugar beet), China 3.8 mil.ha (cotton, papaya, petunia, poplar, sweet pepper, tomato), Paraguay 2.7 mil.ha (soybean) and South Africa 1.8 mil.ha (cotton, corn, soybean). The ISAAA expects that 15 or more countries will plant GM crops from now until 2015, bringing the total number of countries planting GM crops to 40. Asia, Eastern and Southern Africa, West Africa, North Africa and the Middle East are estimated to be the new adopters of GM crops. Countries in Eastern Europe and Latin/Central America may also adopt GM crops. Several new GM crops are expected between now and 2015, one being GM rice. The ISAAA states that pest and disease resistant rice is awaiting approval in China and Golden Rice, which could help curb deficiencies such as Vitamin A, is expected to be available in 2012.

For 13 years, herbicide tolerance has consistently been the dominant trait. In 2008 herbicide tolerant GM crops (soybean, corn, canola, cotton and alfalfa) made up 63% of the global GM market. Stacked double and triple traits occupied 22% of the GM crop area and insect resistant varieties made up the remaining 15%.

GM flax is developed in agronomic traits in the first place. Herbicide tolerance is very important for weed control, as well as fungal resistance of flax for disease resistance, insect resistance for resistance against pests and stress tolerance to adapt to climate and local factors. Quality traits of flax are very interesting as well: modified composition of ingredients (oil composition), and enrichment with health-promoting ingredients, such as flavonoids (antioxidants) and omega-3 fatty acids (by transference of a marine algae to flaxseed, the seeds then contain omega-3 fatty acids EPA and DHA, which

are a feature of fat-laden saltwater fishes). These acids are assumed to have a preventive effect towards hypertension and specific degenerative diseases. Renewable resources are as also important in terms of modified composition (modified elasticity and thermoplastic characteristics of the flaxseed fibre for the synthesis of biological degradable synthetic material), production of pharmaceutical agents (molecular pharming usage of GM flaxseed as a system to produce pharmaceuticals; to date, only experimental) and land reclamation = phytoremediation (heavy metal strained soil) – the plants are modified so that they are able to grow within this soil and can extract heavy metals and then accumulate them within the plant biomass (Vrbová *et al.*, 2009).

Field trials with GM flaxseed have been undertaken in the EU in three applications in three countries (Sweden, Poland, Czech Republic) in 2005–2007. Traits studied in these experiments were oil composition, flavonoids content, elasticity (bioplastics), herbicide tolerance, insect resistance, insect and fungal resistance and heavy metal absorption.

No genetically modified flax is currently grown. A herbicide-resistant GM flax was introduced in 2001, but was soon taken off the market because European importers refused to buy it. In September 2009 it was reported that Canadian flax exports had been contaminated by an unapproved, illegal, genetically modified (GM) variety, known as Triffid. Since linseed derived from GM flax is not authorized in Europe, products containing even minimal amounts cannot be made commercially available. Transgene contamination of flax seed is used widely in the food industry, including bread, and as source of omega-3 fatty acids. On 10 September 2009, EU Rapid Alert System for Food and Feed (RASFF) reported finding an unapproved genetically modified (GM) flax/linseed variety in cereal and bakery products in Germany. In Germany, the GM linseed has been found in at least seven states up to now. Baking ingredients contaminated with the GM linseed had been distributed to 15 German states and exported to other countries by a German company based in Hessen. Linseed is an ingredient in bakedgoods and muesli. Consumption of products containing minute traces of GM linseed does not present a health risk. The GM flax variety FP967 (CDC Triffid) is not authorized for food or feed in the EU; it has tolerance to soil residues of sulfonylurea-based herbicides, and was developed by the Crop Development Centre at the University of Saskatchewan in Canada. Authorization of the variety was rescinded in Canada in 2001. Cultivation of CDC Triffid flax has since been banned. Canada supplies approximately 70% of the total flax/linseed in the EU annually. Because GM flax FP967 is not authorized in the European Union, there is zero tolerance for the variety. That means any raw material or flax/linseed derivative analysed to be positive for FP967 is illegal and not marketable in the EU. The Canadian Grain Commission is investigating how the admixture of the GM flax in linseed products could have occurred.

13.5 Sources of further information and advice

The common catalogue of varieties of agricultural plant species (64 flax and linseed) is downloadable from the Health and Consumers: Plants section of the European Commission website:

http://ec.europa.eu/food/plant/propagation/catalogues/comcat_agri_2008/index_en.htm

There are 132 varieties of flax and linseed originating in the UK, France, Belgium, Czech Republic, the Netherlands, Italy, Lithuania, Poland, Hungary, Austria, Germany, Latvia, Estonia, Finland, Ireland and Denmark. Other useful websites include the following:

www.ecpgr.cgiar.org/epgris/index.htm
www.gmo-compass.org/eng/agri_biotechnology/gmo_planting/
www.inra.fr/Internet/Produits/Lin/index.htm

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13.7 Appendix: abbreviations

AFLP	amplified fragment length polymorphism
ANOVA	analysis of variance
BAP	6-benzylaminopurine
BAZ	Federal Centre for Breeding Research on Cultivated Plants, Braunschweig, Germany
CDC Triffid	a flax (*Linum usitatissimum* L.) variety tolerant to soil residues of triasulfuron and metsulfuron-methyl
CGN	Centre for Genetic Resources, the Netherlands
DH	dihaploids
EST-SST	expressed sequence tag–signature sequence tag
EURISCO	European Plant Genetic Resources Search Catalogue with Passport Data on Ex Situ Collections Maintained in Europe
IAA	indolylacetic acid
INRA	Institut National de la Recherche Agronomigue
IPK	Institute of Plant Genetics and Crop Research, Gatersleben, Germany
ISO	International Organization for Standardization
MS	Murashige and Skoog medium (1962)
NAA	naphthalene acetic acid
RFLP	restriction fragment length polymorphism
SSR	simple sequence repeat
TDZ	thidiazuron
UPOV	International Union for the Protection of New Varieties of Plants

14

Cotton breeding and agro-technology

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Abstract: The concept of modern cotton breeding is to exploit the global gene pool, create novel variation through hybridization and select and stabilize new varieties for local adaptation. The overall goal of most breeding programs is similar, addressing quantity and quality of output, and then regional production constraints. Breeding to improve cotton cultivation involves a systematic method of observation, data collection and statistical analysis of plant performance in a number of different growing environments. This chapter discusses the origin, history and genetic basis of genus *Gossypium*; different breeding methodologies used for cotton improvement; fundamental knowledge of plant growth and development and gives a review of primary breeding targets for cotton.

Key words: breeding, genetics, fiber quality, disease resistance, water use efficiency.

14.1 Introduction

This chapter is intended to provide basic information on concepts of applied cotton breeding. More detailed discussion can be obtained from references and reading material suggested at the end of the chapter. Topics essential to most crop improvement programs including genetic basis, breeding methodology, basic agronomy and physiology, and specific breeding targets are reviewed from the perspective of breeding improved cotton varieties.

Germ plasm (defined as the total genetic variability represented by the available pool of germ cells or seed) improvement was originally achieved in cotton by 'automatic selection', when desirable plants from wild populations were first selected and placed under casual cultivation (Harlan, 1975). Harvest and repeated sowing of selected material was an effective filter for valuable traits and automatic selection was practiced for thousands of years. Yield and traits of interest to the grower and fiber market were targeted around 500 BC, when cotton fiber demand for textiles increased. Breeding to further improve cotton cultivation involves a systematic method of observation, data collection and statistical analysis of plant performance in a number of different growing environments. The success of a breeding program traditionally relies on the skill and experience of the breeder to

recognize even small beneficial changes in plant performance. The concept of modern cotton breeding is to exploit the global gene pool, create novel variation through hybridization and select and stabilize new varieties for local adaptation.

The overall goal of most breeding programs is similar, addressing quantity and quality of output, and then regional production constraints. Most breeding programs seek to increase yield and characteristics associated with the value of the agricultural product. Specific examples in cotton include improvements in fiber length, strength and fineness and white lint color. Regional needs vary around the world and are served by customized adaptive breeding and selection programs. Specific examples in cotton include bacterial blight resistance discovered in South Texas (Bird, 1966) that eventually allowed for cotton cultivation in Australia, and the discovery of a 'storm proof' boll type that allowed cotton cultivation to flourish in West Texas despite damaging winds. Often, sources of resistance that address regional constrains are also genetically associated with poor fiber quality or agronomic characteristics, and breeding must be intensified to improve yield and quality simultaneously with required adaptation.

14.2 Genetic review

Books devoted to information on cotton genetics and genomics give detailed reviews of the origin, history, taxonomy and evolution of *Gossypium* species, as well as available tools and resources. It is important to understand origin and history, genetic diversity and the concepts and consequences of domestication from the applied breeding perspective in order to continue to exploit genetic resources in cotton improvement. Cotton is a self-pollinated crop although natural out-crossing occurs in most environments. Cotton pollen is heavy, and transmitted exclusively by insects. There are both diploid and tetraploid species in cotton. For diploid species, controlled cross-pollination results in maximum genetic segregation in the second filial generation when most individuals are heterozygous and homozygosity is achieved for most characters by the fifth filial generation. The cotton tetraploids contain two complete sets of chromosomes from different species and have been characterized as allopolyploid or amphidiploids. While they are expected to behave genetically as the diploids in breeding, some evidence shows that genetic variability remains after inbreeding (Manning, 1955). Some explanations include selection of phenotypically attractive individual plants that are actually heterozygotes displaying hybrid vigor, more natural out-crossing than expected in a self-pollinated species, a high mutation rate of major genes, irregular pairing of homologous chromosomes from the different genomes and interaction between duplicate genes in the two genomes.

14.2.1 Origin and history

Cotton fabrics have been found dating back to 3500 BC, and their use in civilization is recorded by 500 BC (Hutchinson *et al.*, 1947). The origin of commercial cultivation and modern breeding is intimately tied with the origin of the fiber that is a viable raw material source for textiles. Cotton is grown in some 30 countries, with the top six producing countries being China, India, the USA, Pakistan, Brazil and Uzbekistan (USDA FAS, 2010). Cotton production represents close to half of world textile consumption. The origin as well as the distribution of cultivated cotton is connected with the utilization of the fiber. Fryxell (1979) notes that while 'the cultivated species have been virtually transformed at the hand of man', it is probable that significant lint production and species differentiation took place before man took an interest, and that the four cultivated species domesticated independently, sometime after differentiation (Santhanam and Hutchinson, 1974). The four species known to be cultivated as the world's commercial cotton crop are comprised of two tetraploid, or amphidiploid, species ($2 \times 2n = 52$ chromosomes) known as the 'New World' species; and two diploid species ($2n = 26$) known as the 'Old World' species. The Old World cultivated species include *Gossypium arboreum* and *G. herbaceum* and are minimally grown in India and China. The New World cultivated species include *G. hirsutum* and *G. barbadense* and represent 90% of world cultivation. *G. hirsutum* is the major species grown and includes American 'upland' and Acala varieties. *G. barbadense* is the high quality, long staple species including American Pima, Peruvian Tanguis, Egyptian and Sea Island varieties. These New World species contain two complete sets of chromosomes, the A and D genomes, from different species (Manning, 1955). The A and D genomes are from two different centers of origin, one from the New World and one from the Old World.

One of the most important events in modern cotton development occurred over a million years ago when an Old World species fused with an American species to form the amphidiploids (Dever *et al.*, 2005). All the New World diploids are lint-less, so the lint-bearing genes found in the tetraploids are assumed to have come from the Old World diploid parent. A tremendous range of ecological variation is found among the wild cotton species, but cotton has a perennial growth habit and for the most part, has little botanical use apart from fiber-producing seed. Commercial varieties developed within the domesticated species differ dramatically from their early progenitors in that the plants are compact, efficient and yield abundant fiber (Fig. 14.1). It is important to understand the evolutionary origins of the cultivated species and the spectrum of variation that exists in the genus since this knowledge may be exploited in the improvement of cotton through genetic manipulation and breeding.

(a)

(b)

14.1 Example of diversity in plant type among *Gossypium* species: (a) wild, photoperiodic diploid species; (b) modern, cultivated upland cotton; (c) short-season upland cotton cultivated in wheat stubble and (d) perennial, wild cotton 'tree'. (Photographs courtesy of Monica Sheehan, Texas AgriLife Research, Lubbock.)

(*Continued*)

14.2.2 Genetic variation

Gossypium is a large genus, comprising more than 50 species with new ones still being discovered, which represent a wide range of diversity (Table 14.1). This group of species is thought to come from a single natural lineage,

(c)

(d)

14.1 Continued.

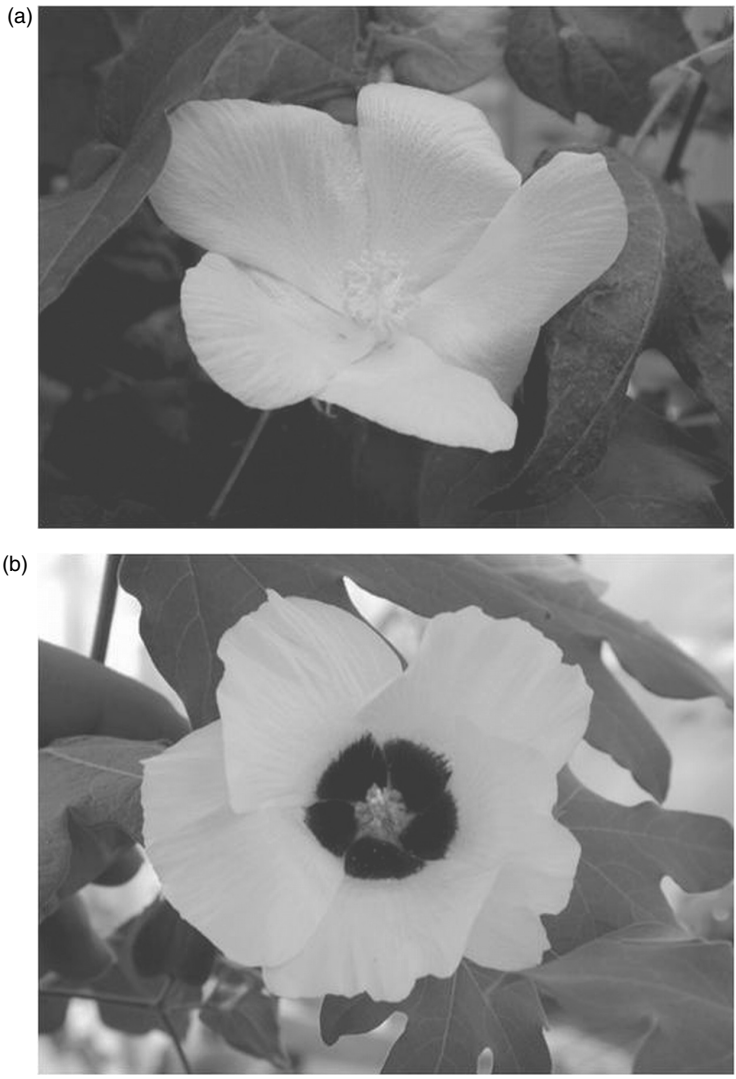
despite their global distribution and morphological and cytogenetic diversity (Seelanan *et al*., 1997). There are four primary centers of diversity in the arid tropics, including in Australia, Africa, Asia and the Americas, in Mexico. The taxonomy has been extensively studied and the classification system is based mostly on morphological and geographical data, although some cytogenetic and molecular data sets are available. The genus as a whole is very diverse (Figs 14.2–14.4) with growth habits ranging from herbaceous

Table 14.1 Identified species in *Gossypium* and centers of origin

African–Asian	American
G. arboreum	*G. thurberi*
G. herbaceum	*G. amourianum*
G. anomalum	*G. harknessii*
G. triphyllum	*G. davidsonii*
G. capitis-virdis	*G. klotschianum*
G. stocksii	*G. aridum*
G. somalense	*G. raimondii*
G. sreysianum	*G. gossypioides*
G. incanum	*G. lobatum*
G. trifurcatum	*G. trilobatum*
G. longicaylx	*G. laxum*
G. trifurcatum	*G. turneri*
G. benidirense	*G. schwendimanii*
G. bricchettii	*G. sp.nov*
G. vollesenii	
G. trifurcatum	
Australian	New World
G. sturtianum	*G. hirsutum*
G. robinsonii	*G. barbadense*
G. bickii	*G. tomentosum*
G. australe	*G. mustelinum*
G. nelsonii	*G. darwinii*
G. anapoides	
G. costulatum	
G. cunninghamii	
G. enthyle	
G. exiguum	
G. londonderriense	
G. marchantii	
G. nobile	
G. pilosum	
G. populifolium	
G. pulchellum	
G. rotundifolium	

Adapted from Wendel *et al.* (2009).

perennials to trees that defoliate during the dry season, flower colors across the spectrum and seed hair from almost non-existent to short, stiff brown fiber to the long, fine white fiber known in commerce (Wendel *et al.*, 1992). This impressive range of diversity has been studied, but rarely exploited in breeding cultivated cotton varieties. Despite difficulty in controlled hybridization and side effects deleterious to commercial cotton variety success, there are several examples of introgressed traits from wild species in breeding programs (McCarty and Percy, 2001).



14.2 Example of diversity in flower type among *Gossypium* species: (a) small, white flower with no petal spot; (b) yellow flower with large petal spot; (c) closed type petals with small petal spot and (d) red flower with dark petal spot. (Photographs courtesy of Monica Sheehan, Texas AgriLife Research, Lubbock.)

(*Continued*)

14.2.3 Domestication

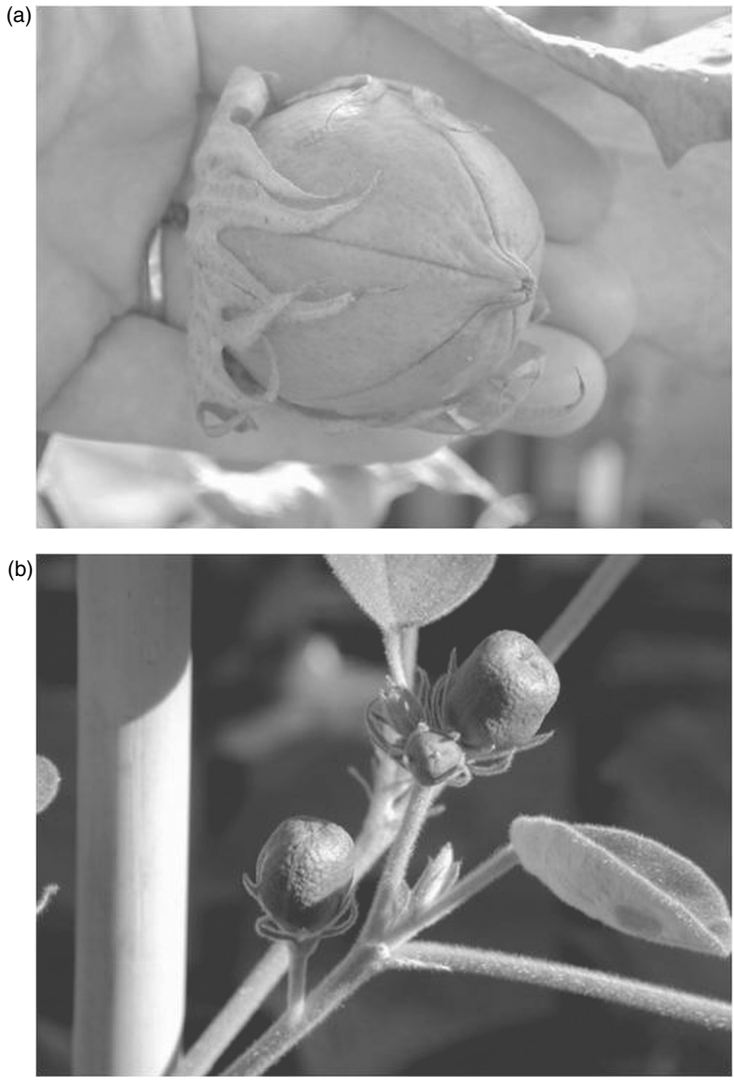
Selection over time for phenotypes adapted for agricultural production changes the population genetic basis as undesirable traits are eliminated and desirable traits retained. Cotton domestication may vary some from other

(c)

(d)

14.2 Continued.

crops because of its perennial growth habit and primary use as a textile product as opposed to food. The fiber itself may have evolved only once, but four separate species were ultimately domesticated independently. A key aspect of domestication is the dispersal of seed; desirable in perennial wild types, but not in cotton varieties developed for production. Cotton fiber is not a selective advantage for seed dispersal, suggesting the lint hairs of commerce evolved under domestication. Two examples of domestication contrary to seed dispersal are boll types that prevent the seed pod from dropping to the ground



14.3 Example of diversity in boll type among *Gossypium* species: (a) normal, cultivated upland 5-lock boll; (b) small, wild, flat-topped pitted boll; (c) small, round boll with striated bracts and (d) deeply pitted boll with hirsute bracts. (Photographs courtesy of Monica Sheehan, Texas AgriLife Research, Lubbock.)

(*Continued*)

after the boll opens and the convoluted, flat ribbon shape of cotton fiber that causes the boll to fluff and allows for cohesiveness necessary in spinning. Seed dormancy common in wild, perennial cotton has been reduced and germination improved so that seed from a previous crop can emerge the next season in seven to ten days at a desired plant density for maximum yield.

(c)

(d)

14.3 Continued.

Productivity and output quality of cultivated cotton varieties is another example of domestication. Increase in boll number per plant and boll size is the most obvious visual result of higher yield in domesticated cotton, but ratio of lint to seed among cotton types is a fairly discrete bimodal distribution with wild cotton peaking at 8–10% lint and domestic varieties ranging from 25 to 45% (Fryxell, 1979). Evidence suggests the percentage of lint in seed cotton was selected early in the domestication process and may have been one of the first traits selected. Lint percentage is correlated with

(a)

(b)

14.4 Example of diversity in fiber development among *Gossypium* species: (a) lintless seed from wild, diploid species; (b) storm-tolerant, cultivated boll with high fiber percentage; (c) cultivated, loose boll type with high fiber percentage and (d) wild, brown-lint species. (Photographs courtesy of Monica Sheehan, Texas AgriLife Research, Lubbock.)

(*Continued*)

smaller seed and higher yield and is still one of the tools breeders use to manipulate multiple genes for improving yield (Miller and Rawlings, 1967; Smith and Coyle, 1997). Fiber length is likely the quality characteristic most affected by domestication. Developmentally, higher rates of fiber elongation

(c)

(d)

14.4 Continued.

following initiation of lint cells post-anthesis and prolonged fiber elongation lead to longer lint fibers. Proportionally, uniformity of fiber length on a seed is similar in short-fibered wild types and longer-fibered domestic types.

Genetic consequences of domestication include some reduction in available genetic diversity and cotton is no exception. The first level of determining domestication and its effect on genetic diversity is pedigree analysis. Cluster

analysis of coefficient of parentage show cotton cultivars released between 1980 and 1990 have a mean coefficient of parentage of 0.07 (Bowman *et al.*, 1997). The assumption that pedigrees accurately reflect final genotype and that ancestral introductions are unrelated may result in high estimates of genetic diversity. Coefficient of parentage compared to genetic similarity index for two modern cultivars and 12 distant parent cultivars indicates a narrower genetic base than previous studies found (Van Esbroeck *et al.*, 1999). Movement of cotton from the same domestication event to different production areas suggests less genetic diversity exists between geographical groups than desired for breeding. Genetic variation is essential in breeding, but since wide introgression can be problematic, narrowing the genetic base continues in cotton cultivar development. As understanding of the relationship among available genetic resources improves, efforts to broaden genetic diversity in resources appropriate for breeding acceptable varieties is expected.

14.3 Breeding methodology

The particular method to use in developing improved cotton varieties depends on the objectives, heritability of the trait, measurement technology, available germ plasm resources, and comfort level of the breeder to assess individual plants versus populations or lines. Most cotton breeding efforts engage in pure line development that results in varieties that are distinct, uniform and stable. Cotton hybrids are developed in India and some areas of China. The breeding methodologies are quite different, and a cotton breeding program will usually develop either pure lines or hybrids, not both.

Most cotton breeding methods share the same basic process with the following steps:

1. recombine desired traits from individual lines by hybridization; inbreed to create variable populations;
2. select desired plant type;
3. fix traits and create pure lines;
4. evaluate lines in target environment;
5. performance test with other standard varieties;
6. release new commercial variety.

The number of lines created and evaluated is very high compared to the number of lines that are suitable for performance testing because of the large number of characteristics to be evaluated.

When a regional production constraint threatens the capability of cotton cultivation, and resistant varieties are the most viable production option, that constraint becomes a primary breeding objective and may influence

breeding methodology. This can happen with an endemic disease that has limited agronomic control or if major end-use markets demand a certain type of fiber. Heritability of objective traits also influences breeding methodology. Qualitatively inherited traits such as flower color and leaf shape have discrete phenotypic categories in a segregating population, are controlled by one or few genes and have no environmental interaction (Falconer and Mackay, 1996). Quantitatively inherited traits such as yield and fiber quality vary continuously in a population, are controlled by many genes, and are highly sensitive to environmental interaction (Sprague, 1996). Applied cotton breeders are most interested in quantitative traits because they convey the highest economic impact to new cotton varieties. All genes are assumed to be governed by Mendelian genetics, so quantitative genetics is an extension of that segregation principle to multiple traits.

One of the limitations in breeding improved cotton varieties is the ability to measure either a specific trait of interest or the combination of traits that results in superior genotypes. Environmental interaction increases phenotypic variation that can confound genetic factors and prevent accurate selection in a single environment. If the environment interaction is such that the ability to select individual plants that would derive a superior genotype is mired, then a breeding methodology relying on population selection instead of individual selection might be considered. Understanding the genetic control of quantitative traits is important in avoiding this limitation (Mangal, 1989).

Genetic resources include existing commercial cultivars, germ plasm lines from public breeding programs that may carry a specific trait of interest, obsolete cultivars and introduced germ plasm available from world collections. Major world cotton collections are maintained by USDA-ARS and Texas A&M University in College Station, Texas, USA; CIRAD in Montpellier, France; and Uzbekistan. Genetic engineering provides an additional prospect of new gene introduction but the primary concern of the applied breeder remains developing cotton varieties that perform well in a production environment regardless of the gene source.

14.3.1 Creating diversity

Despite tremendous variation within the *Gossypium* species, bottlenecks in genetic diversity have occurred during the process of domestication. Regional breeding programs test as many available sources of variation as possible over multiple locations and years to determine the best lines to use as foundation from which to develop improved varieties. At the same time, characteristics are noted to determine lines that can be used as sources in hybridization. Sometimes varieties that are phenotypically uniform in their target environment segregate into morphologically different types in

another environment, indicating a mixture of genotypes that can be further exploited through selection. As variation is exhausted within a line such that direct selection no longer results in significant improvement, controlled cross-pollination is used to introduce the required variability. Transgressive segregates that result in significant improvement usually come from wide crosses, so the common practice of crossing adapted types to create new populations should be supplemented with crosses designed to create additional diversity in a germ plasm pool. Mating schemes include direct crosses between adapted varieties with complementary characteristics, complex crosses with F_1 hybrid plants that are heterozygous for two or more desired characteristics and inter-mating individuals from segregating populations, among other strategies. Crossing between diploid and tetraploid species involves doubling the chromosomes from the diploid source with colchizine; and crossing with photoperiodic race-stocks requires either conversion of the race-stock to a day neutral flowering habit or utilizing timed black-out covers in a greenhouse environment. Chemical mutagenesis has also been used as a method to create variation (Auld *et al.*, 2009).

Commercial programs tend to have the majority effort dedicated to cultivar development, concentrating on competitive market varieties, and a smaller effort in introducing desirable traits from new genetic sources while removing detrimental effects to the point that new genetic material can be used effectively in hybridization. Public breeding programs tend to devote more effort to pure germ plasm development (sometimes referred to as developmental or pre-breeding) and less effort to applied breeding for the purpose of cultivar development. Developmental breeding is the bridge between genetics and applied breeding and successful programs will include a component of both, albeit in different proportions. Once appropriate variability has been established in breeding populations, a decision must be made on how best to proceed for cultivar or line improvement. Whatever breeding methodology is used, the ultimate goal is to combine as many favorable alleles as possible into one plant or line that results in a distinct, stable and uniform variety. The most common methods used in cotton for self-pollinated crops include pedigree breeding, population breeding and backcross breeding. Hybrid development is used rarely in cotton, but employs the same methods to produce parent lines. Recurrent selection is used primarily in research.

14.3.2 Pedigree breeding

Pedigree breeding relies on at least one, and typically two or three, cycles of individual plant selection. The first step in pedigree breeding is controlled cross-pollination with the resulting seed assumed to be similarly

heterozygous, so no selection is made from the F_1 individuals resulting from planting the crosses. Since no selection is made, F_1 seed can be self-pollinated and increased in a counter-season nursery to provide an appropriate amount of F_2 seed for selection the following growing season in the target environment. The F_2 generation theoretically represents maximum genetic segregation and as many plants that resources allow should be selected to capture enough variability in the population to improve probability of identifying superior individuals. If field screening is available, such as a field infested with a target disease, pressure can be applied in the plant selection process. For characteristics measured post-harvest, such as fiber properties, selection pressure is applied after harvesting individual plants and prior to planting the next generation. Seed from F_2 individual plants are planted in a progeny row. The productivity of the F_2 plant and whether replanting is anticipated are factors in the length of the progeny row or if replications are possible.

Progeny rows are evaluated on a visual basis, and in some cases, data from pre-harvest boll samples or yield estimates, and either a second round of individual plant selection or a random boll sample within the best progeny rows is harvested to plant the next generation. Theoretically, uniformity can be achieved by the F_4 generation, but testing more commonly takes place after the F_5 generation. Plant or boll sample seed to progeny row evaluation continues until the breeder identifies a progeny row as appropriately uniform for performance testing or as a good source for crossing. Plants and rows are discarded throughout the process according to selection criteria established for the primary breeding objectives. Once a progeny row is selected for performance testing, seed from the whole row is harvested and processed for preliminary replicated yield trials in as many locations as seed supply allows. At the same time, some seed is reserved to plant in an increase block in a location conducive to high seed quality production where pollinators can be controlled. This seed is used for testing the best lines from preliminary testing at more locations in intermediate tests. Ideally, new varieties are identified after a third round of advanced tests. Appropriate check varieties, such as regional standards or lines that exhibit target characteristics, are utilized throughout the nursery and testing phase of the system. A typical pedigree breeding scheme is illustrated in Table 14.2. Timelines for developing new varieties range from 7 to 10 years, including 1 year for hybridization and F_2 seed production, three to five generations of selection for stabilization, and three to four years of testing and seed increase. Visual assessment by the breeder is a fundamental feature of the pedigree progeny row method. Both in early generation plant selection and progeny row evaluation, success depends on the ability of the breeder to identify performance potential and avoid discarding promising lines. Breeders who have less confidence in subjective evaluation and prefer to rely on more objective data may choose a form of bulk population breeding.

Table 14.2 Typical pedigree breeding scheme for cotton variety development

Year	Generation	Action	Criteria	Location
1	P1 × P2	Hybridize desired genotypes	Goals, parent traits	Anywhere
	F_1	Self-pollinate	Create variability	Winter nursery or greenhouse
2	F_2	Select single plants (1000's) at main breeding location	Leaf characteristics, disease, plant type, fiber	Area of adaptation
3	F_3	Progeny evaluation (100's) at 1 to 2 locations, select 10's	Agronomics, fiber, some on yield	Area of adaptation
4	F_4	Select single plants (1000's) at main breeding location	Disease, plant type, fiber, etc.	Area of adaptation
5	F_5	Progeny evaluation (100's) at 1 to 2 locations	Agronomics, fiber, some on yield	Area of adaptation
	F_6	Seed increase	Seed amount, rogue advanced generation	Winter nursery
6	F_7	Preliminary strain evaluation (10's–100) at 2 to 5 locations	Performance, fiber, agronomics	Area of adaptation
7	F_8	Advanced strain evaluation (10's) at 5 to 10 locations	Performance, fiber, agronomics	Area of adaptation
8	F_9	Elite strain evaluation (1–10's) at 10 to 20 locations enter public strains test	Performance, fiber, agronomics	Area of adaptation
		Plot size increase	Seed amount, purity	Winter nursery
9	F_{10}	Commercial variety tests (1's) at 10 to 20 locations enter public variety tests	Performance, fiber, agronomics	Area of adaptation plus additional regions
		1 to 5 hectare size seed increase	Seed amount, purity	Winter nursery
10	F_{11}	Commercial variety test (1's) at 10 to 20 locations enter public variety tests, farmer strip trials and demos	Performance, fiber, agronomics	Area of adaptation plus additional regions
		50 hectare size seed increase	Seed amount, purity	Production area
11	F_{12+}	Initial commercial release	Superior to current products/competition	Target market

14.3.3 Population breeding

There are many modifications of pedigree breeding and bulk methods that can be characterized as population breeding. Fundamentally, population breeding begins with either a mixed population or segregating F_2 population, from which a random bulk sample is harvested repeatedly over several generations until the percentage of homozygous individuals approaches 100. The total number of individual plant and plots that can be handled in a program influences the number of populations developed each year and the number of selections from each population. Bulk breeding tends to rely more on many populations while pedigree breeding may employ less populations with more selections from each one. Natural selection among populations will favor quantitative traits that influence adaptation, possibly more effectively than a breeder, but can also influence characteristics in opposition to primary breeding objectives. For this reason, many bulk population methods are modified at times in the developmental process by artificial selection.

The idea of modified bulk breeding is to capture the entire variability in a population by taking a seed or boll from each plant in a segregating population (single seed descent) and making individual plant selections in the F_3 or F_4 to create progeny rows for testing potential evaluation. This is desirable if the environment or predilection of the breeder precludes effective plant selection in the F_2 generation. Another modification is to increase enough F_1 seed for a large F_2 population that can be planted in a replicated test. Objective yield and quality data combined with replicated observation identify the most promising populations from which to select or bring forward in a bulk method. This concept assumes all populations tested have similar distribution for desired characteristics in the F_2 population, which may not be the case depending on the genetic distance of the parents. Another technique involves individual plant selection in the F_2 generation, followed by progeny row evaluation and bulking of the best F_3 progeny rows. Replicated testing is performed in the F_4 generation followed either by another year of testing or reselection, depending on performance of $F_{2:4}$ populations. These methods are distinguished from classical pedigree breeding in that maximum genetic variability can be maintained efficiently, but undesirable genotypes are not eliminated early in the development process. More resources are generally expended in testing, rather than nursery evaluation and screening, in population breeding.

14.3.4 Backcross breeding

Backcross breeding has traditionally been used to transfer one or few qualitatively inherited traits to an otherwise high performance variety lacking those traits. The adapted variety is the recurrent parent and the germ plasm line containing the desired trait is the donor parent. The two lines

are cross-pollinated and the resulting F_1 crossed back to the recurrent parent. This process is repeated until the desired theoretical percentage of the recurrent parent combined with the new trait is obtained. After each cross, only those individual F_1 plants with the characteristics being transferred from the donor parent are used in the next backcross. Five backcrosses are considered sufficient to recover the recurrent parent genotype, possibly six if the parents are genetically distant. With the aid of molecular markers, as few as two or three backcrosses is necessary because the plants with higher percentage of recurrent parent alleles that also carry the donor parent trait can be identified.

Although this method has been used successfully to transfer disease resistance and simply-inherited morphological traits, it was not typically the primary methodology used in cultivar development until the advent of traits arising from recombinant DNA technology. Because of strict purity requirements for transgenic cotton varieties and time urgency for developing commercially acceptable varieties for markets that accept and demand biotechnology traits, backcross breeding has become the preferred method for developing transgenic varieties. Some form of pedigree or population breeding is still used to identify new recurrent parent lines acceptable for conversion through backcross breeding.

By 2010, biotech cotton had been cultivated for 15 years (James, 2010). By million hectares, the cotton growing countries with the largest area of total biotech crop cultivation include USA (66.8), Brazil (25.4), Argentina (22.9), India (9.4) and China (3.5). Other countries growing biotech cotton by 2010 include Australia, Pakistan, South Africa, Myanmar, Burkina Faso, Mexico, Colombia and Costa Rica. Effort from gene discovery to new variety launch for biotechnology cotton varieties can range from 11 to 20 years, as illustrated in Table 14.3. Timeline to new variety from initial donor parent cross to recurrent parent is five years. For new biotechnology traits, the initial donor parent is from the genotype used in transformation and regeneration. Generally, three backcrosses, and as few as two using marker-assisted backcrossing, are accomplished in a greenhouse environment where plants used from both parents can be tested for purity prior to controlled cross-pollination. After the required number of backcrosses is achieved, the BC_XF_1 generation is self-pollinated to produce BC_XF_2 seed. Each plant in the segregating population is analyzed to identify individuals that are homozygous and pure for the trait or traits being transferred. These individuals are planted in progeny rows in the target environment or counter-season field depending on when they emerge from the greenhouse. Percentage of plants selected for progeny row evaluation decreases with the number of independently segregating biotechnology events being transferred. Selection or bulk harvest of best rows advance to the F_4 generation, and seed is increased for performance testing. Performance testing in

Table 14.3 General activities and timelines in development of biotechnology cotton varieties

2–10 years **Laboratory**		4–5 years **Greenhouse and field**
Gene discovery, plasmid and vector construction, plant transformation	→	Proof of concept, elite event selection, regulatory approval
	Introgression and variety conversion Purify for gene and plant type, select homozygous plants for pure line development (BC_3-$5F_{2-3}$) 2 generations: field and winter nursery	
Transfer Transfer novel gene from transformed plant to adapted variety 3 generations backcross to adapted variety (BC_3F_1) 2 years: greenhouse		**Test** Test pure lines for gene efficacy, quality and yield performance Select lines for new variety and increase seed (BC_3-$5F_{4-6}$) 2 years: field and counterseason
	Commercialization Minimum 5 years from first cross to limited commercial launch	

backcross breeding can be referred to as component line equivalency testing, as sister lines are compared to the original recurrent parent. Since the performance of recurrent parents is already established, testing may commence for only one or two years before commercial seed increase decision. A typical scheme for developing transgenic cotton varieties using backcross breeding is shown in Table 14.4

14.3.5 Cotton hybrids

Breeding efforts in cotton are primarily for pure line development in a self-pollinated crop. Cotton is thought not to suffer from inbreeding depression, and evidence of heterosis in both interspecific (Marani, 1967) and intraspecific (Davis, 1978) hybrids has been reported. Commercial hybrid seed has had limited success, especially in intensively cultivated developed regions. Emasculation of the female parent in this self-pollinated crop was a limitation until the discovery of a cytoplasmic genetic male sterility system and restorer genes from *G. harknessii* (Meyer, 1975). Reports of heterosis in cotton hybrids claim different levels of productivity advantage, rarely economical in comparison to seed production costs. Some reasons for erratic success are related to the growth habit of the cotton plant and the yield component relationships regarding the yield of cotton fiber.

Cotton's perennial growth habit necessitates a balance between vegetative and fruiting structures in intensively cultivated cotton, to maximize fruit development in a shortened growing season. F_1 hybrid vigor results in an aggressively vegetative plant sometimes to the detriment of boll development. Hybrid vigor also results in a large seed, desirable in crops where seed is the primary component by which yield is measured. Yield of cotton is measured in fiber produced per unit land area, and without within-boll yield component changes for seed cotton weight harvested, a larger seed can result in lower percentage of fiber by weight. Conversely, lint yield improvement has been made in modern cotton cultivars by reducing the seed size, and increasing the percentage of lint within the boll (Kiem, 2002). Developers of hybrid seed must take into account these factors when choosing parents for cotton hybrids. Parents for hybrid cotton are ultimately developed with the same methodology used for cultivar development. Cotton cultivation in India and parts of China where commercial hybrids are grown uses a much lower plant density than in machine-harvested regions. The growing season is typically longer and vigorous and large plants are not so detrimental to yield. More bolls produced over a longer period of time can be considered beneficial where hand harvesting is practiced.

Even with genetic or cytoplasmic sterility and restorer systems, hybrid seed production costs are high and insect pollination is not efficient. Some hybrids are produced through hand emasculation and pollination, a practice

Table 14.4 Typical backcross breeding method scheme for biotechnology cotton variety development

Year	Generation	Action	Criteria	Location
1				
1st trimester	R_0	Screen transformed plants, cross to elite lines (RP), self	Contains functional gene, single insert, single copy	Greenhouse, biotech lab
	x RP	R1… R2… R3… R4 Selfing and selection	Get homozygous line	Greenhouse
			Evaluate efficacy, genetic and molecular tests, regulatory testing – years 2, 3, and 4	Field
2nd trimester	R_0F_1	Cross back to RP	Contains gene, normal plant type	Greenhouse
3rd trimester	BC_1F_1	Cross back to RP	Contains gene, normal plant type	Greenhouse
2				
1st trimester	BC_2F_1	Cross back to RP	Contains gene, normal plant type	Greenhouse
2nd trimester	BC_3F_1	Cross back to RP	Contains gene, normal plant type	Greenhouse
3rd trimester	BC_4F_1	Cross back to RP	Contains gene, normal plant type	Greenhouse
3				
1st trimester	BC_5F_1	Start purification for gene and plant type (self pollination)	Contains gene, normal plant type	Greenhouse
2nd trimester	BC_5F_2	Continue purification (self pollination)	Contains gene, normal plant type, agronomics, disease	Field, adapted area
3rd trimester	BC_5F_3	Select pure lines	Gene homozygous, leaf characteristics, plant type	Winter nursery

(*Continued*)

Table 14.4 Continued

Year	Generation	Action	Criteria	Location
4 Summer	F_4	Efficacy and performance testing at multiple locations	Gene homozygous equivalent performance to RP transgene stability	Field, adapted area
		Seed increase	Purity, type, seed quality	Field, adapted area
Winter	F_5	Seed increase	Purity, type, seed quality	Winter production
5	F_6	Initial commercial release	Value-added product	Target market

restrictive where labor costs are higher. Transgenic technology is offered exclusively through hybrids in India, partially for intellectual property protection. Another advantage of hybrid cotton development for biotechnology trait delivery is that a few parent lines can be converted through backcrossing and many different hybrids can be made using these few converted parent lines. Hybrid cotton has been explored by many companies involved in the seed business to limit the practice of farmer-caught seed, but the high cost of seed production coupled with minimal economic advantages in yield and quality over varieties continues to limit commercial exploitation of hybrid cotton, resulting in less research toward their development.

14.4 Agronomy and physiology

Knowledge of cotton agronomy and physiology is important in breeding, especially during selection, an important component of any cotton breeding scheme (Munro, 1987). In selecting apparently superior plants in a population or superior populations, it is critical to distinguish between those whose superiority is caused by their environment and those which have a superior genotype. This is particularly important in cotton, which responds more than many other crops to small difference in the environment such as spacing, soil, weather and insect attack. Selection may well be directed towards a single character, which has been identified as the primary objective; at the same time it is important that the other good qualities of the material are not lost. In so far as visual characters indicate these qualities, they can be maintained by ensuring that the selected plants conform to the desired type, and this is where a thorough knowledge of the crop is essential. While selecting for a single character such as disease resistance, the experienced breeder will note such things as plant height, vegetative branching,

internode length, boll size and so on, and use these observations in discarding plants or progenies which are off-type and undesirable.

14.4.1 Crop management

A very brief review of cotton crop management will include land preparation, variety selection, planting, weed and insect control, soil fertility, disease pressure, plant growth regulation and harvesting. Cotton grows on a wide range of soil types with adequate root range and pH values from 4.5 to 8.5. Bedding or planting flat depends on whether either drainage or moisture capture is required for root growth. Land may be prepared in terraces to limit soil erosion. At least a portion of seasonal nitrogen requirements and in some instances, pre-plant, incorporated herbicides is applied before planting. Plant spacing can range from 1 plant/m^2 for hybrid cultivation in India, to more than 50,000 plants/ha in ultra-narrow row, no-tillage production in USA. Common planting rate in typical machine-harvested production is ~16,000 plants/ha or ~12 plants/m on 1 m row spacing. Hand harvested cultivation may also use ~1 m row spacing, with just fewer plants/m. Planting season depends on both seasonal water availability and soil temperature. In tropical growing regions, cotton is planted so the rainy period falls during blooming and boll fill and harvesting is accomplished during the dry season. In temperate, short growing season areas, cotton is planted when soil temperatures average 16°C over 10 days.

Variety selection depends first on season length and harvest method (early maturing variety with compact plants versus late maturing, spreading varieties), whether genetically modified varieties are approved, and on the fiber requirements of the primary target market. Local variety trial information is used to determine the best performing varieties for the region and microclimate. Weed control will vary depending on availability and approval of varieties with resistance to over-the-top applications of non-selective herbicides. Where tolerant varieties are not available, pre-plant and residual herbicides are used early followed by elimination of competitive weeds by hand. Seed treatments or in-furrow insecticide applications are used to control early season insects such as thrips during the first 6 weeks after planting. The type of sucking and chewing pests on cotton differs around the world and in some cases varietal selection is impacted by insect pressure. Very hairy leaf varieties are grown in India where jassids are a problem. From a management perspective in cotton, it is important to protect early bolls and to limit late season foliar feeders to alleviate stickiness in the cotton fibers harvested. Root knot and reniform nematodes are also important pests in cotton that limit plant growth. Rotation to a resistant host and tolerant varieties are the best control methods.

The most important nutrients in cotton are nitrogen, phosphorus and potassium. Yield targets and previous crop dictate the amount applied. When heavy nitrogen applications occur in high yield situations, it is often recommended to apply plant growth regulators to achieve optimum vegetative/fruiting balance. Plant growth regulators are recommended for machine-harvested crops when plant height to number of main stem nodes ratio exceeds 1.5. Plant growth regulators are also recommended when nodes above white flower at bloom exceeds 10. Crop termination is imminent when nodes above white flower approach five. If this physiological stage approaches too rapidly, irrigation or foliar nitrogen feeding is considered. When there are four harvestable bolls above the last cracked boll, artificial termination for machine-harvested crops helps condition the crop for harvesting. Cotton can be either defoliated, desiccated or a combination of both prior to harvest. Nitrogen, water and crop termination products are used in a way to minimize non-fiber plant parts in harvested cotton.

14.4.2 Adaptation

More than 50 species of *Gossypium* from at least three different centers of origin have been identified, but four of the species produce seed with spinnable cotton fibers. These four species include two diploid, Old World species, *G. herbaceum* and *G. arboreum*; and two allotetraploid, New World species, *G. hirsutum* and *G. barbadense*. *G. hirsutum*, the most important commercial species, is thought to have originated near present-day Guatemala (Hutchinson *et al*., 1947); and *G. barbadense*, the other commercial New World species, in northwestern South America, west of the Andes (Percy and Wendel, 1990). The two diploid cultivated species originated in Asia (though not definitive for *G. arboreum*) and Southern Africa, in the case of *G. herbaceum* (Hutchinson, 1959). The four cultivated species have been grown now in virtually every major region of the world, but primarily in arid to semi-arid regions of the tropics and subtropics. Commercial cotton cultivation can be found from 37°N in the United States to 34°S in Australia.

Balance of heat, light and water determines the climate at which any green plant needs to grow, and the climate, combined with economic factors, determines the cultivation system to use. Cotton evolved in drier and hotter climates of the world and needs sunshine and high temperatures for optimal development. Minimum temperature for a period of at least 130 days defines the primary adaptation of cotton production, and fiber does not develop in temperatures below 16°C for *G. hirsutum* and 12°C for *G. barbadense*. Temperature is the limiting factor in temperate climates and high altitude, with 1200 m being generally the upper limit.

Extended periods of cloudy conditions lead to fruit shed in cotton, and most cotton is cultivated where ample sunshine is present during the growing

season. In humid areas with cloud cover, such as the Pacific coast of South America, parts of Uganda and the southeastern United States, sufficient radiation from sunlight is still required to produce a good crop. The daily hours of sunshine required for cotton depends on the radiation in calories cm^2; for example, less than 4 h per day in parts of Africa cause fruit shed while 4 hours per day in Ecuador is normal and does not affect fruit set. In very sunny climates, much of plant growth occurs at night and at tropical temperatures, direct sunlight can cancel out the effect of high day temperatures and prevent excessive growth. For cotton adaptation, where excessive vegetative growth is undesirable, minimum or night temperatures are more highly related to adaptation than maximum temperature.

Cotton can be grown in very dry climates where irrigation is required, or in dry-land, or rain-fed situations. When moisture and temperature are sufficient for germination, cotton, unlike many other crops, can produce a salable portion of the crop (fiber) with little in-season moisture. Highest yield occurs in dry areas with full irrigation capability, such as Australia and the irrigated western United States. Growing regions where rainfall occurs during the season experience more inconsistent yields, depending on the timing of the rainfall. Areas of adaptation occur where irrigation timing can be controlled, or where rainfall patterns favor moisture during the planting, pre-bloom and blooming stage, followed by dry conditions during harvest.

14.4.3 Growth habit

Cotton has evolved from a perennial dicotyledon plant to be grown as an annual crop. Growth habit of modern cotton cultivars follows a predictable pattern with vegetative and fruiting structures appearing simultaneously, as in a perennial. The organs that develop throughout the lifespan of the plant include roots, stems, leaves and fruit (squares, flowers and bolls). The cotton plant has a primary taproot with many lateral roots that can form near the soil surface or deeper, depending on water availability. Cotyledon emergence in favorable germination conditions occurs 7–10 days following planting, with 5–9 true leaf nodes developing up the main stem prior to the first fruiting branch. Vertical fruiting index (VFI), or time between the first flower on one fruiting branch to the first flower on the next fruiting branch, averages 3 days. Horizontal fruiting index (HFI), or time between the first flower on a fruiting branch to the second flower on the same fruiting branch, averages 6 days. Structure of fruiting branch development up the main stem is a 3/8 spiral phyllotaxy, with fruiting branches developing progressively up the main stem 3/8 turn around the main stem. Axillary buds on nodes produce a branch or fruiting form, and a leaf. Very rarely a third bud can differentiate into an additional fruiting structure. Occasionally, vegetative branches develop on nodes below the first fruiting branch.

Table 14.5 Average degree day (DD) schedule for cotton growth and development

Stage of development	DD60s required (from planting)	Days (mean)
To emergence	70	7
To first square	500	35
To first bloom	1000	60
To first open boll	1800	110
To 70% open bolls	2250	140

Calculations: average daily temperature in °F – 60 = heat units (DD60); (daily maximum temperature + daily minimum temperature)/2 = average daily temperature in °F.

Timing between major events of cotton plant development depends on effective heat unit accumulation, solar radiation, nutrition and water availability. Effective heat units for cotton are calculated in degree days based on 15–16°C (DD60), as average daily temperature (daily high temperature + daily low temperature/2)–15.68. Planting to emergence is 50–60 DD60, to first square is 425–475 DD60, to first bloom is 775–850 DD60, to first open boll is 1625–1800 DD60 and to harvest-ready is 2100–2400 DD60. The average number of days for each cotton development stages is interpreted in Table 14.5. Internode length, or distance between nodes on a vegetative or fruiting branch also depends on temperature, sunlight, nutrition and water availability, but also on variety. Variety plant structure can vary from compact, cluster fruiting types with very short internodes, almost no space between nodes, to tall, branchy vigorous plants with up to 15 cm between nodes. In many mechanically harvested, intensively cultivated regions, plant structure is managed by hormonal growth regulators to limit internode length. In hand-harvested regions, plant spacing is wider and varieties or hybrids with longer internode lengths are cultivated. Number of main stem nodes on varieties under different management systems can range from ~8 in dry-land, intensively cultivated situations to more than 30 in fully irrigated or high rainfall areas with lower plant populations.

Growth habit of the cotton plant has implications in fiber development and uniformity as lower, earlier bolls are developing under different conditions and different times than bolls higher up the main stem node and farther out on the fruiting branches. The variability among bolls on a plant is important for the breeder to understand, especially in sampling for fiber tests. The first 4–8 first position bolls contribute a high percentage of yield and generally produce the best fiber quality, representing genetic potential. For each individual boll, fiber elongation begins at or just before bloom and continues for approximately 21 days when final fiber length is determined.

Fiber maturation begins just before elongation ceases and continues until the boll is open. Fiber maturity and strength vary among bolls on a plant, and a higher percentage of immature fiber can lead to diminished length uniformity because immature fibers are more prone to break during processing.

14.5 Breeding targets

Primary breeding objectives include those that will influence productivity and quality of output in the target climate and market. Even secondary targets such as disease resistance and water use efficiency are aimed at improving productivity and quality. Those specific targets will be reviewed, though host plant resistance to insects, plant architecture and crop maturity remain important objectives in many cotton breeding programs.

14.5.1 Productivity

Fiber quality and value is important in cotton breeding, but productivity remains the most important economic component, making increased lint yield the primary breeding target. Lint yield is a highly quantitative trait and genetically controlled factors all have significant interaction with environment. Gains in breeding depend on reducing the complexity to components affecting yield that can be selected for in different environments. The most basic components of lint yield based on crop ontology are described by Worley *et al*. (1976). Negative correlations between some of the components make it difficult to improve yield by simply selecting for certain measurable yield components, but yield components such as lint/seed and bolls/area land unit are useful in selecting complementary parents for population development aimed at yield improvement. Breeding for improved yield has altered the balance among yield components over the years. Miller and Rawlings (1967) report correlated response to increased yield of higher lint percent, seed/boll and micronaire with corresponding decrease in boll size, seed size and fiber length. Consequently, most modern cotton varieties in intensive cultivation have smaller seed, more but smaller bolls and coarser fiber than their precedents. In hand-harvested regions, a bigger boll is more desirable, and in regions where seed cotton, as opposed to lint, is sold by the farmer, lint percentage is not as economically critical. An acceptable balance for a target market among contrasting yield components while improving overall yield is a challenge for the cotton breeder.

Cotton's perennial growth habit makes breeding efforts targeting dry matter partitioning and photosynthetic efficiency more complex than in many agronomic crops, though shifting of dry matter from vegetative to reproductive structures continues to be a viable objective for improved yield (Meredith and Wells, 1984). Higher lint yields indicate a redirection of

photosynthetic sources to lint from leaf and seed. Yield potential is assessed in early generations by measuring yield components, avoiding extremes on any one individual component, managing nurseries so that genotypic plant architecture is evident and selecting appropriate numbers to capture genetic variability in a population. The best evaluation for improved yield and yield stability remains small-plot replicated trials of near-homozygous lines in several locations over at least two years and preferably three.

14.5.2 Fiber quality

Fiber quality objectives in breeding programs are partially determined by market price signals for individual fiber properties and raw fiber requirements for regional target markets. Fiber quality is a combination of individual fiber properties that contribute to spinning performance, including fiber length, length uniformity, strength, micronaire (an indirect measure of fineness and maturity) and elongation. Quality also encompasses cleanliness factors that contribute to spinning efficiency and product quality such as leaf trash content, color, short fiber content, other contaminants, and preparation that contributes to neps, small entanglements of fiber that manifest as knots or white specks. Classical cotton breeding has made improvement in characteristics that can be readily measured and show additive to partially dominant quantitative inheritance, such as length, strength and biological fineness (Al-Rawi and Kohel, 1970). Cotton fiber properties, particularly strength, are thought to be influenced by structural organization of the cellulose chains, but since the degree of polymerization in cotton fiber was measured and reported in 1948, little information is reported as it relates to fiber quality breeding since these measurements are difficult. Marx-Figini (1982) determined that secondary wall molecular weight, which has constant degree of polymerization during fiber development, is much higher than in primary wall, where molecular weight varies during fiber development. Timpa and Raney (1989) attempted to link degree of polymerization with varietal differences in fiber strength and report that 38% of variability in HVI-measured fiber strength is attributed to average molecular weight. Later studies added environmental and fruiting zone variables, and molecular weight responds to both, further complicating use of fine fiber structure in breeding. Indirect effect on fiber quality has been accomplished through breeding by reducing leaf trichomes that contribute to trash content and earlier maturity cycles that reduces variability and fiber breakage associated with immature fiber.

Historically, breeders relied on improving quality factors that they could see and feel, such as color, staple length determined by hand and the hand, or scroop, that indicated fiber maturity and fineness. Instruments that could indirectly measure maturity and fineness by resistance to airflow (micronaire),

and directly measure fiber strength (Pressley, Stelometer) and fiber length (Fibrograph) (Ramey, 1999) advanced improvement through breeding even though relatively small numbers could be analyzed economically. High Volume Instruments (HVI) could measure many fiber properties efficiently and breeders could begin to identify more complete fiber profiles that contribute to quality as well as individual fiber characteristics. Advanced Fiber Information System (AFIS) added more direct measurements of fiber length distribution and fiber fineness and maturity. An understanding of the interrelationships among fiber properties and their contribution to spinning quality allows breeders to select for improved quality beyond improving individual characteristics independently (Hequet *et al*., 2007). Fiber improvement through breeding continues to rely on available measurements that can be made efficiently and accurately on a large number of samples.

Fiber quality is a complex quantitative trait and some progress has been made in identifying quantitative trait loci (QTL) associated with some characteristics involved in fiber development (Jiang *et al*., 1998). It is interesting to note that many QTLs for fiber quality were located on the D subgenome, a genome from an ancestor that does not produce spinnable fiber and accounts for most of the genetic variation in fiber traits in the New World allotetraploid species. Yu *et al*. (1998) identified 3 QTLs for fiber strength, 3 QTLs for fiber length and 5 QTLs for fiber fineness that account for about 35–50% of total genetic variability within their population (*G. hirsutum* TM-1 X *G. barbadense* 3-79). While these tools can prove useful, it is still necessary, and indeed possible, to manipulate fiber traits beyond individual properties of length, strength and fineness through classical breeding techniques (Meredith *et al*., 1996).

The genetic expression of fiber traits is highly influenced by environment, albeit length, strength, leaf hair, biological fineness and elongation less than length uniformity, maturity, color, short fiber content and neps. More rapid and repeatable measurements for some of these characteristics would advance breeding improvements further. It is also very important to include check varieties of known fiber quality in nurseries at regular intervals and in tests, so relative, instead of absolute, improvement can be determined in different environments and years. Sampling for fiber quality analysis is also important since fiber properties vary considerably among bolls on a plant. Breeders should be aware of designing nurseries and tests to be able to distinguish genetic fiber quality potential among lines and plants as well as determine plant development, growth habit and disease resistance factors that will impact fiber quality.

14.5.3 Disease resistance

Major diseases affecting cotton that have been targets for resistance breeding include bacterial blight (*Xanthomonas axonopodis* pv. *malvacearum*);

fungal wilts Fusarium wilt (*Fusarium oxysporum* f. sp. *vasinfectum*) and Verticillium wilt (*Verticillium dahlia* and *V. albo-atrum*); and viral disorders like leaf curl, anthocyanosis (blue disease), leaf crumple and cauliflower mosaics; and root-knot (*Meloidogyne incognita*) and reniform (*Rotylenchulus reniformis*) nematode. Diseases for which good sources of cultivar resistance are not utilized or have yet to be identified include seedling diseases (*Rhizoctonia*, *Pythium*), black root rot (*Thielaviopsis basicola*), leaf spots and blights (*Alternaria* sp., *Aschochyta gossypii* and *Cercospora gossypina*), and Phymatotrichopsis root rot (*Phymatotrichopsis omnivora*). Classical breeding techniques remain the most prominent method of conferring disease resistance as adoption of biotech cotton remains primarily in insect pest and herbicide resistance (James, 2010).

Bacterial blight is present in most cotton growing regions, but is minor in some except in the cases of heavy monsoon rains and can cause serious crop losses or failure in others, such as Australia, south Texas and western Africa. Seven resistance genes have been identified in tetraploid cotton, 86% mapped to the D subgenome chromosomes (Wright *et al*., 1998). Breeding for resistance has been successful, with near-immune varieties developed for areas where the disease is endemic. Field inoculation followed by infection ratings is effective in selecting resistant plants in segregating populations as well as selecting resistant lines, and is less influenced by non-uniform field infestation. Backcross breeding has been used successfully to transfer resistant gene/genes to adapted varieties. Expressed Sequence Tags (ESTs) have been developed for bacterial blight, but marker-assisted breeding should be complemented by field evaluation to delay potential development of pathogen resistance to a single gene, even though diversity among the known resistance genes might be negligible (Delannoy *et al*., 2005).

Breeding for resistance to wilt diseases, among the most globally widespread pathogens, has progressed well over the last 30 years (Bell, 1995). Most methodology encompasses identification of resistant sources through greenhouse inoculation or infected field screen and selection of plants and lines in infected fields accompanied by alternate verification in greenhouse inoculation assays. Several sources of partial resistance are available and screening methodology and tolerance mechanisms, while complex, quantitative and partial in nature, are sufficient to identify resistant plants and lines in the segregating state. Lack of uniformity in field infestation or weather conditions not conducive to symptom development can cause selection of false positive resistant plants and line verification following the breeding development cycle is necessary.

Nematodes are present in most cotton-producing regions and are considered serious production deterrents where they occur. Distribution in infested fields is uneven and symptoms are subject to weather conditions, making field selection problematic. Most cultivated varieties are susceptible to nematodes, with a few exceptions for root-knot nematode (RKN) tolerance.

Current RKN tolerant cultivars are derived from 'Auburn 623RNR', 'LA 434-RKR' and 'Acala Nem-X'. While there are few breeding sources available, the resistance is good, heritable, multi-gene, incomplete dominant or additive and with some genetic diversity among the resistance genes, albeit difficult to field-select. Resistance sources for reniform nematode have been identified within diploid species, such as *G. longiocalyx*, and more recently in *G. barbadense* L. accession GB713 (Gutierrez *et al*., 2010). Introgression in adapted cultivars has not yet been successful. Difficulty in field selection and greenhouse assays for discriminating segregating populations, and mapping of resistance genes makes nematode resistance a candidate for marker-assisted breeding. Nematodes can also develop resistance to existing genetic sources, so new sources need to be identified and developed to continue a breeding pipeline for cultivar development. Besides crop rotation and management, resistant varieties are the best option since aldicarb, the only economic crop protection alternative, will be discontinued in cotton.

Viral diseases are most important in tropical or subtropical climates in Africa, Asia, Central and South America, though viral diseases transmitted by whiteflies have increased in areas with insecticide-resistant outbreaks in Pakistan, southwestern USA and Australia. Viral diseases are controlled by eliminating reservoir hosts and insect vectors, but resistant varieties are desirable, especially in integrated pest management systems. Single dominant gene action has been demonstrated in cotton leaf curl virus in Pakistan (Ali, 1997) and markers have been identified. Marker-assisted selection is important for conferring viral disease resistance because laboratory and field screening methods are insufficient for individual plant selection in a segregating population. Breeding strategies for blue disease resistance in Brazil involve identifying resistant sources through laboratory screening, some with cross-tolerance to bunchy top, creating populations and developing lines which are then screened again in the laboratory.

14.5.4 Water use efficiency

The answer many cotton experts give to the question of which cultivars are more 'drought tolerant' is that 'cotton is drought tolerant' and it does originate from perennial plants adapted to semi-arid environments. The concept of water use efficiency (WUE) and effects of drought at different stages of crop development is complex, and in cotton, highest yield is not necessarily achieved from uninterrupted vegetative growth. Agronomic WUE is the ratio between dry matter production and water use (Condon and Hall, 1997); physiologically, it is defined as the ratio between carbon fixed and rate of water transpired (Araus *et al*., 2003). Variations on these concepts are indirectly addressed in a major objective of cotton breeding, improved

productivity. More stress tolerant varieties are developed either by selection under drought conditions followed by testing in both irrigated and dry conditions or by selecting under more optimum conditions for traits that are thought to confer better water use efficiency. Traits associated with WUE are rarely used as selection criteria in breeding because they are difficult to measure, especially on individual plants or large numbers of plants; and heritability and relationship to yield is not well determined.

Water available for crop irrigation is a critical issue and considerable research effort is given to understanding response to water deficit at the physiological and genetic level. More than 100 exotic germ plasm lines were converted and screened to determine mechanisms of water use efficiency in cotton (Roark and Quisenberry, 1977). Genetic differences have been observed for the ability to maintain leaf turgor under moisture deficit (Quisenberry *et al*., 1985) but the trait does not definitively translate to improved water use efficiency. Genetic variability among exotics within the *Gossypium* species for other growth-stress characteristics (Quisenberry *et al*., 1981) indicates potential improvement if important genes that could contribute to water use efficiency that might have been lost during domestication can be restored. Regardless, evaluation issues in breeding remain, as little confidence currently is given to selecting in a single environment for any physiological traits, and heritability among any of the traits individually is still lower than yield heritability (Stiller *et al*., 2005).

As commercial objectives in biotechnology research move from crop protection traits to crop production or agronomic traits, drought tolerance is a primary target for plant-based biotechnology solutions. According to publicly available movement and release permits, some biotechnology approaches are being tested in cotton. Specific strategies are confidential business information, but it is theorized that a single pathway approach may be insufficient. Transgenic approaches evaluating abscisic acid (ABA) response elements have been researched in the public sector (Kerr and Allen, 2011; Rock *et al*., 2011). Since WUE may be addressed indirectly in productivity breeding, whether a classical breeding strategy or biotechnology solution is employed, line testing over a number of locations and years is appropriate evaluation strategy, with the addition of locations that differ in water stress.

14.6 Future trends

The most visible trend in literature for classical cotton breeding is the use of DNA markers and other molecular tools. These tools are expected to not just play a role in cultivar development through marker-assisted breeding or backcrossing, but also to contribute to basic knowledge on cotton

domestication, physiological response and tolerance mechanisms and fiber development. Plant-based biotechnology solutions have been applied in cotton for insect (heliothis) resistance and herbicide resistance. Biotechnology in the form of recombinant DNA is also being pursued for more complex developmental targets such as water and nitrogen use efficiency, especially as global water use requirements increase. Looking into the future for conservation on the processing side of cotton, fiber development traits that go beyond improvement of physical spinning quality to those that enable less water use in dyeing and finishing, and less harsh substances to impart wrinkle resistance and flame retardance may be incorporated at the genetic level. Enabling technology to address the complexity of introgressing multiple biotechnology traits into acceptable cultivars is being explored, including mini-chromosomes and site-specific transformation. Classical cotton breeding in regions accepting recombinant DNA strategies must evolve to handle purity requirements of biotechnology crops while ensuring basic germ plasm improvement is not left behind.

New methods to measure fiber quality structure beyond physical properties are expected to contribute to improved fiber quality if those measurements can be modified to handle large numbers of samples in time to receive data before planting the next growing season. Technology to more accurately measure differences associated with other breeding targets, such as productivity and water use efficiency, could provide the basis for further crop improvement in classical breeding, marker-assisted breeding and biotechnology traits. In order to move successfully into marker-assisted breeding, research to develop the enabling technology must be accomplished with accurate phenotyping.

Conversely, another developing trend is cotton varieties specifically suited to organic, subsistence or sustainable production systems. Cotton is not primarily a food crop, but seed from organic farms is a good feed source for organic dairies and 'natural' beef processors. Organic cotton is a market option for consumers who wish to use their purchasing decisions to encourage sustainable production where it is feasible. In countries where biotechnology is widely accepted, such as the USA, Australia, India, Argentina, Brazil, South Africa, and to a lesser extent, China, the trend has been for seed developers to transition out of offering non-biotechnology varieties for sale. Since most organic certifying agencies do not allow use of varieties that contain recombinant DNA traits, this allows fewer options for organic growers and a threatened pipeline of genetic improvements through classical breeding. The trend here ironically mirrors the first one, that genetic and molecular tools will become available to assist breeders to exploit the rich genetic resource available in *Gossypium* and apply it to basic germ plasm improvement and ultimately the cultivars of tomorrow.

14.7 Conclusions

Much progress has been made in developing cotton for cultivation through natural selection and then breeding cultivars for commercial purposes by hybridization and stabilization. The consequences of domestication include a narrowing of the genetic base available for classical cotton breeding techniques. Molecular technologies offer new techniques to more efficiently exploit germ plasm resources as improvements in fiber quality, disease resistance and water use efficiency become more critical. Productivity and quality of output remain primary objectives and breeding programs and classical methodologies continue to be utilized in breeding for traditional or organic production systems as well as incorporating traits developed from recombinant DNA technology. The basic principles used for creating diversity, selection, stabilization and evaluation guide general methodology and dictate developmental timelines within regional objectives, technology acceptance and production systems.

14.8 Sources of further information and advice

Useful resources containing more detailed information on cotton breeding used extensively in preparation of this chapter include: John M. Munro, *Cotton*, 2nd edn. (1987), New York: John Wiley; C. Wayne Smith and J. Tom Cothren (eds.), *Cotton: Origin, History, Technology, and Production* (1999), New York: John Wiley; and Andrew H. Patterson (ed.), *Genetics and Genomics of Cotton* (2009), New York: Springer. More detail on cotton fiber is available from Amarjit S. Basra (ed.), *Cotton Fibers: Developmental Biology, Quality Improvement and Textile Processing* (1999), Binghamton, NY: Food Product Press; and Menachem Lewin (ed.), *Cotton Fiber Chemistry and Technology* (2007), Boca Raton, Florida: CRC Press. One of the best basic series of articles on the growth and development of the cotton plant, 'How a Cotton Plant Grows', appeared in *Progressive Farmer* magazine in 1982.

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14.10 Appendix: abbreviations

ABA	abscisic acid
BCxFx	backcross number and filial generation
CIRAD	Centre de cooperation internationale en recherché agronomique pour le developpement
DD60	degree days based on 60° Farenheit
DNA	deoxyribonucleic acid
EST	expressed sequence tags
f.	fusarium
f. sp.	forma specialis
Fx X	filial generation
FAS	Foreign Agriculture Service
ha	hectare
HFI	horizontal fruiting index
pv.	pathovar
RKN	root-knot nematode
sp.	species
USDA	United States Department of Agriculture
USDA-ARS	United States Department of Agriculture-Agricultural Research Service
VFI	vertical fruiting index
WUE	water use efficiency

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Fibre flax cultivation in sustainable agriculture

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Abstract: Cultivation technologies based on the selection of cultivars for local environmental conditions, including proper agronomic treatments, according to principles of sustainable agriculture, are important for the reduction of economic, environmental and social costs of agricultural production. This chapter presents current knowledge and practical solutions relating to the cultivation of fibrous flax in sustainable agriculture.

Key words: fibre flax, cultivation, sustainable agriculture.

15.1 Introduction to fibre flax for sustainable agriculture

The intensive agricultural development that followed the Second World War, particularly in European countries, was characterized by a dramatic increase in production. Unfortunately, the price of this increase was overproduction, reduction of income and the growth of unemployment in rural areas. There were environmental consequences too, such as pollution from agrochemicals, reduction of biodiversity, and the greenhouse effect. Contemporary agriculture generates 70% of N_2O, and 50% of CH_4 (Francis *et al*., 2006; Pretty and Smith, 2003). Most technologies used in traditional farming fail to solve conflicts between economical, social and environmental production objectives (Allen, 1993; Common and Perrings, 1992; Francis *et al.*, 2006).

The concept of sustainable development has been presented in detail in Agenda 2001, the conference proceedings from the Earth Forum in Rio de Janeiro in 1992. This document called upon the whole world to undertake every effort to ensure sustainable development.

A simple yet explicit definition of sustainable development was proposed by Brundtland (1987). According to this definition, sustainable development 'meets the needs of the present without compromising the ability of future generations to meet their own needs'.

In 1990, the US government defined sustainable agriculture in Public Law 101-624, Title XVI, Subtitle A, Section 1683, as 'an integrated system of plant

and animal production practices having a site-specific application that will, over the long term, satisfy human food and fiber needs; enhance environmental quality and the natural resource base upon which the agricultural economy depends; make the most efficient use of nonrenewable resources and on-farm resources and integrate, where appropriate, natural biological cycles and controls; sustain the economic viability of farm operations; and enhance the quality of life for farmers and society as a whole' (Sustainable Table: www.sustainabletable.org/intro/whatis/).

One crop that seems especially suited to sustainable cultivation is fibre flax. The scientific name of flax – *Linum ussitatissimum* L. (Kolodziejczyk and Fedec, 1995) – is itself the best explanation for the usability and importance of this crop. The Latin word 'ussitatus' translates as useful, important. The superlative degree of this adjective is 'ussitatissimus', thus, flax should be considered the most useful, the most indispensable plant. The question is, can this 'most useful' crop be produced sustainably? (Heller and Biskupski, 2002).

Flax, is a renewable, biodegradable, environmentally sound raw material with excellent multifunctional performance (Harwood *et al.*, 2008). These factors ease the strain between social, economic, and environmental objectives of production resulting from classical practices (Burger *et al.*, 1995). Fibre flax cultivation technologies for sustainable agriculture should be based on highly effective, regionally adapted cultivars, which are resistant to biotic and abiotic stress. Widespread adoption of low-input, high-tech management systems is also of great importance, as is the increasing reliance on professional consultants and contract work (Fernandes-Quintanilla *et al.*, 2008). Good quality, stable, high yields are crucial to the technological usefulness of cultivars for industrial applications, and therefore add economic value. Applied cultivation treatments should correlate with the development cycle of flax (Marshall *et al.*, 1988), including meeting the environmental requirements of plants, in terms of nutrients, weather and soil.

The yield and its quality are the sum of 'individual yields', obtained from individual plants which make up the population of a particular field. Effective, sustainable cultivation must take into account the specific morphological, anatomical and ontogenetic properties of flax plants, plus the impacts of environmental factors (soil, climate, agronomy) on crop development (El-Hariri *et al.*, 2004; El-Shimy *et al.*, 1997; Heller, 2005).

15.2 Flax growth cycle

Agronomic treatments are crucial to the sustainable cultivation of flax. These treatments must be administered at optimal, precisely set times, which are determined by growth stage. Herbicides should be applied at precisely the time when weeds are most susceptible, that is, when the plants are young, so

that lower doses can be used. The same rule applies to disease management: the sooner fungicides are applied, the more effective the treatment, and the lower the minimum effective dose.

In classical flax ontogenesis the following growth stages have been identified: germination, 'herring bone' stage, fast growth stage, flower bud formation, flowering, green maturity, green-yellow maturity and full maturity. Assessing flax development by this method is imprecise, because each stage covers a long period of time, and includes several sub-stages.

Recently, a more exact method for flax development assessment has been developed. This method is consistent with the BBCH scale. The BBCH scale is a system for the uniform coding of phenologically similar growth stages of all mono- and dicotyledonous plant species. The BBCH key is a decimal system, with 10 principal growth stages and up to 10 secondary ones, starting with seed germination, then progressing through leaf production and extension growth to flowering and senescence. It is therefore a suitable tool for assessing the growth stages of most cultivated crops and weeds, created by uniting many different scales. The abbreviation BBCH derives from Biologische **B**undesanstalt **B**undessortenamt und **CH**emical Industrie (Lancashire *et al.*, 1991; Witzenberger, 1991). The usefulness of the BBCH scale lies, first of all, in its accuracy (Table 15.1). The BBCH scale allows flax farmers to carry out the treatments at optimal times.

15.2.1 Plant development of flax: ontogenesis and morphogenesis

Fibre flax is a spring crop, characterized by a relatively short vegetation period, which in European conditions (average latitude: 48–52°N) lasts on average 90–120 days. The growth and development rate of flax is preconditioned by specificity of grown cultivars and local environmental conditions (weather, soil), as well as the agronomic solutions applied. The author's observations over several years (Heller, 2005) showed that low temperatures and wet weather prolong the vegetation period. A positive correlation exists between the length of the vegetation period and yield size, and also yield quality (Easson, 1989).

The period between sowing (BBCH 00) and full germination (BBCH 11) lasts 6–12 days. Plants usually reach BBCH 14 (4 pairs of true leaves) 25–30 days after sowing, and the so-called fast growth stage (BBCH 32) after 35–40 days. The bud formation stage (BBCH 51) begins after 50–55 days of vegetation, then full bloom usually takes place after 60–65 days. Plants typically begin reaching the green maturity stage 70–80 days after sowing. Flax is usually pulled out at green-yellow maturity stage (BBCH 83) after 90–120 days of vegetation. Fibre flax plants switch from vegetative development to

Table 15.1 The BBCH scale of growth stages of flax (*Linum usitatissimum* L.)

Code	Description of growth stages
Principal growth stage 0: germination	
00	Dry seed
01	Beginning of water imbibition, beginning of seed swelling
03	End of water imbibition, end of seed swelling
05	Radicle (root) emerged from seed
06	Elongation of radicle (first root), formation of root hairs and lateral roots
07	Hypocotyl with cotyledons breaking through seed coat
08	Hypocotyl with cotyledons growing towards soil surface
09	Cotyledons break through soil surface
Principal growth stage 1: leaf development (main shoot), young plant elongation	
10	Cotyledons completely unfolded
11	First leaf pair unfolded
12	2 leaf pairs unfolded
13	3 leaf pairs unfolded
14...	Stages continuous until...
19	9 or more true leaf pairs unfolded
Principal growth stage 3: shoot development	
32	Stem 20% of final length (20 cm)
33	Stem 30% of final length (30 cm)
34	Stem 40% of final length (40 cm)
35	Stem 50% of final length (50 cm)
36...	Stages continuous until...
39	Stem 90% of final length
Principal growth stage 5: inflorescence emergence	
51	First flower buds visible
55	First individual flowers visible
59	First flower petals visible
Principal growth stage 6: flowering	
60	First flowers open (sporadically)
61	Beginning of flowering: 10% of flowers open
62	20% of flowers open
63	30% of flowers open
64	40% of flowers open
65	Full flowering: 50% of flowers open, first petals may be fallen and dry
67	Flowering finishing: majority of petals fallen or dry
69	End of flowering: capsules of flax set visible
Principal growth stage 7: development of flax capsules	
71	10% of flax capsules have reached final size
73	30% of flax capsules have reached final size
75	50% of flax capsules have reached final size
77	70% of flax capsules have reached final size
79	Nearly all flax capsules have reached final size

(*Continued*)

Table 15.1 Continued

Code	Description of growth stages
Principal growth stage 8: ripening of flax capsules	
83	Green-yellow maturity of flax. Stems are yellow up to 1/3 of height. Leaves fallen down from up to ¼ of stem height. Flax capsules set beginning to yellow.
85	Yellow maturity of flax. Stems are yellow, leaves fallen down from up to 2/3 of stem height. Flax capsules are yellow. Seeds fully emerged and beginning to brown.
89	Fully ripe. The straw is dark yellow. Capsules and pedicels are brown. Seeds are dry.
Principal growth stage 9: senescence	
97	Plant dead or dormant
99	Seeds are harvested, dormancy stage

reproductive development once they are 14 cm tall (BBCH 16). The growth of flax plants is nonlinear.

The highest dynamics in plant shoot development were observed between the 30th and 70th day of flax growth, i.e. from BBCH 32 to BBCH 71. The largest increase in biomass was observed from BBCH 32 to BBCH 71.

The formation of fibres in flax begins between stages BBCH 32 and BBCH 33, i.e. when flax is 20–30 cm tall. At green maturity stage (BBCH 71–77), when seed bolls are formed, fibres cease to form.

15.3 The role of cultivars in sustainable flax cultivation

The use of highly efficient cultivars is very important in the sustainable cultivation of flax fibre. Such cultivars are characterized by good resistance to biotic and abiotic stress (Fouilloux, 1989). From the breeder's point of view, the most important traits of fibre flax are inherited to a degree, but are also strongly influenced by environmental conditions such as soil, climate and agronomic treatments (Bi-Fu *et al*., 2002).

Flax breeding programmes (see this volume Chapter 12) are aimed at developing cultivars resistant to biological stresses in the environment (i.e. diseases), and also at increasing resistance to abiotic stresses, including extremes of temperature and moisture (Hoffmann, 1979). Periods of insufficient moisture have a negative effect on yield quality and quantity. Excess moisture, especially when sowing densities and nitrogen content in the soil are high, can result in lodging.

In breeding for protection against disease, priority is given to developing cultivars resistant to strains of fungi of *Fusarium oxysporum*, *Melmpsora*

lini and *Erysiphe polygoni* (Kozłowski *et al.*, 2000). For instance, among cultivars resistant to Fusarium wilt, the following Polish examples should be mentioned: Artemida, Modran, Nike, Atena and Sara.

An interesting direction in breeding is the development of cultivars resistant to low air temperatures (Dodd *et al.*, 2000). French cultivars Adelie and Boreal can be planted in autumn, and are characterized by higher frost tolerance.

Other important developments are tolerance to drought and early maturing cultivars (Zwinger, 2000). The French cultivar Diane and Polish cultivar Nike are highly resistant to moisture deficiencies. Varieties with short vegetation periods are the Finnish Helmi and Martta.

15.4 The importance of crop rotation

Proper crop rotation is essential for growing fibre flax in accordance with the requirements of sustainable agriculture (Raymond *et al.*, 2010), which focus on limiting environmental costs (mineral fertilizers, crop protection chemicals) as well as economic and energy costs (Zika-Prandl, 2008). A good forecrop reduces the need for mineral fertilizers and pesticides, and is crucial to reducing of the number of tillage treatments. The right forecrop can decrease the costs of flax cultivation in terms of energy and money (Sultana, 1983). In Europe, cereals make good forecrops for flax, especially oats, which accelerate the process of soil self-cleaning from Fusarium wilt. Another good choice is root crops, which leave the soil clean, free of weeds, and with good structure. Potatoes are especially suitable, because they do not use up too many nutrients or too much moisture, and also limit the occurrence of Fusarium wilt. Potato crops are harvested early, allowing for good soil preparation in autumn. Beets, due to their long tap roots, loosen deeper layers of the soil and so allow flax to form stronger root systems. However, in dry conditions this can be a disadvantage, because beets use up moisture from deeper layers of the soil, leaving insufficient water for flax cultivation.

A basic rule that should be followed when growing flax: it should not be grown on the same field more than once in 7 years. This is the time required for the soil to clean itself of Fusarium pathogens. Following this rule will ensure high yields and pathogen free plants.

15.5 Flax cultivation requirements

The factors crucial for good yields of fibrous flax are: growing cultivars adapted to regional climatic conditions and applying proper agronomic regimes in terms of soil cultivation, fertilization, sowing rate and technique, post emergence treatments as well as on time and properly conducted flax pulling and dew retting.

15.5.1 Weather conditions

Fibre flax should be grown in areas where the annual precipitation is at least 600–650 mm, and where at least 110–150 mm of rain falls in the vegetation period.

Flax plants transpire very high amounts of water. The transpiration coefficient is the amount of water necessary to produce one unit of dry matter. In flax, this, is very high: 400–600.

The amount of moisture available during the vegetation of flax is a major factor in determining yield level and quality. Fibre flax does not require high temperatures. Indeed, high temperatures during vegetation clearly have a negative effect on growth and development. Moderate temperatures (18–20°C) and the accompanying cloud cover promote high yields (Xinwen, 1997). Mild solar operation contributes to good stem growth, which results in a good anatomical stem structure and high long fibre efficiency (Agosti *et al*., 2005).

15.5.2 Soil preparation

Optimum yield conditions can be achieved by sowing flax on fertile, medium-heavy soils, in good culture, particularly humus sandy clay soils that create no crust, and with regulated water/soil/air ratio.

Flax can also be grown on newly cultivated post-forest, post-pasture, post-meadow (even if used for many years) and set aside soils.

Soil requirements for flax can be summarised as follows:

- Soil should be deep and have a loose lumpy structure, allowing for air access to roots and soil microflora, as well as the ability to evacuate excess water.
- Soil should show a sufficient sorption ability (ability to absorb and sustain water and nutrients contained in it, namely the ability to economically manage water in the whole duration of vegetation period).
- Soil should have the proper degree of acidity – close to neutral, pH 6.5–6.9.

Fulfilling the above conditions results in slow but correct use of nutrients contained in the soil, as well as nutrients supplied in the form of mineral or organic fertilizers.

Forecrops that leave the field early, e.g. barley, require stubble-breaking as soon as possible, followed by harrowing to break up furrow slices and protect the soil against excessive water loss. Plants leaving the field late, e.g. potatoes, usually leave the soil clean and loose, therefore the harrow is applied to level the furrows followed by winter ploughing (no stubble-breaking is necessary in this case).

In order to reduce cultivation costs (energetic as well as economic), spring tillage should be limited to the most necessary treatments:

- dragging, aimed at stopping transpiration from the soil and induction of weed seeds to germinate;
- loosening soil structure with a harrow before sowing, which also destroys germinating weeds; a cultivator with stiff of semi-stiff teeth should be used, depending on the type and condition of the soil;
- rolling of soil that is too loose or dispersed, where the seeds might be placed too deep;
- pre-sowing and post-sowing harrowing with light harrows, to reduce water transpiration and cover the planted seeds (if necessary).

15.5.3 Fertilization

Being an annual spring plant, fibre flax has a poorly developed root system. Take-up of nutrients by flax is low, especially in the first period of vegetation (BBCH 00 – BBCH 14). After that period a fast growth stage follows (BBCH 32) when daily growth can reach 2–4 cm. In this stage flax takes up nutrients intensively, reaching maximum uptake at the bud formation (BBCH 51–59) and flowering (BBCH 61–69) stages. Seven tons of flax contains approximately 66 kg of nitrogen (N), 32 kg of phosphorus (P_2O_5), 120 kg of potassium (K_2O), 30 kg of calcium (CaO) and 43 kg of magnesium (MgO) (Endres *et al.*, 2002). Fibre flax takes up relatively large amounts of potassium and, unusually, takes in more magnesium than calcium.

Flax requires very precise fertilization, because particular nutrients affect qualitative features of the fibre. To ensure a good yield of high quality fibre, it is recommended to provide the macronutrients in the following ratio: N: P_2O_5: K_2O as 1:2:3 which should correspond to the following amounts: 20–40 kg N, 60–80 kg P_2O_5, 90–120 kg K_2O per hectare. Sixty per cent of the nitrogen dose should be applied before sowing, and the remaining amount should be applied after germination, depending on plant, soil and climatic conditions. After germination, nitrogen is applied during the first stage of growth, because later applications can have negative effects on fibre quality (Dempsey, 1975). Calcium should not be applied directly before sowing flax, as this can also reduce the quality of the fibre. Good quality fibre can be obtained providing at least seven nutrients are correctly applied (NPK, Cu, Zn, B and Mg). Nitrogen is necessary for growth, but excessive doses cause thickening of stems and reduce fibre strength (Mańkowski and Szukała, 1998). Despite the proven negative effect of high nitrogen doses on the quality of fibre, some publications still recommend high doses, even over 100 kg ha^{-1} (El- Hariri *et al.*, 1998).

The beneficial effect of potassium is only revealed when nitrogen is correctly applied. Excessive doses of nitrogen, however, can reduce the length of stem and fibre content. Potassium has beneficial influence on both fibre strength and elasticity, and is also vital to the dew retting process. The recommended doses of potassium vary from 50 kg ha^{-1} to 180 kg ha^{-1}.

Phosphorus is indispensable if the straw is to reach its proper length and form the proper number of fibre bundles in each stem (Grant and Bailey, 1989). Excessive doses of phosphorus, however, lead to shortening and branching of the stem, which reduces the fibre's tensile strength.

The optimum pH of the soil for flax is 6.5–6.9. Excessive doses of calcium cause fibre breaking and its lignification; direct application of calcium should therefore be avoided. Magnesium is also an important element for flax. Magnesium deficiency causes leaf chlorosis and stem shortening, while optimum supplementation ensures good technical length of straw.

When growing flax on soils that do not require the addition of calcium but have low magnesium content, it is recommended to apply magnesium fertilizers at 40–80 kg MgO per ha. Flax is susceptible to deficiencies of copper, boron and zinc. Soils with low boron content should be fertilized with single superphosphate with boron added. These fertilizers should be applied to heavy soils in autumn and to light soils in spring.

On organic-mineral or half-bog soils, on newly cultivated fields, a supplement of copper in the form of copper sulphite should be applied at 25 kg ha^{-1}.

Zinc is important for plant health. Soil pH has a significant effect on zinc assimilation: the more acidic is the soil reaction, the better the uptake of zinc. Since flax is grown on soil with pH close to neutral, it may be necessary to supplement zinc ($ZnSO_4$) at 15 kg ha^{-1} using a $ZnSO_4$ solution at 500 g ha^{-1} (Marchenkov *et al.*, 2003).

15.5.4 Sowing

In Europe, flax is sown in the period when the upper layer of soil is warmed up to 7–9°C (fenologically, when marsh marigold (*Caltha sp.*) and wood anemone (*Anemone nemorosa* L.) bloom). Depending on region, this corresponds the period from 15 March–15 April in France (Sultana, 1983), up to first decade of May in Northern Ireland. Usually, the earlier flax is sown, the longer is the vegetation period, which has a positive effect on the fibre yield and quality (Easson, 1989; Heller, 1992). In some regions, flax is cultivated as a secondary crop and sown in October or November (El-Hariri *et al.*, 2004).

Sowing method

Use of qualified sowing seeds is recommended – these are healthy seeds treated with fungicides. The optimum sowing amount of fibrous flax is:

- industrial plantations: 110–130 kg ha^{-1} (2000–2400 seeds per 1 m^2),
- seed plantations: 50–70 kg ha^{-1} (1000–1100 seeds per 1 m^2).

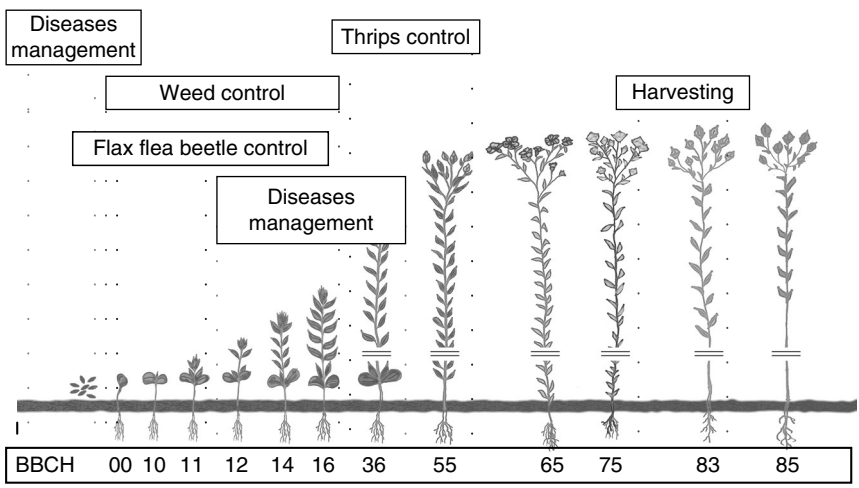
According to information available in the literature, even higher sowing amounts are used, reaching 140 kg ha^{-1} (Fouilloux, 1989) or even 170–180 kg ha^{-1} (El-Hariri *et al.*, 2004) to ensure the plant density of 3000 plants m^{-2}. However, in practice the optimum stand at harvest time is 1600–1800 plants m^{-2} (Mańkowski and Szukała, 1998).

15.5.5 Post-emergent cultivation – plant protection

See Fig. 15.1 for the scheme of plant protection during fibre flax ontogenesis.

Control of soil crust

On heavy, crusty and confluent soils, the use of a spiked roller or ring roller is recommended, and sometimes a pre-sowing light harrow.



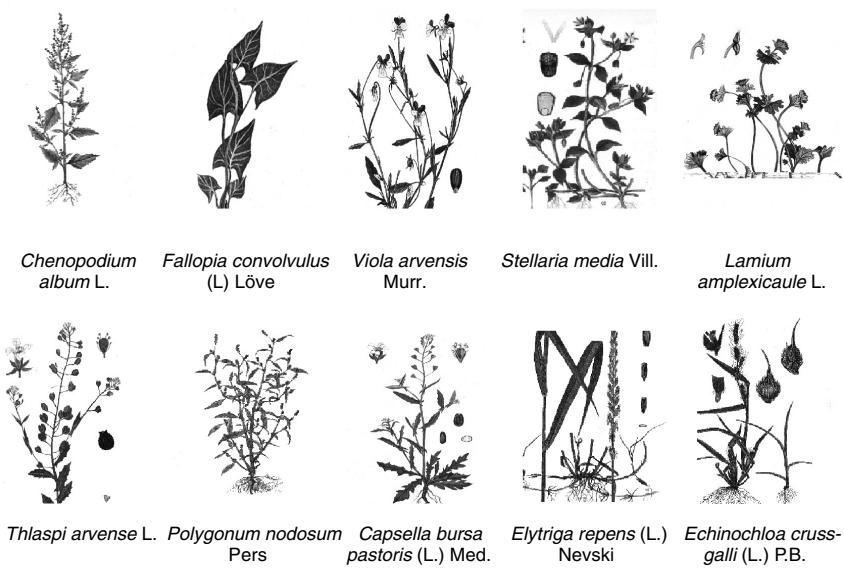
15.1 The scheme of plant protection during fibre flax ontogenesis (Kozłowski, 2006).

Weed control

Weeds compete with flax plants for nutrients and water, and overshadow the crop. Weeds also often transmit diseases and pests, cause lodging, and make pulling and dew retting of flax more difficult. Due to its delicate morphological structure (single, non-branched stem, small leaves and poorly developed root system), flax competes poorly with weeds for its life space (Hocking *et al.*, 1987; Maddens, 1989; McSheffrey *et al.*, 1992). Therefore, the degree of weed infestation has a significant effect on yield size and quality, and is crucial to the profitability of cultivation. Weed infestation accounts for 15–20% reduction in yield. The degree of weed infestation in flax determines the level of straw yield and quality of fibre, namely its divisibility, delicacy, buttery feeling, heaviness, strength, colour and uniformity. Therefore, efficiency of weed control indirectly affects fibre yield and quality (Maddens, 1989).

The predominant weeds in flax cultivation in Europe are the typical species that infest root crops and small grains. The most frequent and most numerous species in fibre flax are: *Chenopodium album* L., *Fallopia convolvulus* (L) Löve, *Viola arvensis* Murr., *Stellaria media* Vill., *Lamium amplexicaule* L., *Thlaspi arvense* L., *Elytriga repens* (L.) Nevski, *Polygonum nodosum* Pers. and *Echinochloa cruss-galli* (L.) P.B. (Heller, 2001; Maddens, 1989) (Fig. 15.2).

The basic yield in cultivation of fibre flax is straw from which the fibre is extracted. Despite significant progress in mastering the methods of



15.2 The most frequent and most numerous species in fibre flax (Heller, 1998).

cultivation, harvest and processing, no method of weed removal from straw and fibre has been developed. Flax straw, and in consequence the fibre, contaminated with weed remnants constitutes an inferior raw material. The price of such material is low and unsatisfactory for the flax grower.

According to requirements of sustainable agriculture, rational weed control should wisely combine tillage treatments (to destroy weeds mechanically) with chemical control using herbicides. Herbicides only supplement mechanical weed control methods. The efficacy of chemical weed control products depends on many factors: correct choice of herbicide and adjuvant considering the status and the degree of weed infestation, doses applied, time and method of application, and environmental conditions (weather, conditions of the crop, level of agronomy, etc.) (Heller and Rólski, 2001; Maddens, 1989).

Herbicides can be divided into pre-emergence (applied on the soil before germination of the crop) and post-emergence treatments (applied after germination).

Herbicides are classified by use: some control dicotyledonous plants (so called broad-leaf), while others (graminicides) control monocotyledonous weeds (grasses). monocotyledonous weeds (grasses).

Soil herbicides applied directly after sowing (BBCH 00) are effective if the weather in spring is warm and wet, and the soil remains in good culture. Directly after flax sowing the soil herbicides linuron or mixture of linuron and lenacil can be used.

When flax plants are 6–12 cm tall (herring bone stage – BBCH 12–14) weed control can be achieved by conducting on-leaf application of the following herbicides: MCPA, bentazone, chlorosulfuron, thifensulfuron methyl, amidosulfuron, sulcotrione, bromoxynil, metsulfuron methyl, flupyrsulfuron- methyl, linuron + chlorosulfuron and clopyralid.

Recommended graminicides for flax include: asulam, fluaizof-P-buthyl, haloxyfop-R, diclofop-methyl, chizalofop-P-etyhl, trialat, TCA-Na, chletodym, cykloksydym, EPTC, fenoxaprop-P ethyl and other products (Frisen, 1988; Heller, 1992; Maddens, 1989; Marshall *et al.*, 1995; Šmirous, 1989).

Post-emergence herbicides are more efficient when used early on younger and more susceptible weeds. Moreover, it was found that the use of herbicide mixtures and the addition of adjuvants into the spraying mixture increase their efficacy in weed control. Adjuvants are a group of chemicals that improve the evenness of plant coverage; use of a spraying mixture results in higher efficacy of herbicides and reduction of recommended dose, especially in difficult-to-wet plants.

Graminicides cannot be combined with herbicides controlling broad-leaf weeds (Friesen, 1988). To control grasses, spraying should be conducted 5–6 days before or after the broad-leaf weed control treatments are applied.

Disease management

Disease is a considerable problem in flax cultivation. Flax diseases are mostly caused by fungi. High plant density on the field (1800 plants m^{-2}) creates a micro-climate favourable to the development of pathogenic microorganisms, and promotes infections and the development of diseases. Different diseases affect flax plants in different regions, due to local soil and climatic conditions (Rashid, 2003). Diseases can be divided into two groups: the first group includes root mycosis and diseases caused by soil resident fungi, which attack root systems and plants in the initial stage of vegetation, causing rotting and dying of seedlings and older plants. The second group includes diseases of stems and leaves on which the mycelium or pericarp grow.

According to the requirements of sustainable farming, effective protection of flax against disease lies in integrating preventive measures with rationally used plant protection chemicals. The main issue is to create the optimum conditions for growth and development of the protected crop, which increases the resistance to biotic and abiotic stresses. The main factors crucial for effective protection of fibre flax against diseases are: use of certified sowing seeds, use of recommended cultivars, proper crop rotation, correct seed dressing, and application of post-emergence fungicides. The effectiveness of applied fungicides depends on the correct choice of treatment in connection to an encountered pathogen, dose, and time and method of application (Andruszewska, 2001; Beaudoin, 1989; Rashid, 2003; Sultana, 1983).

Fursarium wilt is the most frequently encountered disease in most countries in Europe, causing loss of 80–100% of yields (Beaudoin, 1989; Sharma and Mathur, 1971). The causal agent of Fusarium wilt is *Fusarium oxysporium* Schlechtend. f. sp. *Lini* (Brayford, 1996). Protection against Fusarium wilt comes down to preventive measures (Rólski *et al.*, 2000), namely: proper crop rotation (at least a 6-year break in flax cultivation on the same field), cultivation of resistant cultivars, seed treatment with products containing such active ingredients (a.i.) as carbendazim, tiuram, cyproconazole, fludioxonil, flutriazole, thiabendazole, captam and mancozeb (Heller *et al.*, 2006).

Recommended products for on-leaf applications are based on benomyl, prochlorase, carbendazim + fluquinconazole and flutriafol. These substances effectively protect against progressive tracheamycosis, providing they are used early, when plants are 15–20 cm tall.

Another dangerous disease of fibre flax is Anthracnose, which is caused by *Colletotrichum lini* (Westerdijk) Tochinai (Rashid, 2003). It occurs all over the world in regions where flax is cultivated. It is particularly a problem in Byelorussia, Czech Republic, Lithuania, Ukraine and France. Protection of plantations against Anthracnose comes down to preventive measures: correct crop rotation, growing resistant cultivars, using pathogen-free sowing seeds, and employing seed dressing treatments containing carbendazim + tiuram,

carbendazim, flutriafol + thiabendazole, cyproconazole + fludioxonil and iprodione + carbendazim (Heller *et al.*, 2006; Jankauskiene *et al.*, 2004; Sultana, 1983). During the vegetation period, the on-leaf application of chemicals containing copper oxychloride, benomyl and prochlorase.

Septoriosis, commonly called flax pasmo, can have a significant financial impact for flax growers. The disease is caused by *Septoria linicola* (Speg.), Garassini (Convey, 1962). It is a fungus that develops in leaf tissue, seed bolls and stems, where it causes negative changes in fibre quality. In Poland, Septoriosis was a quarantine disease until recently, and so does not occur there. Rashid (2003) lists the following effective protective methods against pasmo: crop rotation (3-year break before cultivation of flax on the same field), early sowing, and use of qualified, pathogen-free seeds. Among the recommended treatments for flax protection against Septoriosis are the following, which should in most cases be applied twice during vegetation period:

- spraying treatments: benomyl, carbendazim, prochlorase;
- seed dressing: cyproconazole + fludioxonil, flutriazole, carbendazim, flutriafol, azoksystrobin, fludioxonil + cyproconazole, tebuconazole + triadimefon, flusilazole + carbendazim (Heller *et al.*, 2006).

Grey mildew (caused by *Botrytis cinerea*) is a big problem in France and England, where it is connected with high air humidity in flax cultivation areas. The recommended preventive measure is seed dressing with products based on carboxin + thiram or carbendazim. The recommended post-emergence fungicides (applied in the initial period of vegetation) to control grey mildew contain the following active ingredients: carbendazim + copper oxyquinolate, ipriodione + carbendazim, maneb, tiuram, vinclozolin, prochlorase and ipriodione (Heller *et al.*, 2006).

Fomoza belongs to a category of diseases that attack stems, leaves and inflorescence of fibre flax. Damaged tissues are then covered with mycelium containing spores or pericarps of fungi. The disease is caused by *Phoma exiqua var.* Desmas. *var. Linicola* (Naumov and Vass) Maas (Mass, 1965). In the Czech Republic, France and England the application of on-leaf fungicides based on the following active ingredients is recommended: prochlorase and ipriodione + carbendazim. Additionally in the Czech Republic it is recommended to dress the seeds with carboxin + tiuram (Heller *et al.*, 2006).

In Europe, especially in France and England, black mould is a big problem. The disease is caused by three species of fungi: *Alternaria linicola*, *A. Alternata* and *A. Infectoria* (Evans *et al.*, 1995). Rashid (2003) mentions the following preventive measures: crop rotation, cultivars resistant to black mould, and seed dressing (Singh, 1998). Effective seed dressing treatments include: ipriodione + carbendazim, copper oxyquinolate + carbendazim, carboxin + tiuram, and also on-leaf application of maneb and tiuram.

Oidium, also referred to as 'powdery mildew of flax', is caused by *Oidium lini*. It does not normally cause major financial problems, because it appears very late in the growth cycle, namely after seed bolls are formed. This is commonly the case in Europe. In case of late sowing (April, May), however, powdery mildew can destroy whole plants, or prevent them from forming seed bolls (Beaudoin, 1989; Saharan and Saharan, 1994). This happens in conditions of high humidity and temperature. In countries where such conditions occur the application of fungicides like triadimefon, benomyl, prochlorase, vinclozolin and triforine in the form of spray, is recommended (Heller *et al*., 2006).

Rust is common in regions where fibre flax is cultivated. The disease is caused by *Melampsora lini* (Ehrenb.) Desman (Burdon and Jarosz, 1992). Characteristic symptoms of rust are bright orange and powdery pustules, which develop on leaves, stems and bolls. Stems overgrown by the mycelium produce very low value, bad quality fibre. The best method (from an economical and environmental point of view) for controlling the disease is the use of resistant cultivars (Rashid, 2003).

Pest control

Plantations of fibre flax can become infested by various pests, such as flee beetles, flax thrips, cutworms and gamma moths. Polyphags, including crane flies and nematodes, can also cause problems. The biggest losses are caused by flea beetles (*Longitarsus parvulus* Payk. i *Aphtona euphorbiae* Schrank.) and thrips (*Trips linarius* Ladureu and *T. angusticeps* Uz.) (Haydock and Pooley, 1997; Jankauskiene, 2004). The choice of insecticides recommended for fibre flax covers such products as lambda-cyhalothrin, acephate, alphamethrin, beta-cyfluthrin, delta-methrin, esfenvalerate, metomyl omethoate, vamidithion and acetamipryd.

Attention to preventive measures is crucial to the sustainable production of flax (Gheorge *et al.*, 1990). For instance, early sowing reduces the risk of damage caused by flee beetle and thrips (Rashid, 2003). Moreover, using seed dressing treatments with both fungicidal and insecticidal effect, e.g. those based on tiametoksam + metalaksyl-M + fludioksonil, considerably limits the environmental costs of flax cultivation.

Biostimulators for increasing plant resistance to drought

Global warming has an enormous impact on agricultural conditions (Ziska and Bunce, 1997). Climate changes are predicted to have the following effects in the period 2001–2050:

- elongation of the growing season by about 40%;
- reduced precipitation (in summer by 0–20% and in autumn and winter by 9–13%);

- increased transpiration in summer (9–17%);
- fewer days with temperatures below 0°C (about 70%);
- a threefold increase in days with temperatures above 25°C;
- increase in UV-B radiation in summer by 3–8% and by 4% in winter;
- increase in CO_2 content in the atmosphere by 5.4% compared to the current day level (Harris and Hossel, 2001).

Many regions in Europe are suffering from a water shortage, which can be put down to general climatic changes. Lack of water is the limiting factor on yield size in these areas, particularly periodic water shortages during vegetation. Fibre flax is particularly susceptible to water deficiency. Numerous studies (Tobler, 1928; Wannemacher, 1949) have proved that water availability has an important impact on the yield of straw, seed and fibre, as well as on the quality (thinness) of the fibre obtained.

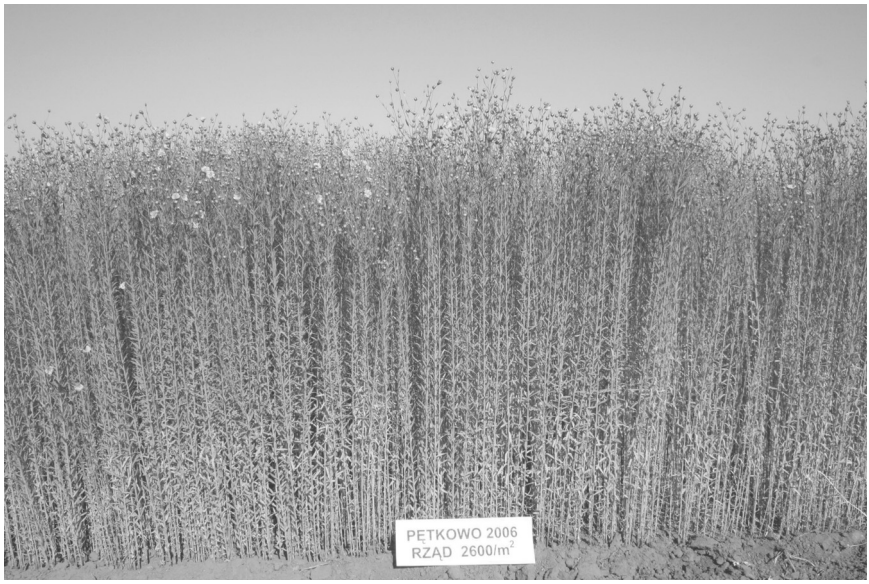
Application of ASA (acetyl salicylic acid) (0.2 kg ha^{-1}), and Atonik (0.5 l ha^{-1}) in conditions of controlled drought stress (soil moisture content at 25% field water capacity [FWC]) has a beneficial effect on growth, development and yields of flax. Correct application can result in a significant increase in total flax yield and straw yield, and increased fibre yield of 12.8–25.5% (Heller, 2005).

The application of PAA-Na and PAA-NH_4 (poly (aspartic) acid compounds) in conditions of controlled drought stress had a beneficial effect on flax growth, development and yields. The total and technical length of straw increased when PAA compounds (seed dressing and post-emergent application) were applied at a soil moisture content of 25% FWC. The application of PAA also resulted in a significant increase of total yield and straw yield. When tested, PAA compounds were shown to modify the fibre efficiency in relation to the straw yield. The best results were obtained by applying PAA compounds as seed dressing treatments (2–3 g/1 kg seeds) and by spraying post-emergent plants (500 g/ha) (Heller, 2008).

15.6 Flax harvest

In order to produce high quality fibre, it is crucial to harvest flax at the right time. Properties such as thinness, greasy feel, degree of lignification and strength are indicative of high quality flax fibre (Mańkowski and Szukała, 1998).

Time of harvest is determined by cultivar, degree of straw maturity and yield use. Flax is typically harvested when the straw is at the green-yellow maturity stage (BBCH 83) (Fig. 15.3). If the harvest is delayed, poor quality fibre is the result. There is no standard calendar date for the harvesting of fibre flax; it is too dependent on environmental conditions, which can vary widely. Instead, the BBCH scale is almost universally used.



15.3 Typically, fibre flax is harvested at the green-yellow maturity stage of the straw (BBCH 83).

In agricultural practice the following maturity stages of flax are identified:

1. *Green maturity (BBCH 75)* – takes place more or less a week after flowers fade. Stems are green all along their length, while leaves are beginning to yellow at the lower part of the stem. Seed bolls are still green and seeds are white-green and soft. Fibre is not fully formed; it is soft, weak, very thin, and greenish in colour. Flax used to be harvested at this stage in Belgium and the Netherlands to obtain the special sort of fibre needed to produce batiste and delicate embroidery.
2. *Early yellow maturity (BBCH 83)* – takes place approximately one week after green maturity. Stems turn yellowish for up to 1/3 of their height. Leaves fall from ¼ of the stem. Seed bolls begin to turn yellow. Seeds develop and begin turning yellow as well. Fibre cells are tidily packed in fibre bundles, and cell walls thicken. Lignification of fibres is still not very advanced. Harvesting flax at the early yellow stage is commonly recommended and is standard practice on commercial plantations (Fig. 15.3).
3. *Yellow maturity (BBCH 85)* – is a stage reached by plants one week after early yellow maturity stage. Stems are completely yellow; leaves have fallen from 2/3 of the stem. Seed bolls are yellow and the oldest ones turn brown. Seeds are completely formed and brown at the ends.

4. *Full maturity (BBCH 99)* – follows the yellow maturity stage approximately 10–12 days later. Straw is dark yellow, brown at the bottom, and seed bolls and peduncles are brown as well. Seeds are dry and rattle when hit. Seed bolls tend to open.

15.7 Future trends in fibre flax growing for sustainable agriculture

SWOT analysis can assist in the evaluation of the importance and prospects of sustainable fibre flax cultivation (Kotler *et al.*, 1996).

15.7.1 Strengths

One of the conditions for sustainable agriculture is differentiation of plant and animal production (Glaser, 2007). Differentiation of plant production lies in increasing the area and importance of, for example, non-food crops (Merz and Callaghan, 1997). Flax, like other fibrous plants, should play a significant role in sustainable farming (Allam, 2004; Decanniere, 1989; Heller and Biskupski, 2002; Kozłowski *et al.*, 1998; Muir and Westcott, 2003). Many countries have centuries-old traditions of flax production. Experienced farmers, together with the right environmental, economic and social conditions make sustainable production possible, including the following factors:

- suitable soil conditions for fibre flax;
- appropriate breeding, ensuring cultivation of cultivars with high economical value;
- farming technology, skill and experience;
- high yield value;
- flax fibre is a renewable, biodegradable raw material;
- flax fibre has excellent hygienic and use value (Akin *et al.*, 1999);
- flax fibre is free from pesticide residues (2–3 treatments are carried out – on average 0.25 kg of active ingredients per hectare while in cotton there are over 10 treatments);
- high value and versatility of flax products. These include raw and processed materials. Fibre, seeds and shives are the raw materials, while woven fabrics, knitted fabrics, oil, fodder, food products, semi-pharmaceuticals (with anti-cancer and anti-sclerosis action), paints, lignocellulosic boards, biocomposites, biofuels and biolubricants are included amongst the processed products. Flax is a good plant for diversifying crop rotation, because it improves soil structure;
- a favourable employment market for developments in agriculture and industry;

- possibility of reclaiming land contaminated by industrial activity – flax is a non-food plant, which can be grown on polluted soils with technical use of the yield;
- flax is a good crop for sustainable agriculture.

15.7.2 Weaknesses

Flax belongs to a group of annual spring crops characterized by their short vegetation period and susceptibility to unfavourable environmental conditions. These characteristics result in poor yield stability.

Other weaknesses include:

- fibre flax should be grown at low temperature and high levels of humidity – in many regions where fibre flax is grown the precipitation level and temperature are not sufficient to obtain high yields and good fibre quality;
- lack of research into gene expression affecting fibre formation during morphogenesis;
- in very humid conditions flax is often lodged;
- poor yield fidelity – the yield and its quality strongly depend on environmental conditions;
- it is difficult to control the process of dew retting; weather conditions affect dew-retted fibre quality;
- it is difficult to obtain large quantities of good quality fibre;
- few cultivars are resistant to drought and high temperatures;
- few flax varieties are resistant to lodging;
- it is difficult to develop environmentally friendly cultivation technologies for flax production due to the lack of non-chemical methods for protecting crops against weeds and diseases.

15.7.3 Opportunities

The following factors are crucial to the development of flax as a prospective sustainable crop (Decanniere, 1989; Kozłowski and Manyś, 1994):

- society's increasingly pro-environmental attitude;
- by 2050, the world population will have doubled, the increase of fibre consumption will reach 130 million t per annum (now 70 million t), including 38 million t of fibres;
- newly developed technologies for extraction and processing of natural fibres (new machinery and technologies);

- systematic increase in the scope of flax fibre utilization (e.g. non-textile application of flax fibre – geotextiles, etc.);
- the climatic zone in which cotton can be grown (44°N–36°S) is smaller than that of flax;
- areas turned over to cotton monoculture, e.g. in the area of Caspian Sea, suffer from degradation connected with biological imbalances;
- value of flax brand and image increases due to PR, advertising and design;
- an increasing appreciation of the advantages of natural fibres.

15.7.4 Threats

Threats associated with the cultivation and use of flax fibre have been identified (Decanniere, 1989):

- greenhouse effect – could cause droughts and high temperatures, creating conditions unsuitable for flax;
- problems with yield fidelity – it is hard to obtain large amounts of good quality raw material;
- limited awareness of the advantages of natural fibres;
- product price rather than quality is the main factor (on many market segments) when buying decisions are made;
- insufficient education and PR about the advantages of natural fibres;
- lack of market research – no accurate estimate of hidden market segments for bio-products made from fibre crops;
- conflict between crop price, and the price of raw material for industry;
- conflict between the need for lower prices and acceptable profits for producers;
- flax fibre has only a small percentage of the large world market for natural and man-made fibres; annual consumption of all fibres is 10.5 kg/capita while consumption of flax fibre is only 0.06 kg/capita;
- it is difficult to develop organic technologies for flax cultivation due to a lack of non-chemical methods of weed control.

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16

Prevention of fungal growth in natural fibres

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Abstract: The chapter discusses the problem of biodeterioration of natural fibres, examining the microorganisms causing their microbiological decomposition and the conditions enhancing their growth. Different methods of protecting textile materials based on natural fibres are also discussed, including the application of chemical and natural biocides. Applying biocides during the finishing processes of natural textile materials allows antimicrobial barrier properties to be obtained.

Key words: natural fibres, biodeterioration, biodegradation, biocides, antimicrobial finishing, bio-availability.

16.1 Introduction

Natural fibres serve as an excellent raw material for 'green products'. They are biodegradable, fully recyclable and relatively cheap to produce, and are characterized by low specific weight, thermal and acoustic parameters with favourable values, high hygroscopicity and also by a tendency not to accumulate electrostatic charges on their surface.

In specific conditions, i.e. high humidity and temperature, natural fibres and textiles (nonwovens and fabrics) are susceptible to biodegradation. The biodeterioration caused by microorganisms affects finishing and insulation materials containing natural fibres, which are used in the automotive and construction industries. Protection against biodeterioration is of great importance not only in the apparel sector, but also in the paper industry and in libraries, archives and museums, where microbiological purity of air is essential. Hygienic finishing is also of utmost importance for textiles used by humans, especially those used for medical purposes.

In unprotected materials made from natural fibres, microorganisms cause the degradation of cellulose, which is the main component of natural fibres. This leads to a loss of strength in the material and causes the emission of odours, ultimately leading to a worsening of the microbiological purity of the air.

16.2 Key issues of fungal growth, especially mildew, in natural fibres

The problem of biodeterioration affects the materials containing natural fibers that are used in automotive, building and paper industries. High humidity and temperature cause that fungi, especially mildew decompose cellulose, that is, the main component of natural fibres.

16.2.1 Microorganisms causing biodeterioration of textile materials from natural fibres

Fungi, especially mildew, play the biggest role in the biodeterioration of textile materials made from natural fibres and in the aerobic degradation of cellulose. The optimal conditions for their growth are a relative humidity of 70–90%, a temperature of 24–30°C and a pH of about 6.0. The most active fungi in this process are: *Chaetomium*, *Stachybotrys*, *Alternaria*, *Vertcillium*, *Trichoderma*, *Penicillium* and *Aspergillus.* Bacteria that prefer very high relative humidity (96–99%) and pH 6.8–8.0 are less significant in this respect. The following bacteria strains are the main cellulose decomposing agents: *Cytophaga*, *Sporocytophaga*, *Bacillus* and *Clostridium* (Szostak-Kotowa, 2004).

Mildew is a group of fungi that play an essential role, both positive and negative, in many branches of economy. Mildew is characterized by a wide range of biochemical properties. The main applications of mildew are as follows:

- biotechnological production of antibiotics, enzymes, organic acids, lipids and hormones;
- food industry (meat processing, cheese production);
- pro-ecological activity (biological processing of wastewater, composting of organic waste).

Mildew is able to produce a number of enzymes (amylase, pectinase, cellulase, proteinase, lipase, catalase), which can be used in the industrial areas mentioned above, demonstrating the positive impact of mildew on the economy. One example is cellulolytic enzymes – cellulases. Strains of *Trichoderma viride* and *Chaetomium globosum* fungi, which show cellulolytic activity, are used to test the efficiency of treatments applied on fabrics and coverings to protect them against fungi activity (Żakowska and Piotrowska, 2008).

However, mildew can also have a negative impact, decomposing food, paper, leather and wood. The materials most susceptible to biodeterioration are textiles: fibres, fabrics, nonwovens, floor coverings and carpets of both chemical and natural origin.

Microbiological decomposition of natural fibre textile materials affects both the raw material (i.e. the fibres) and fabrics and other items made of natural raw materials, including linen, hemp, cotton, jute, ramie, sisal and manila. Leather, paper and blended fabrics made of natural and chemical fibres such as polyamide, polyacrylonitrile, polypropylene, polyethylene and polyester are also affected by microbiological decomposition.

The mildew forms most often found in textiles are shown in Table 16.1 (Zyska and Żakowska, 2005).

16.2.2 Methods of determining the resistance of natural fibre textile materials to the action of mildew

In studies dealing with the resistance of textiles to mildew action the following methods are the most commonly used:

- *The agar method*, which allows the resistance to fungi of cellulose and cellulose derived products to be determined, and enables evaluation of anti-fungi finishing. This method is also used for testing the efficiency of textile finishing against selected test fungi and comparing various types of finishing *in vitro*. The following mildew strains are employed in this method, all of which are harmful to human health: *Aspergillus niger van Tieghem*, *Chaetomium globosum Kunze*, *Gliocladium virens Miller*, *Paecilomyces variotii Bainier*, *Penicillium ochrochloron Biourge*. The samples are subjected to incubation in a chamber, at a temperature of 29 ± 1°C and relative humidity of 90 ± 5%, for 4 weeks. After the

Table 16.1 Mildew most often found in textiles

Type of material	Fungus species
Fibres, fabrics and items made of natural raw materials	*Chaetomium globosum, Aspergillus niger*
Viscose fibres	*Chaetomium globosum*
Fibres, blended fabrics containing natural and chemical fibres	*Aspergillus niger, Penicillium funiculosum, Chaetomium globosum*
Leather	*Aspergillus niger, Penicillium wortmannii, Chaetomium cupreum*
Paper (including: wallpapers, gypsum-cardboard panels, roofing board, library materials)	*Aspergillus flavus, Aspergillus fumigatus, Aspergillus versicolor, Aspergillus niger, Penicillium chrysogeum, Penicillium funiculosum, Chaetomium globosum, Trichoderma viride, Cladosporium herbarum*

test, mildew growth on the samples is evaluated both visually and with the use of a stereoscopic microscope according to a 6-degree scale (see Table 16.2). If no visible mildew is observed, the area of growth inhibition is tested on the agar near the sample. Then the breaking strength of the materials is tested by checking the breaking force (Standard EN 14119:2003).

- *The soil burial test*, which is aimed at determining the resistance of textile materials containing cellulose to the action of microorganisms occurring in the soil. The test is used for comparison of protected and unprotected samples of the same material. The material tested is buried in soil with the required biological activity for a given period, after which the breaking strength is tested. The method allows a comparison of the breaking strength before and after the material is buried in the ground. The buried samples are subjected to incubation at a temperature of 29 ± 1°C and relative humidity from 90% to 100%, for a period of no longer than 9 days, up until the point at which the unfinished samples show approximately an 80% drop in breaking strength. The final assessment in the soil burial method also involves visual evaluation with a microscope that allows changes in colour, structural surface damage, loss of cellulose fibres and overall appearance of the sample to be observed in light (Standard EN ISO 11721-1:2001).

With regard to resistance to bacteria, there are two basic methods used:

- The *quality screening method*, where the bactericidal properties of the material are tested using the following bacteria species that pose a health risk to humans: *Staphylococcus aureus* (Gram positive bacteria) and

Table 16.2 Evaluation of fungal growth on the samples

Growth degree	Evaluation
0[a]	No visible growth evaluated microscopically
1[a]	No visible growth evaluated with naked eye, but clearly visible microscopically
2	Growth visible with naked eye, covering up to 25% of tested surface
3	Growth visible with naked eye, covering up to 50% of tested surface
4	Considerable growth, covering more than 50% of tested surface
5	Very intense growth, covering all tested surface

[a] If the fungal growth on agar around the tested sample is partially or completely inhibited, then the inhibition zone must be measured.

Klebsiella pneumoniae (Gram negative bacteria). Other suitable species can also be used, including the Gram-positive bacteria *Corynebacterium xerosis*, *Bacillus licheniformis*, *Micrococcus flavus* and *Staphylococcus haemolyticus* and the Gram-negative bacteria *Escherichia coli* and *Pseudomonas aeruginosa*. Before the test, 25 × 50 mm samples of the material are soaked in 0.2 mL of fresh synthetic sweat with an acidic pH. Then linear inoculation with the above-mentioned bacteria is carried out on the Trypticase Soy Agar (TSA). The stripes of the tested materials soaked with synthetic sweat are placed on the TSA medium, perpendicular to the line of bacterial inoculation. Samples are placed in an incubator at a temperature of 37 ± 2°C for 24 h. After completion of the test, the growth inhibition zone is shown on the bottom side of the sample (Standard AATCC 147-1998).

- The *quantity method*, which tests the antibacterial activity of the material against the microorganism that shows the highest resistance. In this test four 20 × 20 mm samples are inoculated with 0.1 mL of bacterial suspension and then incubated at 37 ± 2°C. After 2 h they are rinsed in a sterile physiological salt solution, and centrifuged for 15 min at a speed of 150 rpm. After this preparation, a series of dilutions are prepared and each is inoculated at 0.1 and 1 mL on a sterile Petri dish. The samples soaked in agar are incubated at 37 ± 2°C, for 24 h, and finally all growing bacteria colonies are calculated. After the test both the bacteriostatic activity (growth inhibition) and the bactericidal activity of the textile materials protected with biocides are tested **(**Standard AATCC 100–1998).

16.3 Methods of preventing fungal growth, especially mildew, in natural fibres

In high humidity conditions natural fibres require protection against biodeterioration caused by fungi, especially mildew. The methods of preventing fungi growth involve different approach to the problem, for example, can be based on chemical and genetic modification of natural fibres, and use of chemical or natural biocides.

16.3.1 Protection of natural fibre textile materials against growth of microorganisms

Of all microorganisms, mildew forms show the greatest ability to decompose cellulose in a natural environment. Mildew growth can occur during, for instance, a poorly controlled production cycle or as a result of incorrect storage of materials. The main factor causing mildew growth is high air

humidity of over 70%. This study has shown that the main negative effect of microbiological decay of textiles is strength reduction. The bast fibres are characterized by low resistance: after 16 days of testing the tensile strength was reduced by 83–96%. For cotton fabric with unit weight of 250 g/m^2, tensile strength was reduced by 82–98% after 14 days of testing. For cotton fabric with unit weight of 910 g/m^2, the tensile strength reduced by 92% after 6 days of soil burial testing. Viscose fibres showed a complete loss of strength, while cotton fibres retained 50% of their initial strength. With cotton and viscose fabrics with a unit weight of 880 and 930 g/m^2, slower decomposition was observed using the soil test than that observed for cotton fibres. Man-made fibres and fabrics show higher resistance to mildew. After 240 days of testing the strength of polyacrylonitrile fibres was reduced by 5–13%, and the elongation was also reduced by 15–32%. After the same test period, the strength of polyester and polyamide fibres was reduced by 20% and 18%, respectively, and an increase in swelling and reduction in elongation was also observed. Tests conducted on floor coverings made of polyvinyl chloride on a natural fibre backing such as jute felt, flax-hemp backing and also on backing made of polyester fibres and synthetic fibre waste showed no resistance to cellulolytic fungi after as little as 14 days of testing. Tests conducted on carpet covering on a backing made of natural fibres in the form of natural and synthetic fibre mesh, and on a backing made of synthetic fibres and polyurethane foam, mostly showed no resistance to growth of mildew fungi after 4 weeks of testing. Only the floor covering on a polyurethane foam backing proved resistant to mildew growth (Zyska, 1999).

For this reason natural fibres and natural fibre products require protection against microbiological decomposition. Protection may be provided simply by coating the cellulose, by chemically or biochemically modifying the cellulose, or through the use of chemical inhibitors such as fungicides and biocides from plant origin.

16.3.2 Methods of protecting natural fibre textile materials against mildew growth

Chemical and genetic modification of natural fibres

Cellulose, the main component of natural fibres, can be modified by chemical reaction so that it is no longer susceptible to microbiological attack.

Chemical modification of natural fibres includes the following methods, among others:

- *Acetylation* – the method is based on the reaction of lignocellulosic material with acetic anhydride at elevated temperatures, usually without a catalyst. The acetylation strongly reduces hydrophilicity and in fact can make the material hydrophobic. In addition, modification

of this sort causes bulking of the cell wall and renders the material less susceptible to biological decay. Biological protection is afforded by lower equilibrium moisture content. Moreover, the cell wall polymer chemistry is modified, so that the fibres are not recognized by enzymes (Pott, 2004).

- *Mercerization* – this method leads to an increase in the amount of amorphous cellulose at the expense of crystalline cellulose. The most important modification that this method is designed to achieve is the removal of hydrogen bonding in the network structure. The following reaction proceeds as a result of treatment with alkalis: Fibre–OH + NaOH → Fibre–O-Na+ + H_2O. The effect of the treatment with NaOH depends on the concentration of the alkaline solution, its temperature and the duration of the treatment. Optimum conditions for mercerization increase the degree of bonding at the polymer–fibre interface and lead to a rise in the rupture stress of cellulose (Kozłowski and Władyka-Przybylak, 2004). These treatments are not fungicidal, but they are preservative and as such they compete with fungicidal treatments.

Studies on the protection of cotton and cotton-polyester fabrics (100% cotton, 52% cotton and 48% polyester, 33% cotton and 67% polyester) were conducted, with both raw and finished fabrics tested. The finishing covered: dressing removal, mercerization, bleaching, washing and applying end-use finish. The resistance of cotton and cotton-polyester fabrics to mildew growth was determined by a soil test. The degree of biodeterioration was assessed after 1, 2, 5, 8 and 11 weeks. The evaluation of the effect of microorganisms was based on the extent of reduction of tearing strength. Cotton fabrics, both raw and finished, had undergone complete degradation after only two weeks. The most resistant was the blend of 33% cotton and 67% polyester fibres (Szostak-Kotowa *et al.*, 1988). In other studies, plain-wave 100% cotton fabric was finished with a non-formaldehyde containing product based on imidazolidinone: 1,3-dimethyl-4,5-dihydroxyethylene urea. In pre-treatment processes the fabric was bleached in an H_2O_2 bath, mercerized in NaOH solution and neutralized with diluted CH_3COOH solution. The resistance of finished and unfinished cotton fabrics to the action of soil microorganisms was again determined by the soil burial test. After 12 days of testing, the unfinished sample was completely degraded. The finished sample was undamaged, although a colour change was observed on the sample surface. Smaller colour changes indicated that the finished samples of cotton fabric are less susceptible to biodeterioration than the unfinished samples (Tomšič *et al.*, 2007).

Genetic modification of natural fibres is a new area of research which might allow cellulose fibres to be developed that are genetically resistant

to the destructive action of microorganisms. The results of studies on the resistance of polyhydroxyalkanoates (PHA) to mildew and bacteria are not currently available. More data concerning genetic modification of natural fibres can be found in Chapter 17 of this volume.

Use of chemical biocides

Unfortunately, most chemical biocides that have long been used in the textile industry pose environmental hazards and health risks. Cooper fungicides, e.g. copper naphtenate, copper hydroxynaphtenate and copper 8-quinolinolate, were widely used in the 1940s and 1950s. In the 1960s copper borate solubilized for aqueous application in a complex with zirconium was developed. This was applied on a denim cotton fabric and subjected to a soil test. After 5 weeks of testing a 100% fabric strength preservation was observed. Similar fabric tests conducted in a 6-month trial showed a 60% reduction of strength, yet no visual change in appearance. For comparison, the cotton fabric samples retained only 25% of their initial strength and intensive growth of mildew was observed (Block, 1967). The current biocide market for the textile applications is presented in Table 16.3 (Libudzisz and Kowal, 2000; Szostak-Kot, 2005).

Tests were carried out on cotton fabrics protected by different biocides (Afrotin LC, Mystox ELN, Mystox WFA, Antiback MF, Antiback MFB, Fungitex OP, Fungitex ROP). The fabric samples were washed three times and exposed to *Aspergillus niger*. The best results were observed for fabric protected by Fungitex OP (benzimidazole derivatives), which showed no mildew growth after three washes (Seventekin and Ucarici, 1993). Tests were also carried out on flax fibre and nonwovens protected by Cupramina B, a market product, based on boron and copper compounds. High efficiency was obtained for these materials, which showed no mildew growth, no loss of breaking force and no change in odour. Cupramina B is characterized by

Table 16.3 Biocides used in the textile industry

Trade name	Active ingredient
Tolcide C 30	2′(thiocyanmethylthio) benzothiazole
Preventol GD	2,2′-dihydroxy-5,5-dichlorodifenylmethane
Preventol O extra	o-phenylenephenol
Myacide	2-bromo-2-nitropropane-1,3-diol
Cunilate 2419-75	8-hydroxyquinolinene copper
Giv Gard DXN	6-acetoxy-2,4-dimethyl-1,3-dioxane
Densil P	dithio-2,2′-bis(N-methylbenzamide)
Mystox LPL	5-chlorophenol laurynian
Fungitex ROP	bis(chlorophenylhydroxy)methane
Sanitized BSC	thiobendazol

a low toxicity for humans and warm-blooded animals and can be used in interiors designed for human habitation or long-term use. Consequently it is widely used to protect natural nonwovens and wood for the automotive and building industries (Kozłowski and Walentowska, 2006; Walentowska and Kozłowski, 2006).

A study was carried out on the anti-fungal efficiency of chemical biocides for aerial disinfection by thermal fogging in libraries and archives. The fungi strains used were those most frequently found as contaminants in these environments, namely: *Aspergillus niger*, *Aspergillus ustus*, *Aspergillus flavus*, *Paecilomyces variotii*, *Trichoderma viride*, *Chaetomium globosum*, *Myrothecium verrucaria*, *Stachybotrys atra*, *Cladosporium herbarum* and *Penicillium chrysogenum*, as well as combinations of these. The following biocides were tested: Econazole, Orthophenylphenol, Imazalil and Thiabendazole. Orthophenylphenol and Imazalil at concentrations of 3% and 5% respectively were the most efficient at protecting against both individual fungi strains and the mixture of 10 tested strains. The effect of Thiabendazole on the properties of paper was also tested. No significant effect on the fibre strength or level of oxidation was observed. Thiabendazole at a concentration of 10% proved very efficient at both air purification and disinfection of surfaces. As well causing no damage to paper, Thiabendazole is also known to cause no visible damage to varnished or painted surfaces and metal shelves. Additionally it does not leave greasy stains on surfaces or an unpleasant odour in rooms (Rakotonirainy *et al*., 1999).

Other related data can be found in Vol. 2, Chapter 14 'Antimicrobial natural fibres' of the *Handbook of Natural Fibres*.

Use of natural biocides

Of the natural biocides, which have become the centre of interest in recent years, the most important are plant extracts and oils and chitosan. The bactericidal and fungicidal properties of essential oils have been well known for some time. They are particularly widely used for sanitary and cosmetic applications, in the pharmaceutical and food industries and for plant protection. Active substances (alkaloids, flavonoids, terpenes, tannins) that are present in essential oils such as those from thyme, oregano, clove, sage, camomile and mint have natural antimicrobial properties (Rios and Recio, 2005). Bakkali *et al.* (2008) claim there are about 3000 essential oils known to science, of which 300 are of economic importance; they are complex natural mixtures which can contain about 20–60 components at high concentrations (20–70%) with other components present in trace amounts. For example, carvacrol (30%) and thymol (27%) are the major components of essential oil from *Origanum compactum*, while menthol (59%) and menthone (19%) are the main components of essential oil from *Mentha piperita*. These major

components determine the biological properties of the essential oils. The influence of essential oils from sage, mint, hyssop, camomile and oregano on the growth of Gram-negative and Gram-positive bacteria was studied. In the case of sage, mint, chamomile and hyssop bacteriostatic activity was observed. Oregano oil shows both bacteriostatic activity and bactericidal activity, most likely due to its high content of phenolic compounds (Marino *et al*., 2001).

Chitosan is a natural, non-toxic and biodegradable natural polysaccharide. The only difference between chitosan and cellulose is that the former has an amine group in the C-2 position while in the latter this position contains a hydroxyl group (Sivaramakrishnan, 2007). Chitosan is a deacetylated derivative of chitin, which is derived from marine shells and molluscs. The antimicrobial activity of chitosan is also used for other purposes, including improving dyeing performance, as a deodorant, and as an anti-static agent. However, a major limitation in their application in the textiles field is that they are only effective against microorganisms at high concentrations, when they are able to form a film on the surface of the fabric, decreasing air permeability (Joshi *et al*., 2009).

16.4 Future trends

A new generation of biocides – with relatively low toxicity to human and environment – are subject of scientific research and set future trends. Application of plant derived biocides, modification by polyhexamethyleneguanidine hydrochloride (PHMG) and silver nanoparticles open the possibility to obtain antimicrobial barriers for natural fibres.

16.4.1 Developments in use of biocides for the protection of natural fibre textile materials: environmental and health considerations

New regulations have certainly limited the application of many commonly used biocides. Therefore there is growing demand for the replacement of old and sometimes environmentally unsafe compounds with newer more environmentally friendly alternatives. Furthermore, EU Directive 98/8/EC dealt with the introduction of biocidal products into the market and required the withdrawal of products based on substances that are exceptionally toxic to humans and the environment. The development of ecological biocides is especially important because of the REACH system (**R**egistration, **E**valuation and **A**uthorisation of **Ch**emicals), introduced by the EU (EC Regulation No. 1907/2006 of the European Parliament and of the Council of 18 December 2006). The REACH specifies the principles of

using chemical substances in order to increase environmental safety and eliminate health hazards.

Other compounds with low toxicity that are effective in protecting against biodeterioration are quaternary ammonium salts (QACs) which are widely known to be bioactive and have high anti-microbial activity. The first quaternary ammonium salt was obtained by Menschutkin in 1890 (Menschutkin, 1890). In 1916, Jacobs discovered that QACs had antibacterial properties (Jacobs, 1916). These compounds were first used in disinfection in 1935 (Domagk, 1935), and in 1977 for wood preservation (Butcher, 1977; Butcher *et al.*, 1977). In 1997, Seddon discovered ionic liquids, which are a new group of QACs (Seddon, 1997). Ionic liquids usually consist of an organic cation and an inorganic or organic anion, which are liquids at temperature below 100°C. These salts have a number of interesting properties, namely: a wide liquid range of about 300°C, non-volatility, ability to act as solvents for organic and non-organic compounds, high thermal stability, high ionic conductivity, anti-electrostatic properties and easy recyclability; as a result, they are of increasing interest. Studies have also proved that ionic liquids are characterized by antimicrobial activity (Pernak *et al.*, 2003, 2004a). These compounds were successfully used in protection of cellulose materials like wood (Pernak *et al.*, 2004b) and paper (Przybysz *et al.*, 2005) against biodeterioration. The antimicrobial activity of ionic liquids strongly depends on their structure, and especially on the type of anion they contain. $(NO_3)^-$ and $(NO_2)^-$ salts are very effective agents against bacteria and fungi (Pernak *et al.*, 2006). Among the quaternary ammonium compounds, quaternary silicones like 3-trimethoxy-silylpropyl-dimethyloctadecyl ammonium chloride have been used as a durable odour preventative on socks. However, it is less than 90% effective on bacteria and has limited activity against fungi (Sivaramakrishnan, 2007).

The research on eco-friendly antimicrobial finishing of natural textiles has also covered silver nanoparticles. Metallic silver combined with zeolite and dispersed in the polymer before extrusion spinning provides a polyester fibre that can be intimately blended with cotton to produce a durable antimicrobial composite with excellent antibacterial and anti-fungal properties (Sivaramakrishnan, 2007). The antimicrobial properties of chitosan and silver nanoparticles on cotton fabric were tested. The emulsion imparted a very good and durable antibacterial property (against *Escherichia coli* and *Staphylococcus aureus*) on cotton at low concentrations (Hu *et al.*, 2005). Scholz *et al.* (2005) tested the antibacterial and anti-fungal properties of fabrics consisting of SiO_2 fibres coated with precious metal PVD layers using the magnetron sputtering technique. Layers of silver, copper, gold, platinum and platinum/rhodium were deposited on fabrics. Silver and copper were effective against bacteria *Stapholucoccus aureus* and *Klebsiella pneumoniae*. Other coating metals did not show any antibacterial effect. In the

fungal test, the samples were exposed to *Chaetomium globosum*, *Aspergillus niger* and *Penicillium funiculosum*. Copper was effective against all mildew: no mildew growth was observed on the surface of fabric after copper treatment. Other metals did not have any anti-fungal effect: after treatment, mildew growth covered from 25% to 100% of fabric surface.

Other compounds that inhibit the growth of microorganisms are peptides. Because of their high efficacy in fighting numerous pathogens, antimicrobial peptides have promising potential for use in agriculture, medicine and the food industry. This is of particular importance as the pesticides traditionally used for plant protection and the antibiotics widely used in healthcare have caused an increase in the resistance shown by pathogens. However, before antimicrobial peptides can be used, a number of regulations must be passed and, in the case of medical applications, clinical trials must be run to prove their effectiveness. In agriculture, positive results were obtained from tests on using one of these peptides, namely polyoxin, in commercially available fungicides. Polyoxin inhibits the synthesis of chitin, the basic component of cell walls in fungi. Polyoxin B effectively inhibited the growth of mildew in fruit trees and Polyoxin D zinc salt was used on peat soils as a fungicide against soil pathogens, for example *Rhizoctonia solani* (Keymanesh *et al.*, 2009). In the near future it would be interesting to test the viability of using antimicrobial peptides in the protection of natural fibre textiles against biodeterioration. Another one of these peptides, Cecropin B, was used to modify the *Bombyx mori* silk fibroin films (obtained from the giant silkmoth *Hyalophora cekropia*) by the carbodiimide chemistry method (Bai *et al.*, 2008). As a result of this modification, a smaller contact angle was achieved, along with greater surface roughness in the silk fibroin films. Those properties could prove important in preventing the contamination of the silkworm fibre biomaterial. A further natural peptides, lysozyme, is an enzyme obtained from chicken egg whites. Its action involves the decomposition of bacteria cell walls, especially of Gram-positive bacteria. Lysozyme can be combined with other anti-bacterial compounds in order to use such combinations in medicine, e.g. for wound dressings or creams. In the Air Force Research Laboratory at Florida's Tyndall Air Force Base a new material was developed by combining lysozyme and silver acetate in methanol; the mixture was then exposed to light and tested microbiologically. The tests indicated that the following microorganisms were susceptible to the action of the mixture: *Escherichia coli*, *Staphylococcus aureus*, *Bacillus anthracis* and *Candida albicans* (Halford, 2009).

Modified cellulose fibres containing covalent bounded biocidal preparations based on polyhexametylenoguanidine copolymer (PHMG) and its reactive derivatives are protected against *Staphylococcus aureus.* This implies the possibility of using them in protective wear in the healthcare field. PHMG is a polyhexametylenoguanidine and guanidine copolymer

with a relatively wide spectrum of biocidal activity. It belongs to the so called AMMS Group, or Antimicrobial Macromolecules, similar in structure and mode of action to natural proteins that fight infections, i.e. HDPS. Studies on the spectrum of activity of PHMG derivatives and their possible applications are also being conducted in Poland.

Biocides of plant origin such as extracts and essential oils have become the focus of research in various branches of economy. These biocides are used in plant protection, food processing and the cosmetics industry and in the production of packaging, medical, finishing and insulation materials containing natural fibres. They are also used to preserve museum exhibits and antique book collections. The use of essential oils from clove, cinnamon and oregano as antimicrobial solutions in paper packaging was studied. Cinnamon oil used in a paraffin coating totally inhibited growth of *Aspergillus flavus* (Rodriguez *et al*., 2007). The studies on the antimicrobial properties of mint oil in protecting food against infestation with *Aspergillus spp.* and *Penicillium spp*. fungi, and *Escherichia coli*, *Bacillus ssp.* and *Staphylococcus aureus* bacteria, have shown its anti-fungal and antibacterial effectiveness (Gulluce *et al*., 2007). Tests were carried out on the action of essential oil against the mildew most commonly present in libraries and archives, namely *Aspergillus niger*, *Chaetomium globosum*, *Penicillium frequentans* and *Paecilomyces variotii*. The vapours of different essential oils (armoise, boldo, clove, eucalyptus, lavender, tea tree, thuja, wormseed) were investigated. The different compounds were preliminarily screened by a micro-atmosphere method. Of the essential oils, eucalyptus oil (*Eucalyptus globolus*) showed moderate anti-fungal activity and wormseed oil (*Chenopodium ambrosioides*) was the most effective at inhibiting mildew growth (Rakotonirainy and Lavédrine, 2005).

Studies were also conducted on the anti-fungal efficiency of mint oil and mint extract in the protection of flax fibre used as a raw material in the production of nonwovens and fabrics. Mint oil and extract were applied by padding from a methanol solution at 20% concentration. 5 mL of the solution was used on 1 g of dry flax fibre. The tested samples of flax fibre were exposed to the action of the following fungi mixture: *Aspergillus niger* van Tieghem, *Chaetomium globosum* Kunze, *Gliocladium virens* Miller, *Paecilomyces variotii* Bainier, *Penicillium ochrochloron* Biourge. The flax fibre samples were allocated on an agar medium and inoculated with a suspension of the testing fungi. Samples were incubated at a temperature of 29 ± 1°C and relative air humidity of 90%, for 4 weeks. After the test, an evaluation of the anti-fungal properties was carried out on the basis of visual assessment of the degree of mildew growth and by testing the specific strength of the flax fibre. Mint extract did not show anti-fungal properties. Mint oil showed slightly lower anti-fungal activity: fungi growth was not visible with the naked eye, but single traces of fungi were visible microscopically, and a slightly higher

Table 16.4 Anti-fungal properties of flax fibre protected with biocides

Biocide	Degree of fungal growth (scale from 0 to 5)	Loss of specific strength (%)
Mint oil	1	22
Mint extract	4	83
Control unprotected	5	100

loss of specific strength was observed. The results are shown in Table 16.4 (Foksowicz-Flaczyk and Walentowska, 2008).

The anti-fungal efficiency of grapefruit extract at protecting natural textiles was tested. The biocide was applied to flax, needled nonwoven by the padding method, by applying 5 mL of solution per gram of non-woven. This was repeated six times, after which the protected samples were dried for 24 h in ambient temperature. The protected samples were exposed to *Chaetomium globosum* Kunze, at a temperature of 29 ± 1°C and relative air humidity of 90%, for 14 days. The protective effect of the grapefruit extract was evaluated visually based on the degree of mildew growth on the samples and also by testing the relative loss of breaking force. Maximum efficiency was observed for these biocides used on flax nonwovens: no fungi growth and no loss of breaking force occurred. Unprotected nonwoven material underwent complete decomposition, with mildew growth observed on all sample surfaces, along with 100% loss of breaking force (Kozłowski and Walentowska, 2006).

Thyme oil was found to display high anti-fungal activity when used to protect linen–cotton blended fabric. Thyme oil was applied by padding from methanol solutions of 20%, 12% and 8%; 1.2 mL of solution was used per gram of the dry fabric. The samples were exposed to the action of the following fungi mixture: *Aspergillus niger* van Tieghem, *Chaetomium globosum* Kunze, *Gliocladium virens* Miller, *Paecilomyces variotii* Bainier and *Penicillium ochrochloron* Biourge. The tested samples were allocated on an agar medium and inoculated with a suspension of testing fungi. The samples were incubated at a temperature of 29 ± 1°C and relative air humidity of 90% for 4 weeks. For all concentrations, no visible growth was observed under microscopic evaluation, and no loss of breaking force occurred. For comparison, control samples showed the highest degree of decomposition (5th degree) and 100% loss of breaking force. The results are shown in Table 16.5 (Kozłowski *et al*., 2008).

An assessment of the antibacterial activity of some commercial textile products was conducted. Socks, briefs, shoe insoles, foot spray, shoe spray, foot powder and hygienic pads were tested for resistance to *Staphylococus aureus* and *Escherichia coli*. For most products, some degree of bacterial

Table 16.5 Anti-fungal properties of linen–cotton blended fabric protected with thyme oil

Biocide	Degree of fungal growth (scale from 0 to 5)	Loss of breaking force (%)
Thyme oil 20%	0	0
Thyme oil 12%	0	0
Thyme oil 8%	0	0
Reference samples (with methanol)	5	100

inhibition was achieved, except for socks and briefs. Future work should be aimed at studying the use of herbal extracts and chitosan as ecological antimicrobial agents for finishing purposes (Thilagavathi *et al*., 2006).

16.4.2 Bio-availability of compounds used in the protection of natural fibre textile materials

The bio-availability of the compounds involved is key in ensuring the safe use of biocides (for protection against bacteria, fungi – including mildew – and insects) in fibre and textile applications. Bio-availability is related to the possible migration of these substances from textiles to the human body and their harmful effects on human physiology. Examples of substances with easy bio-availability are ZnO and Ag nanoparticles. Varsha *et al.* (2011) proposed an approach involving the attachment of chitosan molecules onto cotton cellulose fabric via coupling reaction. This induced covalent attachment following the incorporation of ZnO microparticles into the chitosan layer by an 'equilibration-cum-hydrothermal' approach. The fabric shows antibacterial efficiency against *Escherichia coli*, indicating its potential for use in biomedical applications.

16.5 Conclusion

The possibility of producing antimicrobial barriers (against bacteria, and fungi, including mildew) for natural fibre textiles through the use of biocides of natural origin or chemical biocides of low toxicity allows for the elimination of toxic chemical biocides. Conducting research into improvements in protection against biodeterioration is inextricably bound up with the market demand and growing interest in functional natural fibres.

Natural biocides have now been rediscovered and have become a safe alternative to traditional compounds, leading to the whole area of biodeterioration prevention becoming more eco-friendly and increasingly harmless for human health.

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17

Genetic engineering and biotechnology of natural textile fiber plants

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Abstract: Since ancient times, fiber crops have been closely connected with everyday life of human beings and have been utilized for many basic purposes including food, clothing and housing. Although over 2000 different species of fibrous plants are known, the list of commercially important fiber plants is not long, encompassing cotton, flax, jute, hemp, kenaf and ramie. Hard, leaf or structural fibers are obtained from agaves, Manila hemp and Mauritius hemp. From seed and fruit fibers, cotton, kapok and coconut have found commercial application. The same fiber may be used for different purposes. Genetic engineering and biotechnology give opportunities to improve the yield and quality of fiber crops precisely and more efficiently as compared with traditional breeding methods. From commercially available biotech crops, cotton is one of the most popular transgenic crops constituting 49% of total cotton production.

Key words: biotech crops, cotton, flax, jute, hemp.

17.1 Introduction: global status of commercialized biotech crops

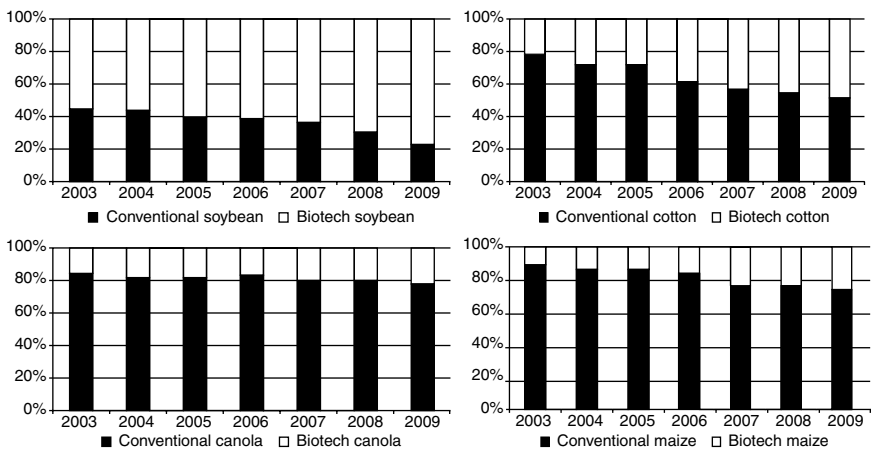
Biotech crops are cultivated for commercial purposes in many developing as well as industrial countries all over the world. Among a number of various genetically modified (GM) crop plants the only fibrous plant cultivated commercially remains cotton. Alike to other GM plants the most common in cultivation are those varieties of fibrous plants that are characterized by resistance to herbicide and diseases caused by pathogens or pests. In bast fibrous plant cultivation genetic modifications towards the control of lignin and pectin content are important.

17.1.1 Area and distribution of biotech crops

Bearing in mind considerable, long-term advantages in areas of environmental protection and economy associated with the cultivation of crops

cultivated using biotechnological methods, such plants are grown not only in commercial agriculture but also in millions of poor smallholdings all over the world. In 2009, these biotech crops were cultivated in 25 countries: in 15 developing and 10 industrial countries, on the total area of 134 million ha. Beginning in 1996, when first varieties became commercially available, till 2008, the total areas under biotech crops cultivation amounted to 800 million ha, i.e., 2 billion acres. The dynamics of area increase deserves attention; after 10 years the area amounted to one million and it doubled after just 3 years. It is expected that in 2015, the last year of the second decade since the introduction of biotech crops, they will be grown in 40 countries, primarily in developing countries. The year 2015 is also the year of Millennium Development Goals in which, according to the social declaration, it was planned to halve poverty and hunger indices in the world and the cultivation of biotech crops can contribute significantly to reaching this goals (James, 2008).

A large number of various genetically modified (GM) crop plants (especially industrial plants) have already been obtained but most frequently, they were investigated only in experiments carried out in glasshouse facilities (Thomson, 2003). Commercially available GM varieties include: soybean, maize, cotton, rapeseed as well as sugar cane, lucerne and papaya as well as several other crop plants (cultivated on the total area of less than 0.1 million ha). Soybean remains the most important biotech crop which in 2008 was cultivated on the area of 65.8 million ha and constituted 53% of the global cultivation area of all crop plants obtained with the assistance of biotechnological methods. At the present time, soybean is the only species whose transgenic cultivations exceed considerably traditional cultivations reaching 77% of all area. Figure 17.1 presents proportions of transgenic cultivations after the introduction of biotech crops.



17.1 Global adoption rates (%) for principal biotech crops (www.isaaa.org).

Table 17.1 Global area of biotech crops in 2008, by crop

Crop	Area in 2008 (millions of hectares)	% of global area	+/- compared with 2007	% compared with 2007
Soybean	65.8	53	7.3	+13
Maize	37.3	30	2.1	+6
Cotton	15.5	12	0.5	+3
Canola	5.9	5	0.4	+7
Sugar beet	0.3	< 1	0.3	–
Alfalfa	0.1	< 1	–	–
Papaya	< 0.1	< 1	< 0.1	–
Others	< 0.1	< 1	< 0.1	–
Total	125.0	100	+12.3	+9.4

Source: James (2008).

Until 2008, the only fibrous plant among biotech crops cultivated commercially was cotton, occupying 15.5 million ha, that is, 12% of the world cultivation area and, hence, the third plant in regard of the occupied area obtained with the assistance of biotechnological methods (James, 2008). At the present time, transgenic cotton comes close to exceeding 50% of cultivation area. The cultivation area of biotech crops in 2008 is shown in Table 17.1. Among the listed plants is cotton, the only representative of fibrous plants on the list of most frequently cultivated transgenic plants.

17.1.2 Major important traits of biotech crops

From among all GM plants, the most common in cultivations are those varieties that are characterized by resistance to herbicides or pests or varieties combining both these traits. From the moment the plants obtained by biotechnological methods were allowed to be cultivated until commercial cultivation in 1996 to 2008, the most frequently cultivated biotech crops were those with increased resistance to herbicides. It is now the second year in succession that varieties with stacked double or triple traits occupied a greater area than plants resistant to pests (see Table 17.2) (James, 2008).

Factors exerting a significant impact on yields are plant diseases caused by pathogens or losses due to pest feeding. Increased plant resistance in this respect translates not only into higher yields but, equally importantly, into increased organic matter bulk which can be ploughed under after harvest. The most recognized example of securing resistance to pests is the development of transgenic plants containing the *Bt* gene which codes a toxin naturally manufactured by *Bacillus thuringiensis* bacteria to which pests are susceptible. Toxin binding by specific receptors found in the epithelial cells of the gastrointestinal tract of insect caterpillars leads to the development of pores in their midguts and, consequently, to death (Thomson, 2003). Investigations

Table 17.2 Global area of biotech crops in 2008, by trait

Trait	Area in 2008 (millions of hectares)	% of global area	% compared with 2007
Herbicide tolerance	79	63	+9
Stacked double and triple traits	26.9	22	+23
Insect resistant	19.1	15	–6

Source: James (2008).

associated with possibilities of insecticide utilization already have a long history. *Bt* genes contain fragments rich in A/T residues, while plant genes usually contain more G/C residues. In order to solve this problem, *Bt* genes were synthesized in parts or in whole. In addition, shortened *Bt* genes are applied which code exclusively the toxic part of protein – delta endotoxin (Koziel and Carozzi, 1997). The only fibrous plant that contains the *Bt* gene is cotton which, thanks to genetic modification, is resistant to cotton bollworm.

Agricultural output is further reduced by losses associated with infections caused by viruses or fungi. Among registered GM crops characterized by resistance against viral diseases, there are no fibrous plants but there are crop plants such as: potato, plumb, vegetable marrow and papaya (CERA, 2010; Davis *et al*., 2004; Fuchs and Gonsalves, 1995; Gonsalves, 1998; Lawson *et al*., 1990; Rogan *et al*., 2000). Studies are currently in progress to develop manioc resistant to African cassava mosaic virus (Hong and Stanley, 1996).

17.1.3 Main desired traits of biotech fibrous plants

In bast fibrous plant production, the most important traits are genetic modifications towards the control of lignin and pectin content. It is considered to apply GM to increase the biomass of bast plants such as hemp and kenaf as well as towards the production of higher oil content in plants. Another possibility is the incorporation of phosphate groups (including PO_3, PO_4) into cellulose with the aim of obtaining modified cellulose with higher thermal resistance. An additional important feature is resistance of GM crops to biotic and environmental stress, including pests, fungi, salinity and drought (Kozłowski *et al*., 2009a, 2009b). For all bast fibrous plants it is still very important to obtain more homogeneous and fine fiber.

17.2 Fibrous biotech crops

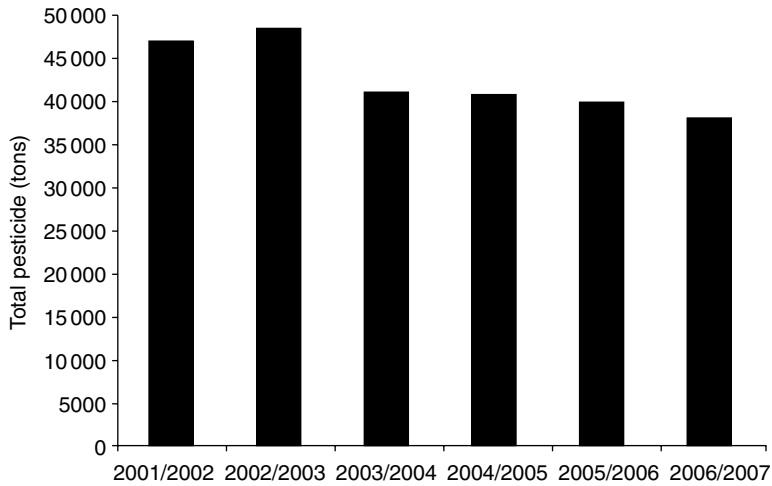
Among biotech crops cotton is the only fibrous plants grown commercially on a significant area. The most common varieties of the cultivated modified cotton included varieties resistant to herbicides. Cultivation of Bt cotton

in India revolutionized cotton production in this country. Development of biotechnology in China is fund by both public and private sectors. Chinese biotechnological companies developed Bt cotton varieties that compete on the global market with cultivars obtained thanks to international private investments. Despite a considerable interest in flax within the area of basic sciences, the only commercially available GM flax cultivar is CDC Triffid resistant to herbicides. The possibility of using biotechnological methods in breeding other fibrous plants remains in the area of scientific research.

17.2.1 Cotton

Cotton (*Gossypium hirsutum* L.) is an excellent source of natural fiber for the textile industry and cotton cultivations are of significant economical importance for the world market. Among crop plants obtained with the assistance of biotechnological methods, cotton is the only fibrous plants grown commercially on a significant area. In 2009, the area under cultivation of biotech cotton reached 16.2 million ha which constitutes 49% of the global total area of cotton cultivation. The area increase of the biotech cotton in India by 1.4 million ha, accompanied by a simultaneous decline of the area in the USA by 750 000 ha, resulted in the ultimate area increase by 3% placing biotech cotton as the third plant (after soybean and maize) on the list of all biotechnological cultivations. In 2009, biotech cotton was cultivated in 11 countries. Apart from India, increases of cultivation areas were also recorded in Australia, Mexico, Columbia and Republic of South Africa, whereas outside the USA, areas under cotton cultivation also declined in Brazil and Argentina. In China, on the other hand, there were no changes in the area of cotton cultivation. In 2008, cultivation of biotech cotton was introduced in Burkina Faso where, in the first year, it was grown on the area of 8500 ha and the area increased to 115 000 ha a year later (James, 2008). Costa Rica began biotech cotton cultivation in 2009. Globally, the total area under cotton cultivation declined by 6% which was associated with increased attractiveness, primarily, of financial inputs, of other cultivations, especially soybean and maize (Guitchounts, 2009; James, 2008). The most common varieties of the cultivated modified cotton included varieties resistant to herbicides. They occupied 76% of the biotech cotton cultivated area and 9% of the area under all biotech crops. Cultivation of *Bt* cotton resulted in the decline of insecticide consumption; in the USA, in 1998 the consumption of insecticides dropped by 900 000 kg in comparison with quantities used in previous years (Thomson, 2003). In 2001, American farmers growing *Bt* cotton used 15 million fewer doses of insecticides in comparison with farmers cultivating conventional varieties (Gianessi and Carpenter, 1999). *Bt* cotton had been cultivated in China since 1997 and reduced insecticide consumption by 80% (Xia *et al.*, 1999). What is even

more important, cultivation of *Bt* cotton in six provinces of this country led to a tenfold decrease in disasters caused by the feeding of cotton bollworm on other plants, such as maize, soybean, wheat, peanuts, melons, sesame and vegetables (Wu *et al*., 2008). So it was not only farmers who cultivated cotton who benefited but also growers of other crop plants. Considerable reductions in herbicide consumption were also recorded in India (see Fig. 17.2). Traditional cotton cultivations used up 42% of all insecticides applied in this country. Since the time cultivation of *Bt* cotton was introduced, such insecticides constitute only 18% of all pesticides market (see Table 17.3). During a period of 6 years (from 2001 to 2006) insecticide consumption dropped by 20%. In 2006, over 6 tons of the insecticide active ingredient were saved, worth approximately US$80 million (Choudhary and Gaur, 2010).

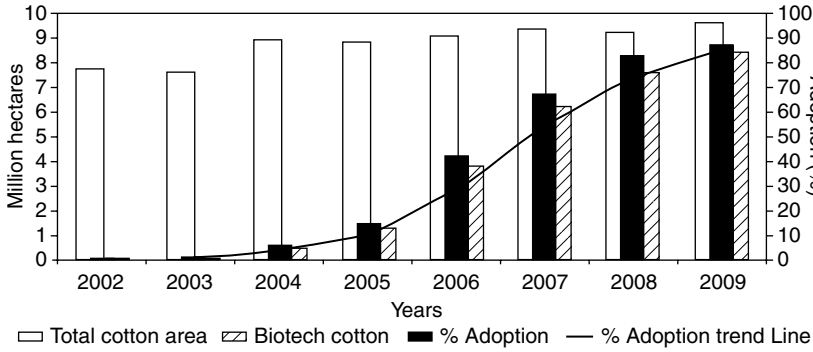


17.2 Pesticides usage in India, 2001–2006 (metric tons of technical grade or active ingredient) (Choudhary and Gaur, 2010).

Table 17.3 Value of the total pesticide market in India in 1998 and 2006 relative to the value of the cotton insecticide market

Item/year	1998	2006
Total pesticide market	770 million US$	900 million US$
Cotton insecticides as % of total pesticide market	30%	18%
Cotton insecticides as % of total insecticide market	42%	28%
Value in millions of US$ of cotton bollworm market and (savings due to *Bt* cotton) in 2006 over 1998	147 million US$	65 million US$ (savings of 82 million US$, or 56% compared with 1998)

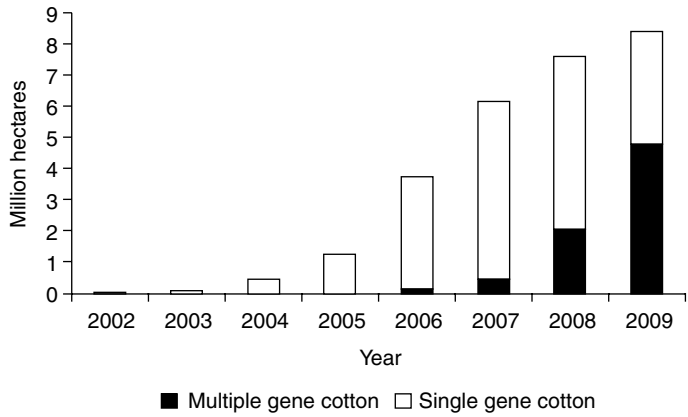
Source: Choudhary and Gaur (2010).



17.3 Adoption of *Bt* cotton in India for the 8-year period, 2002–2009 (Choudhary and Gaur, 2010).

Cultivation of *Bt* cotton in India revolutionized cotton production in this country. The huge importance of this biotech crop for the economy is presented in a comprehensive study entitled: '*Bt* Cotton in India: A Country Profile' (Choudhary and Gaur, 2010). During a period of 7 years (2002–2008), farmers' incomes from cotton cultivation reached US$5.1 billion and India turned from a cotton importer to the biggest world exporter of this crop. In previous years, cotton cultivation in India constituted 25% of the global cultivation but due to the lowest efficiency in the world, only 12% of its production derived from India. Replacement of the traditional cotton by *Bt* cotton resulted in a significant yield increase from 308 kg/ha (2001–2002) to 526 kg/ha (2008–2009). In the current vegetation season, yields are predicted to increase to 568 kg/ha, i.e., by 50% higher in comparison with those obtained in traditional cultivation. The observed dynamic area growth of the *Bt* cotton cultivations reached 6.2 million ha in 2007 and, consequently, became the largest cultivation area in the world. Year 2009 was a consecutive one in a period of 5 years in which the highest successive global increases of areas cultivated under biotech cotton varieties were recorded. In years 2006–2007 India overtook the USA and became the second – after China – largest cotton producer in the world. In the 2001–2002 season, India produced a total of 15.8 million bales of cotton, while in the record 2007–2008 season it produced 31.5 million bales. Such significant increase in production resulted mainly from the cultivation of *Bt* cotton (CAB, 2009). At the present time, cotton cultivation is highly profitable and cotton fiber constitutes 75% of fiber used in the textile industry in India (see Fig. 17.3). Cotton affects lives of 60 million people: farmers as well as multitudes of industrial workers beginning from processing of the raw material and ending with trade.

In recent years, there has been a distinct increase of interest among Indian farmers in hybrid lines (*Bt* cotton hybrids) containing several (most frequently two) additional genes. Apart from resistance to American bollworm, Pink bollworm and Spotted bollworm multiple *Bt* cotton hybrids are



17.4 Adoption of single and multiple gene *Bt* cotton hybrids from 2002 to 2009 (Choudhary and Gaur, 2010).

also resistant against *Spodoptera* larvae. Farmers growing these varieties get additional benefits, on the one hand, from fewer sprays against *Spodoptera* and, on the other, from increased yields by 8–10% in comparison with single *Bt* cotton hybrids (see Fig. 17.4).

The dynamic increase of interest in the cultivation of modified cotton varieties led to the development at the Central Institute of Cotton Research of a domestic cotton cultivar *Bikaneri Nerma* (BN) containing an active *cry1Ac* gene known as BNLA-601 and it was granted a cultivation permission in 2008 (Choudhary and Gaur, 2010; James, 2008; Santhy *et al.*, 2009). It is estimated that farmers' income from the cultivation of biotech cotton during a period of 12 years (1996–2007) amounted to US$13.6 billion, of which US$3.3 billion was in 2007 (Choudhary and Gaur, 2010).

In China, in 2008, *Bt* cotton was cultivated by 7.1 million farmers on the area of 3.8 million ha which constituted 68% of the area under cotton cultivation in this country which amounts to 5.7 million ha. The area of farms in China is very small; mean area of arable land in a farm is 0.8 ha and the mean area of a cotton field is 0.6 ha. The Centre for Chinese Agricultural Policy (CCAP, 2010) calculated that in farms in which *Bt* cotton was cultivated yields increased, on average, by 9.6% and the application of insecticides declined by 60%. This was important both for the environment as well as for the health of farmers and gave savings in the order of US$220 per ha. Bearing in mind the fact that the farmers' daily income is less than US$1, the achieved savings contributed to a significant improvement of living standards (James, 2008). It is also worth emphasizing that both public and private sectors are involved in financing the development of biotechnology in China. Chinese biotechnological companies developed *Bt* cotton varieties that compete on the global market with cultivars obtained thanks to international private investments. Opinions of politicians

about the strategic importance of agro biotechnology for securing increased productivity and food safety of the country as well as the competitiveness of the Chinese market on the world arena found a direct reflection in the development of biotechnology in this country. With quite high probability, it can be predicted that in future China will become one of the world leaders in this field. Even now, more than a dozen biotech crops are being tested in China in field conditions, including the following three most important ones – rice, maize and wheat – as well as cotton and others (James, 2008).

The importance of agro biotechnology in new cultivar breeding of fibrous plants has also been noticed in Egypt where investigations are under way aiming at developing cotton resistant to drought and salinity. Also in Egypt research projects are in progress with the aim to introduce *Bt* genes into Pima cotton (*Gossypium barbadense*) preconditioning resistance against cotton bollworm as well as other Lepidoptera.

Advanced investigations concerning possibilities of cotton modification for the food industry are also being conducted at the Commonwealth Scientific and Industrial Research Organization (CSIRO), Australia's national science agency. CSIRO has genetically modified cottonseed to produce high-oleic oils suitable for cooking purposes, but without cholesterol-raising *trans* fatty acids often found in processed cooking oils. The high-oleic cottonseed line has performed normally in field testing. A high-stearate form of cottonseed oil has also been produced, which will provide a nutritionally superior hardstock for margarine production.

Genetically modified cotton varieties can potentially be crossed with wild cotton populations or even with other species of cotton, e.g., upland cotton (*Gossypium hirsutum*) with Hawaiian cotton (*Gossypium tomentosum*) or Pima cotton (Daniell *et al*., 2005). It seems that one possible solution to this problem can be modification of the chloroplast DNA by the *Bt* gene (Daniell *et al*., 2005; Hagemann, 2004). A permanent transformation of the cotton plastid genome has been described and inheritance of the transgene in female line (Kumar *et al*., 2004). *In vitro*-produced transgenic cotton lines were grown in the growth chamber along with non-transgenic plants grown under similar conditions. Growth of chloroplast transgenic lines, onset of flowering, floral parts, boll formation and seed setting were similar to the untransformed cotton plants. Seedlings from F1 crosses (non-transgenic ♀x♂ transgenic) were able to germinate on kanamycin selection medium but failed to grow further, whereas transgenic seeds were resistant to kanamycin and germinated well, producing copious roots and leaves. This confirms earlier observations that there is no paternal or biparental inheritance of chloroplast genomes in cotton and that the chloroplast transgenic trait is inherited maternally (Daniell *et al*., 2005; Kumar *et al*., 2004).

Information concerning GM cotton cultivars resistant to insects and herbicides are collected in Table 17.4 (CERA, 2010).

Table 17.4 GM insect resistant cotton

No.	Event	Company	Description
1.	15985	Monsanto Company	Insect resistant cotton derived by transformation of the DP50B parent variety, which contained event 531 (expressing Cry1Ac protein), with purified plasmid DNA containing the *cry2Ab* gene from *B. thuringiensis* subsp. *kurstaki*.
2.	19–51A	DuPont Canada Agricultural Products	Introduction of a variant form of acetolactate synthase (ALS).
3.	281-24-236	DOW AgroSciences LLC	Insect-resistant cotton produced by inserting the cry1F gene from *Bacillus thuringiensis* var. *aizawai*. The PAT encoding gene from *Streptomyces viridochromogenes* was introduced as a selectable marker.
4.	3006-210-23	DOW AgroSciences LLC	Insect-resistant cotton produced by inserting the *cry1Ac* gene from *Bacillus thuringiensis* subsp. *kurstaki*. The PAT encoding gene from *Streptomyces viridochromogenes* was introduced as a selectable marker.
5.	31807/31808	Calgene Inc.	Insect-resistant and bromoxynil herbicide tolerant cotton produced by inserting the *cry1Ac* gene from *Bacillus thuringiensis* and a nitrilase encoding gene from *Klebsiella pneumoniae*.
6.	BXN	Calgene Inc.	Bromoxynil herbicide tolerant cotton produced by inserting a nitrilase encoding gene from *Klebsiella pneumoniae*.
7.	COT102	Syngenta Seeds, Inc.	Insect-resistant cotton produced by inserting the *vip3A(a)* gene from *Bacillus thuringiensis* AB88. The APH4 encoding gene from *E. coli* was introduced as a selectable marker.

(*Continued*)

Table 17.4 Continued

No.	Event	Company	Description
8.	COT67B	Syngenta Seeds, Inc.	Insect-resistant cotton produced by inserting a full-length *cry1Ab* gene from *Bacillus thuringiensis*. The APH4 encoding gene from *E. coli* was introduced as a selectable marker.
9.	DAS-21Ø23-5 x DAS-24236-5	DOW AgroSciences LLC	WideStrike™, a stacked insect-resistant cotton derived from conventional cross-breeding of parental lines 3006-210-23 (OECD identifier: DAS-21Ø23-5) and 281-24-236 (OECD identifier: DAS-24236-5).
10.	DAS-21Ø23-5 x DAS-24236-5 x MON-Ø1445-2	DOW AgroSciences LLC	WideStrike™/Roundup Ready® cotton, a stacked insect-resistant and glyphosate-tolerant cotton derived from conventional cross-breeding of WideStrike cotton (OECD identifier: DAS-21Ø23-5 x DAS-24236-5) with MON1445 (OECD identifier: MON-Ø1445-2).
11.	DAS-21Ø23-5 x DAS-24236-5 x MON88913	DOW AgroSciences LLC and Pioneer Hi-Bred International Inc.	Stacked insect-resistant and glyphosate-tolerant cotton derived from conventional cross-breeding of WideStrike cotton (OECD identifier: DAS-21Ø23-5 x DAS-24236-5) with MON88913, known as RoundupReady Flex (OECD identifier: MON-88913-8).
12.	Event-1	JK Agri Genetics Ltd (India)	Insect-resistant cotton produced by inserting the *cry1Ac* gene from *Bacillus thuringiensis* subsp. *kurstaki* HD-73 (B.t.k.).
13.	GHB614	Bayer CropScience USA LP	Glyphosate herbicide tolerant cotton produced by inserting a double-mutated form of the enzyme 5-enolpyruvyl shikimate-3-phosphate synthase (EPSPS) from *Zea mays*.

Table 17.4 Continued

No.	Event	Company	Description
14.	LLCotton25	Bayer CropScience (Aventis CropScience (AgrEvo))	Glufosinate ammonium herbicide tolerant cotton produced by inserting a modified phosphinothricin acetyltransferase (PAT) encoding gene from the soil bacterium *Streptomyces hygroscopicus*.
15.	LLCotton25 x MON15985	Bayer CropScience (Aventis CropScience (AgrEvo))	Stacked herbicide tolerant and insect resistant cotton combining tolerance to glufosinate ammonium herbicide from LLCotton25 (OECD identifier: ACS-GHØØ1-3) with resistance to insects from MON15985 (OECD identifier: MON-15985-7).
16.	MON-15985-7 x MON-Ø1445-2	Monsanto Company	Stacked insect resistant and herbicide tolerant cotton derived from conventional cross-breeding of the parental lines 15985 (OECD identifier: MON-15985-7) and MON1445 (OECD identifier: MON-Ø1445-2).
17.	MON-ØØ531-6 x MON-Ø1445-2	Monsanto Company	Stacked insect resistant and herbicide tolerant cotton derived from conventional cross-breeding of the parental lines MON531 (OECD identifier: MON-ØØ531-6) and MON1445 (OECD identifier: MON-Ø1445-2).
18.	MON1445/1698	Monsanto Company	Glyphosate herbicide tolerant cotton produced by inserting a naturally glyphosate tolerant form of the enzyme 5-enolpyruvyl shikimate-3-phosphate synthase (EPSPS) from *A. tumefaciens* strain CP4.

(*Continued*)

Table 17.4 Continued

No.	Event	Company	Description
19.	MON15985 x MON88913	Monsanto Company	Stacked insect resistant and glyphosate tolerant cotton produced by conventional cross-breeding of the parental lines MON88913 (OECD identifier: MON-88913-8) and 15985 (OECD identifier: MON-15985-7). Glyphosate tolerance is derived from MON88913 which contains two genes encoding the enzyme 5-enolypyruvyl shikimate-3-phosphate synthase (EPSPS) from the CP4 strain of *Agrobacterium tumefaciens*. Insect resistance is derived from MON15985 which was produced by transformation of the DP50B parent variety, which contained event 531 (expressing Cry1Ac protein), with purified plasmid DNA containing the *cry2Ab* gene from *B. thuringiensis* subsp. *kurstaki*.
20.	MON531/757/1076	Monsanto Company	Insect-resistant cotton produced by inserting the *cry1Ac* gene from *Bacillus thuringiensis* subsp. *kurstaki* HD-73 (B.t.k.).
21.	MON88913	Monsanto Company	Glyphosate herbicide tolerant cotton produced by inserting two genes encoding the enzyme 5-enolypyruvyl shikimate-3-phosphate synthase (EPSPS) from the CP4 strain of *Agrobacterium tumefaciens*.

Source: CERA (2010).

17.2.2 Flax

Flax (*Linum usitatissimum* L.) is a valuable fibrous and oily plant and its cultivation is of significant economic importance (Abbadi *et al*., 2004; Acikgoz and Kockar, 2007). The plant has been the subject of many investigations

from the area of plant biotechnology comprising biochemical problems, plant regeneration in *in vitro* cultures as well as interactions between plants and pathogens. A comprehensive review study concerning investigations from the field of tissue cultures and biotechnology was published by the team of Pretova (Millam *et al*., 2005). Flax was the subject of pioneer research from the area of plant tissue culture concerning indirect organogenesis, plant regeneration from adventitious shoots and pathogen development on leaves in *in vitro* cultures (Gamborg and Shyluk, 1976; Link and Eggera, 1946; Murray *et al*., 1977; Turel and Ledingham, 1957). As a species easily regenerating in *in vitro* cultures, flax was readily employed in investigations of embryo cultures and transformation of other plant species, among others, rapeseed and cereals (Millam *et al*., 2005). In successive years, the composition of the culture medium used in the process of organogenesis was optimized (Lane, 1979; Pretova, 1974; Rybczynski, 1975) and processes of direct and indirect somatic embryogenesis were performed (Cunha and Ferreira, 1996; Dedicova *et al*., 2000; Tejavathi *et al*., 2000). Investigations associated with obtaining haploid and diploid flax plants in *in vitro* cultures were of special importance (Bergman and Friedt, 1997; Murray *et al*., 1977). Flax regenerations from callus formed on fragments of hypocotyls, cotyledons and leaves (McHughen and Swartz, 1984), from zygotic embryos (Pretova and Williams, 1986), microspores (Nichterlein and Friedt, 1993) and anther cultures (Chen and Dribnenki, 2002) and from protoplasts (Ling and Binding, 1992) were described. In addition, enhanced regeneration resistance obtained from anther cells on *Fusarium oxysporum* causing fungal wilt was also reported (Rutkowska-Krause *et al*., 2003). It is believed that flax protoplast systems will become a very valuable material in investigations of new promoters and desirable genes (Evtimova *et al*., 2005). At the present time, cotyledon- and leaf-derived callus is utilized for plant regeneration, while hypocotyls are used in studies involving genetic transformations (Millam *et al*., 2005).

Despite considerable interest in flax within the area of basic sciences, the only commercially available GM flax cultivar is CDC Triffid resistant against sulfonylurea herbicides such as triasulfuron and metsulfuron-methyl. In 1996, permission was granted in Canada for the introduction of this variety into the environment and utilization as a feed for animals. Two years later, its cultivation for human consumption was allowed in Canada, while in the same year, the USA authorities allowed the variety to be cultivated for both feed and human consumption. Sulfonylurea herbicides target and bind to the acetolactate synthase, an enzyme involved in the biosynthesis of branched-chain amino acids: valine, leucine and isoleucine, resulting in the accumulation of toxic levels of alpha-ketoglutarate and subsequent plant death. The CDC Triffid was developed from the Norlin flax variety by insertion of the acetolactate synthase gene (*als*) from a chlorsulfuron tolerant line of *A. thaliana*. The variant *als* gene differs from the wild type *A. thaliana* gene by one nucleotide

and the resulting ALS enzyme differs by one amino acid from the wild type ALS enzyme. Successful transformants were subsequently grown on medium containing chlorsulfuron to confirm the expression of the inserted chlorsulfuron-tolerant *als* gene (FD/OFB-098-047-A, 1999; CERA, 2010).

At the present time, the economic significance of flax rests, mainly, on the introduction into cultivation of high-yielding varieties of oily flax. Flax oil provides a valuable supplementation of the diet with alfa-linolenic acid (18:3). It can also be used to produce biofuels and chemical fillers as well as in painting, lacquering and in production of linoleum (Acikgoz and Kockar, 2007). At present, 3 million tons of flax oil are produced in the world annually. Cultivations of the highest economical and industrial importance can be found in Argentina, India, China, Canada, USA and Russia (Millam *et al*., 2005). Modification of flax metabolism aiming at increasing the contents of carotenoids – nutrients important for humans – in flax seeds has been described. One of the following two genes was introduced into plants: gene coding phytoene synthase from soil bacteria *Pantoea ananatis* or fatty acid elongase 1 (FAE1) gene from *A. thaliana*. The obtained transgenic plants produced orange seeds containing phytoene, alpha-carotene and beta-carotene as well as increased quantities of lutein (Fujisawa *et al*., 2008; Fujisawa and Misawa, 2010). On the other hand, CSIRO is conducting investigations associated with possibilities of utilizing the Linola cultivar for food and industrial purposes (CSIRO, 2010).

Investigations have been in progress for several years now aiming at flax modification to synthesize in plants, *in statu nascendi*, biopolymers (see Chapter 10 in this volume, 'Bioengineered natural textile fibers'). Recently, researchers have described silencing of the gene coding cinnamyl alcohol dehydrogenase (CAD), an enzyme playing a key role in the process of lignin synthesis. The cloning of the CAD gene in an RNAi construction and its introduction into plants made it possible to obtain flax of lower lignin content and, hence, of better fiber quality (Wróbel-Kwiatkowska *et al*., 2007). It seems that further investigations will follow this direction as the possibility of obtaining plants with desirable traits is very attractive.

17.2.3 Jute

Jute provides high quality fiber which finds application in the production of a wide range of articles. From among 100 species of jute, only two, namely, *Corchorus capsularis* L. and *Corchorus olitorius* L. are cultivated widely (Sarker *et al*., 2007). These species are self-pollinating which explains their low genetic variability with regards to possibilities of adaptation to changing environmental conditions, fiber quality and yield as well as resistance to diseases and pests (Basu *et al*., 2004). *C. capsularis* is the species more resistant to drought and flooding but less resistant to pests and diseases.

The fiber of this species is white but not as strong as in other jute species. On the other hand, *C. olitorius* is relatively resistant to diseases and its fiber is strong (Roy *et al*., 2006). It would be very attractive to combine desirable characters of the two species in a single genotype and, understandably, attempts have been made for years to cross them but, so far, they have been unsuccessful (Chaudhuri and Mia, 1961; Datta *et al*., 1960; Finlow, 1917, 1921, 1923; Hoque *et al*., 1988; Islam, 1964; Islam and Rashid, 1961; Patel and Datta, 1960; Sajib *et al*., 2008).

Many regeneration protocols of jute in *in vitro* tissue cultures have been described but regeneration of *C. capsularis* is very difficult and that of *C. olitorius*, practically impossible (Hossain *et al*., 1994; Islam *et al*., 1982; Khatun *et al*., 1993; Rahman *et al*., 1985; Saha *et al*., 1999; Sarker *et al*., 2007; Seraj *et al*., 1992). Difficulties were also reported in the process of jute transformation (Ghosh *et al*., 2002; Sarker *et al*., 2007). It was not until 2008 that an article was published in which *C. olitorius* transformation independent of cell cultures was reported employing *Agrobacterium tumefaciens* (Sajib *et al*., 2008). A simple transformation protocol appears to be a turning point in jute research and allows its modification to obtain desirable characters.

17.2.4 Hemp

Hemp (*Cannabis sativa* L.), is valued not only for its fiber but also for its oil and finds application, among others, in food, textile and paper industries but also in medicine and pharmacology. Widespread hemp cultivation is limited by possibilities of utilization of some varieties to manufacture narcotics but, at present, a distinct interest is observed in the cultivation of new hemp varieties obtained with the assistance of biotechnological methods. Research performed recently has aimed to produce plants with resistance to pests, diseases and, most recently, with enhanced fiber elasticity. Transgenic cultivars of hemp can be used for synthesis of polyhydroxyalkanoates (PHA) and other biopolymers as an alternative to plastics for commercial production. Hemp does not have a long history of research and applications in plant tissue culture so there is only a small number of reports concerning tissue cultures of hemp. Most studies concerning hemp tissue cultures were aimed at developing a cell culture system to obtain secondary metabolites. Only a few reports have described tissue culture conditions intended for plant regeneration (Feeney and Punja, 2003; Slusarkiewicz-Jarzina *et al*., 2005; Wielgus *et al*., 2008).

17.2.5 Other fiber crops

Fibers may be extracted from various parts of different plants and used for commercial purposes and home industry. Major crops of technical or

economic importance include: bast fibers represented by flax, hemp, kenaf, jute, nettle and ramie; leaf fibers like sisal, abaca, banana and henequen; grass fibers including esparto, bamboo and reed, seed hairs cotton, kapok and milk weed and palm fibers represented by coconut husk coir. Long, hard fiber plants like sisal, abaca or manila hemp are traditionally used for cordage, such as for binder and baler twine and for ropes. Soft or bast fibers like flax, hemp, jute and kenaf are used for textiles. Miscellaneous fibers like 'broom' root, Spanish moss and coir from coconut husks can be used for upholstery, brushes or cordage and floor coverings. Short, one-celled fibers like cotton and kapok are used as textile fiber.

Fiber crops are widely used in the paper industry and, after chemical modification, are used for the production of viscose or cellophane. Delivery and storage of the fiber source can be a major issue in production of pulp. Recently natural fibers are used as valuable components of composite materials.

Ramie (*Boehmeria nivea*) is also known as Chinese grass. White ramie, green ramie or rhea is one of the oldest textile fibers known in ancient Egypt and China. Ramie fiber is used in fine linen and other clothing fabrics, upholstery, canvas, filter cloths, sewing threads, gas mantles, fishing nets and marine packings. Short fibers and wastes are used for the production of high quality papers, such as banknotes and cigarette papers. Ramie is used for preparation of plant-derived ecological bioplastics made from the cellulose in wood or grass instead of petroleum. Ramie can also be considered as a good candidate for phytoremediation of sites contaminated by heavy metals. Kenaf (*Hibiscus cannabinus*) belongs to the most promising non-wood fiber alternatives for pulp and paper production.

Fiber plants can be used as energy plants for the development of bioenergy. Miscanthus and switchgrass (*Panicum virgatum L.*) are being developed as biomass crops largely for fuel and energy. Cellulose can be converted to ethanol as fuel for vehicles at much lower production costs compared to annual fiber crops or other biomass crops. Fiber plants could also be used for biopolymer synthesis. Switchgrass has been developed for energy in North America, Canada and, more recently, in Europe (Zeng-Hui and Hong-Bo, 2010).

The conversion of plant biomass into fuels is a complex process depending upon plant species and cell type. This knowledge of the molecular basis of these natural variations in wall composition will be a valuable resource that can be used for generation of designer biofuel crops using either selected breeding methods or recombinant DNA techniques.

Renewable biomaterials can be obtained by direct use of biomass or biomass components like fibers, natural rubber, starch, cellulose, sugars or oils. Industrial biotechnology involving fermentation (white biotechnology) or conversion using chemical methods delivers new compounds.

Biopolymers could also be obtained by production in transgenic plants (green biotechnology).

17.3 Future trends

Most fiber crop genotypes are specialized for the production of either fiber or oil, but not for both products. For example, the terms fiber flax and linseed are often used to distinguish between plants cultivated specifically for fiber or oil. The quantity and quality (especially length) of linseed fibers is generally insufficient for production of high quality linen textiles. In fact, the presence of fibers in stems is a major impediment to linseed cultivation, as the fibers may bind to harvesting and processing equipment and are slow to decompose in soil. A preferable solution involves a practical use for the straw, such as utilization in industrial products that require strong fibers of moderate length, including composites, nonwoven materials, and specialty papers. This would create a truly dual-purpose crop with increased appeal to growers and others in the value chain. A comparison of harvest data for selected crops shows that approximately six times more land is planted in case of linseed than in fiber flax.

A major limitation to the expanded production of high value fibrous plants like flax, hemp, jute and kenaf for textiles is the requirement for retting in which flax straw is exposed to microorganisms to help separate the fibers from the surrounding tissues. The release of the fibers occurs through the enzymatic degradation of pectins and phenolics and this opens new research directions and should be enhanced by biotechnological methods, reducing costs and time of processing. Breeding or genetic engineering of flax plants towards weaker connections between fiber bundles and adjacent cells would therefore allow more efficient production of high-quality fibers. Complex understanding of the connections between fibers and other cells would allow improvements in the use of microbes and enzymes in retting of flax stems.

Composite materials contain a reinforcing material such as fiberglass, embedded in a polymer matrix. During the past decades, composites have found such wide application in worldwide production that their disposal is now becoming a concern. For biotechnologists new applications tasks are open in the development of composites based on renewable materials. The use of plant fibers as an alternative to synthetic fibers in composites has many important benefits including their abundance (often as waste materials or crop residue), biodegradability, CO_2 neutrality (i.e., no net CO_2 release to atmosphere during product life cycle), low abrasion (which reduces mechanical, dermal, and respiratory irritation), and low density (which makes the fibers lighter than many alternatives).

Natural fibers obtained from hemp, jute or flax have a very high tensile strength, often greater than that of other types of fiber (e.g., cotton), and

comparable to glass or steel on a strength-per-weight basis. As disadvantages of the use of natural fibers, when compared with synthetic alternatives, one should mention non-uniform dimensions or other hydrophilic properties and withstanding high temperatures. Nevertheless, fibrous plants have been found to be suitable replacements for synthetic reinforcing material, when they are exposed to treatments such as sodium hydroxide, acetic anhydride, formic acid, helium cold plasma, silanization and autoclaving.

The potential impact of natural fibers in production of composites and expanding industrial application makes this field an active area of research for biotechnologists. There is broad expectation that composite reinforcement, rather than linen production, is the most immediate way to developing practical, dual-purpose fibrous plants. Another potential end use is in papermaking and supplementation of hardwood kraft pulps, as a cost-effective method of increasing tear strength, bulk and opacity.

Paper pulp manufacturing is the first non-food industrial utilization of plant biomass (Gutierrez *et al.*, 2001). In theory, all vascular plants in nature can be used as sources of cellulosic fibers for paper and pulp manufacture; however, both availability and production costs limit the natural source of fiber. There is an increasing interest in using non-wood fibers, mainly for specialty paper production. Among the non-wood plants, straw has been used in Asia, Africa, Eastern Europe and Latin America for the production of paper pulp as well as in Spain and other European countries for the manufacturing of high-quality pulps for specialty papers. Environmental studies focus on flax, hemp, jute and kenaf straw as potential sources of fiber from which high quality paper products are produced. Both crops have been two of the most important fiber crops in Europe, although in some areas hemp is more widespread than flax. Hemp and flax cultivation have excellent agronomic characteristics, they can also be excellent predecessors in crop rotation, and in addition can provide high fiber yields. At the present time, there is increasing interest in using non-woody fibers as raw materials for pulp mills and production of hemp and flax will very likely expand (González-García *et al.*, 2010).

Characterization of genes involved in production of fibers and understanding their function develops a great potential for the creation of a designer fiber crop. Ideally, fiber crops should be suitable for simultaneous production of high-value oils and fibers. This opens new possibilities for genetic engineering aimed at fiber quality improvement including increased strength and hydrophobicity of fibers for use in composites, increased softness for textiles, and increased yield and processing efficiency for all applications. Already, the production of a bioplastic within the lumen of transgenic flax fibers has been achieved, with a resulting increase in tensile strength. In other studies, transgenic manipulation of pectin-related genes in flax cell cultures indicates potential for development of plants with improved retting abilities.

For improvement of flax fiber properties, transgenic flax overexpressing the biodegradable bacterial polymer polyhydroxybutyrate (PHB) synthesis genes was prepared (Szopa *et al.*, 2009). Transgenic plants have more highly structured cellulose in fibers with PHB strongly bound by covalent ester and hydrogen bonds. The composite containing fibers from transgenic plants was significantly stronger and stiffer than the composites containing fibers from the control plants. Scanning electron microscopy of the fracture surface of composite sheets showed that fibers from transgenic plants adhered to the polypropylene matrix significantly better. The composite containing fibers from transgenic plants induced almost no platelet aggregation and may be used for biomedicine purposes in contact with blood. Fibers from transgenic plants showed lower amounts of residual substances (pectins, parenchyma, fats and waxes) on their surface and the retting was more effective for transgenic plants. Unfortunately the negative standpoint about transgenic flax of bast fibers producers in Europe, for example the European Flax and Hemp Confederation (Confédération Européenne Du Lin et Du Chanvre), has weakened interest in genetic modification of flax compared with Asia and the USA.

The future adoption of biotech crops in developing countries in the period 2010–2020 will depend mainly on three major issues: (1) establishment of appropriate regulatory systems; (2) political support for the adoption of biotech crops contributing to a more affordable and secure supply of food, feed and fiber; (3) continuation and expansion of supplies of appropriate biotech crops with expected priority needs for developing countries.

Although the most widely commercialized genetically modified crops are herbicide-tolerant soybeans and insect-resistant maize and cotton, there is also a strong need to develop crops resistant to biotic and abiotic stresses. Such crops are critical for sustainable food production in the developing world by the year 2025. Both transgenic and other technologies have the potential to contribute to such development. In case of fibrous plants, the gap in knowledge should be overcome which includes efficacy of biotech crop compared to conventionally bred varieties with similar traits, their risks of invasiveness and impact on other organisms, and the possibility of creation of new plant viral diseases. Some of these gaps may soon be filled by new technologies. It is very likely that research into the biology and biotechnological application of fibrous plants will continue and expand in the next decades.

17.4 Conclusions

1. By 2015, the final year of the second decade of introduction of biotech crops, cultivations will include over 40 countries, mainly developing countries. Year 2015 is also the year of Millennium Development Goals

in which – in accordance with the social declaration – poverty and hunger index in the world was planned to be reduced by half thanks to cultivation of biotech crops.
2. Soybean remains the most important biotech crop. In 2008, it was cultivated on the area of 65.8 million ha which constitutes over 53% of the global cultivation area of all plants obtained using biotechnological methods. Until 2008, the only fibrous plant among biotech crops cultivated commercially was cotton.
3. Among all GM plants, the most common in cultivation are varieties characterized by resistance to herbicides and resistance to pests or varieties combining both these traits.
4. In bast fibrous plant production, the most important are genetic modifications towards the control of lignin and pectin content. It is planned to apply GM to increase the biomass of bast plants such as hemp and kenaf as well as towards the production of higher oil content in plants.
5. Countries other than the USA are now producing and cultivating their own GM crops designed for certain climate conditions and new regulations are required which may bear a close similarity between USA and Europe, and guide GM production in developing countries.

17.5 Sources of further information and advice

- ec.europa.eu/food/food/biotechnology/index_en.htm
- www.agbios.com/main.php
- www.agbioworld.org
- www.isaaa.org/
- www.oecd.org/department/0,3355,en_2649_34385_1_1_1_1_1,00.html

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17.7 Appendix: abbreviations

ALS	acetolactate synthase
als	acetolactate synthase gene
BNLA-601	cotton cultivar *Bikaneri Nerma* (BN) containing an active *cry1Ac* gene
Bt	toxin naturally manufactured by *Bacillus thuringiensis* gene
CAB	Cotton Advisory Board
CAD	cinnamyl alcohol dehydrogenase
CDC	Triffid genetically modified flax cultivar resistant against sulfonylurea herbicides
CSIRO	Commonwealth Scientific and Industrial Research Organization
FAE1	fatty acid elongase 1 gene
F1	the first filial generation seeds/plants or animal offspring
GM	genetic modification
PHA	polyhydroxyalkanoates
PHB	polyhydroxybutyrate
PO_3, PO_4	phosphate groups
RNAi	RNA interference system within living cells that helps to control activity of genes

18

Wild silk: wild silk enterprise programs to alleviate poverty and protect habitats

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Abstract: We propose that earnings from sustainable, small-scale, wild silk enterprises can replace earnings from non-sustainable harvesting of forest resources. In the first section of the chapter we review the macrostructure of wild silks and those factors most likely to affect wild silk economic value. In the second part of the chapter we review four wild silk enterprise projects whose goals are poverty alleviation and biodiversity protection. We compare project effectiveness in terms of numbers of people employed, returns to farmers and potential habitat restoration. Wild silk production, and similar small-scale enterprises, may be a more effective conservation and poverty alleviation tool than payments for ecosystem services (PES) or Reducing Emissions from Deforestation and Forest Degradation (REDD) in developing countries where governments are unstable and people are chronically poor.

Key words: wild silk, payment for ecosystem services, poverty alleviation, enterprise conservation.

18.1 Introduction

Although many countries harbor diverse species of wild silk moths, with the exception of India, wild silk has been largely ignored for commercial use. The term 'wild silk' is used here to describe all silk not obtained from the domesticated silkworm, *Bombyx mori*. We propose that wild silk enterprise can be developed as a profitable, ecologically sustainable livelihood to replace the need to earn income through non-sustainable forest extractions.

Silk threads spun by the wild silkworm *Antheraea assamensis* (Saturniidae; native to deciduous, highland forests of northeast India) and *Antheraea mylitta* (Saturniidae; native of tropical, humid, southern India) were used in jewelry 2000 years prior to the documented use of silk produced by the domesticated silkworm, *Bombyx mori*, in China (2250–330 BC; Good, 1995; Good *et al*., 2009). Today, production of tasar silk and silk textiles is India's

most important cottage industry and is particularly important in rural communities. Multiple strains or 'ecoraces' of *Antheraea mylitta* are collected from humid, tropical forests that extend east to southwest across the central Indian plateau (Jolly *et al*., 1974; Reddy, 2010) (Table 18.1). More recently developed is the hybrid tasar, *A. proylei*, a silk moth whose host plants are indigenous species of oak trees. *A. proylei* was introduced across the foothills of the Himalayas for small-scale industry and as economic tool giving value to endemic oaks. Silk textiles produced from *A. proylei* and *A. mylitta* cocoons are commercially important within India, but have not been exported in high volumes. In the past 10 years, the demonstrated success of wild silk production to generate income for rural and isolated communities in India (small investment, return within a relatively short period, full and part-time employment for many) and a recognition of the effects of habitat loss on silk moth species diversity, has revitalized interest in identifying the species remaining and how they can be conserved (Kakati and Chutia, 2009; Reddy, 2010; Saratchandra and Singh, 2002).

In countries throughout the tropics and sub-tropics, valuable wild species of silkworms are abundant, and native populations of the moths are underutilized and understudied (Iizuka, 2002). Unfortunately, aid programs designed to alleviate poverty in the developing world frequently side-step the important but time-consuming research needed to develop production of native silks and instead simply import foreign species of silkworms, both 'wild' and domesticated, for income generating programs (e.g. Agrifood Consulting International, 2007; Patil *et al*., 2009). Wild silk production that makes use of endemic silk moths can alleviate poverty while conserving native plant and animal species. Use of imported silk producers devalues local flora and fauna in the eyes of farmers and results in a lost opportunity to conserve native species and habitats for economic returns.

In this chapter we describe the macrostructure of 'wild' silks that have been, or could be, produced commercially. We then analyze and compare four programs that have developed and implemented wild silk production to reduce poverty and conserve habitats. We argue that sustainable silk enterprise that makes use of native species of silk moths and host plants and that builds farmer independence and commercial networks may be a more effective conservation tool in extremely poor countries than Payments for Ecosystem Services (PES) and payments through Reducing Emissions from Deforestation and Forest Degradation (REDD) programs.

18.2 Definition of silk

Silk proteins have evolved repeatedly among arthropods (insects and spiders) and are used for multiple proposes such as protective shelters, protection for eggs, food capture nets and flight (Craig *et al*., 1999; Sutherland

Table 18.1 Silk moths raised/collected for textile production

Species (common name)	Cocoon weight (g), color	Cocoon value (US$)	Fiber characteristics	Number of generations/ year	Habitat/ elevation	Countries where projects are located	Reference
Antheraea assamensis (Muga)	0.42 g Golden-brown, golden-yellow, glossy white; soft Filament length 500–800 m	0.02	Porous, reeling difficult	India – 5–6 generations/ year 20–53 days depending on summer or winter crop	500–1500 m Upper Assam (NE India); best conditions 23–27°C; temperature has profound effect	India	Sahu *et al.*, 1998; Sonwalker, 1993; Choudhury *et al.*, 1998; Sahu *et al.*, 1998; Akai, 2000b; Kakati and Chutia, 2009 http://southasia.oneworld.net/fromthegrassroots/a-fillip-for-the-silk-industry-in-northeastern-india
Antheraea mylitta Drury (tropical tasar)	0.81–1.96 g Filament length 800–1400 m Yellow, grey	0.04	Porous, reeling difficult	1–3 per year depending on eco-race, habitat and host plant	Widely distributed, semi-deciduous forest; from southern territory Western Ghats to Assam in the east and Himachal Pradesh in the west	India	Jolly *et al.*, 1974; Thangavelu *et al.*, 2002; Sonwalker, 1993

Antheraea mylitta: ecoraces			Porous, reeling difficult	2		India	Sharma and Sharma, 2006; Thangavelu *et al.*, 2002; Sonwalker, 1993
Jata	1.6–2.34 g Fibre length 800–1400 m	0.05	Porous	1	152 m	India	Reddy *et al.*, 2009
Modal	2.25–3.5 g Fibre length 800–1400 m	0.6	Porous	2	610 m	India	Reddy *et al.*, 2009
Sukinda	0.99–2.0 g Fibre length 800–1400 m	0.01	Porous	3	107 m	India	Reddy *et al.*, 2009
Daba	1.25–2.36 g Fibre length 800–1400 m	0.03	Porous	2,3	152 m	India	Reddy *et al.*, 2009
Antheraea roylei	0.55–0.80 g Double layer, white		Porous, reeling difficult	2	199–3841 m humid, sub-tropic, warm temperate	India	Kakati and Chutia, 2009; Sonwalker, 1993
Antheraea proylei (temperate tasar)	0.9 g; soft Filament length 600–700 m		Porous reeling difficult				Sonwalker, 1993
Anthereae pernyi		Slightly lower price than *B. mori*	Porous, reeling difficult		Temperate	North China	Akai, 2000b

(*Continued*)

Table 18.1 Continued

Species (common name)	Cocoon weight (g), color	Cocoon value (US$)	Fiber characteristics	Number of generations/ year	Habitat/ elevation	Countries where projects are located	Reference
Antherina suraka	Bronze silk 0.20 g	0.02*	Porous, spinning difficult	2–3	Mixed elevation; rainforest 145–1371 m	Madagascar	CPALI – *wholesale price; unpublished data
Samia cynthia (Eri)	Brown, brick red, white; size variable with region 0.4–0.6 g		Porous, spun	1–3	Tropical, humid 10–24°C 74–77% RH; 1300–1700 m; primary host plant castor (fast growing in domestic culture	North-eastern India	Ramalakshmi, 2000; Vijayan *et al.*, 2006
Cricula trifenestrata	0.102–0.228 g	0.02	Numerous fine tubules per filament	2	145–1371 m humid, agricultural sites	Indonesia	Kakati and Chutia, 2009; Situmorang, 2002; Nurmalitasari and Kuroda, 2002 ; Akai *et al.*, 1996
Attacus atlas	Whitish, double layer		Porous			Indonesia	Situmorang, 2002; Nurmalitasari and Kuroda, 2002; Akai *et al.*, 1996
Gonometa postica Walter	1.8–2.0 g		Compact	2	786 m dry, acacia woodland	Kenya	Akai, 2000b; Kioko *et al.*, 2000

Gonometa rufobrunnea Aurivillius	White, yellow 1.49 g		Compact	2		South Africa, Namibia, Botswana	Veldtman *et al.*, 2002
Bombyx mori (domesticated silkworm)	1.3–1.6 g		Compact filaments, extremely uniform, fiber decreases in diameter from the outer to inner cocoon		Selected for temperate, tropical, montane, lowland	Worldwide	
Borocera madagascariensis	Brown, covered with spines	0.3	Compact fibers	2	Montane, tapia woodland	Madagascar	Razafimantsoa, pers. com.
Anaphe	Brown, larvae aggregate to build common cocoon with 2–300 individuals	?	Compact fibers 18 μm inner filaments (0.0007 in); outer, 0.0004–0.0007 in	1	Montane	Ghana, Nigeria, Uganda, Kenya, Madagascar, Mozambique	McKinney and Eicher, 2009; Gowdey, 1953; Razafimanantosoa *et al.*, 2006

et al., 2009). Silk fibers are made up of repetitive sequences of amino acids, stored in liquid form and drawn into thread when sheared or 'spun' at secretion.

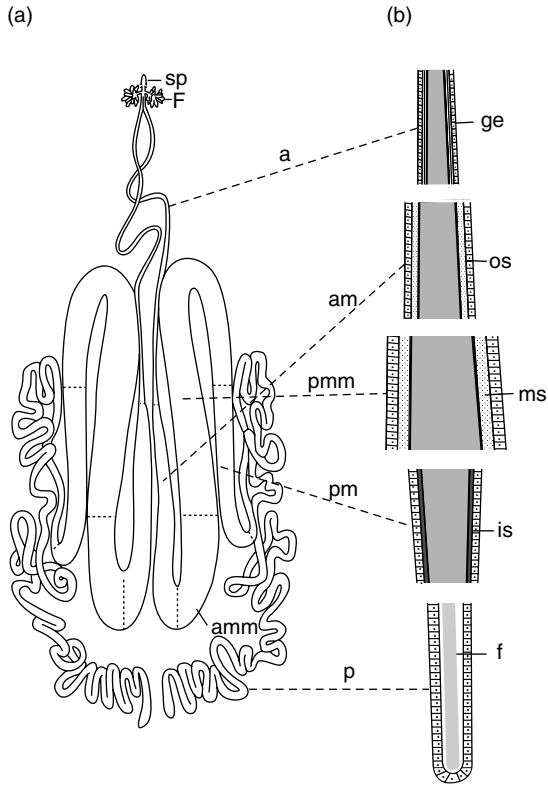
The most prolific silk producers are spiders (order Araneae) and insects in the order Lepidoptera. Spiders produce relatively small amounts of multiple kinds of silk every day throughout their lives. Although some Lepidoptera may produce small amounts of silk daily to mark foraging trails (e.g., larvae in the Lasiocampidae), production is not in volumes large enough for commercial exploitation. Silk spun by Lepidoptera that is used commercially for textiles is derived from cocoons, or the fiber case, that larvae spin to protect themselves from microbial degradation and predators during metmorphosis (Zhao *et al*., 2005). Silk produced by moth larvae in the families Saturniidae, Lasiocompidae and Thaumetopoeidae have been used for commercial textiles (Peilger, 1993).

The properties of silk that affect their economic value are surface reflection, fiber microstructure and biomechanical properties. While we discuss only a limited selection of wild silks, they represent commercially viable silks spun by Lepidoptera that can be found in a variety of habitats.

18.3 Silk structure and function

All silk-producing Lepidoptera have a 3-part silk gland where the different protein components that make up silk fibers are produced (Fig. 18.1). In general, silk is a composite protein made up of β-pleated sheets packed into crystalline arrays with the accompanying proteins forms, α-helices, β-spirals and spacer regions of anomalous amino acid sequences. These protein components, together with physiological processing in the gut, spinning speed and the sculpting of the fiber's surfaces when the protein passes through the larval spinnerets, result in different biomechanical and surface properties as well as different hydrophilicities (affinity for water).

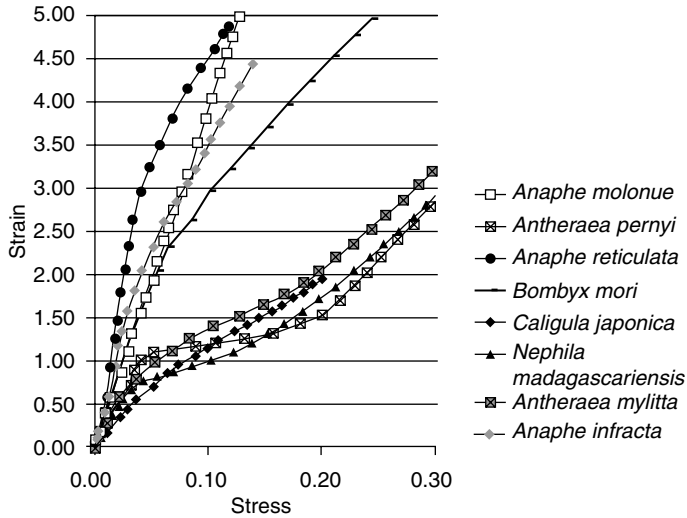
While there is a great deal of detailed information on the mechanical properties of silk spun by *B. mori*, there is very little information on the strength, elasticity, energy absorption and hydrophilicity of wild silk fibers. Molecular data, however, show that silk produced by the Bombycidae (e.g., *B. mori*) and Saturniidae (e.g., *Antheraea*), the most commercially important families silk producers, differ in molecular organization and composition (Sezutsu and Yukuhiro, 2000; Zhou *et al*., 2000). Silk spun by Saturniidae insects is similar to the ampullate gland produced by spiders and is more elastic (and probably more hydrophilic) than silk produced by the Bombycidae as well as silkworms in the Thaumetopoeidae (bagworms) (Komatsu, 1994) (Fig. 18.2). Furthermore, larval Bombycidae, Thaumetopoeidae and Lasiocampidae spin silk that is compact in macrostructure while the silk fibrils produced by Saturniidae are porous (Fig. 18.3) (Akai, 2000; Narumi *et al*., 1994).



18.1 (a)The silk gland of *Bombyx mori*. (b) The secretory materials found in various regions of the gland: a, anterior silk gland; am, anterior division of the middle silk gland; amm, anterior part of the middle division of the middle silk gland; F, Filippi's gland; f, fibroin; ge, gland epithelium; is, inner layer sericin; ms, middle layer sericin; os, outer layer sericin; p, posterior silk gland; pm, posterior division of the middle silk gland; pmm, posterior part of the middle division of the middle silk gland; sp, spinneret. (Reprinted from Akai, 1976.)

18.3.1 Bombycidae

Bombyx mori, domesticated from the wild species *Bombyx mandarina*, includes multiple ecoraces of silk moths that produce cocoons that differ in size and color. The natural pigments that color the cocoons yellow, pink, gold, yellow green and green are either soluble, yellowish carotenoids (Tabunoki *et al*., 2004) or green flavonoids (Kurioka and Yamazaki, 2002). Pigmentation is confined to the sericin that coats the thread and hence can be removed. The length of cocoon filament varies from 300 to 1700 m depending on the moth's ecorace (Akai, 2000). The surface of the thread produced by *Bombyx mori* is extremely uniform (a function of spinneret



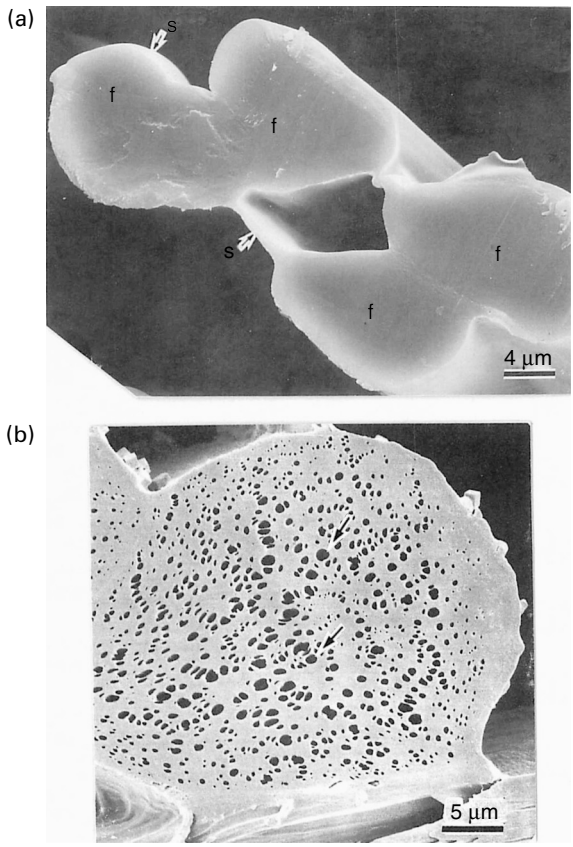
18.2 Comparison of mechanical properties of silk produced by the domesticated silkworm *Bombyx mori*, the spider *Nephila madagascariensis*, and 'wild' silkworm species *Anaphe molonue*, *Anaphe reticulata*, *Anaphe infracta* (Thaeumetepoideae) and *Antheraea pernyi*, *Antheraea mylitta* and *Caligula japonica* (Saturniidae). Silk produced by *Anaphe* spp., and *B. mori* have a higher breaking point but are stiffer than silk produced by the Saturniidae moths and the spider, *N. madagascariensis.* (Data from Komatsu, 1994.)

morphology), a property that is important for its commercial value (Akai, 2000).

The tensile strength of silk thread sampled from *Bombyx mori* cocoons is about 0.5 gigapascals (GPa), with breaking elongation of 15%, and a breaking energy (toughness) 6×10^4 J kg^{-1} (Shao and Vollrath, 2002). *B. mori* silk is more elastic than silk spun by species of *Anaphe* (Thaumetopoidae, 'bagworms') but with a similar breaking point. In comparison to silk produced by Saturniidae, *B. mori* silk is less elastic but with a higher breaking point (Fig. 18.2). Sericin coats the outer filaments of the cocoon and the filament decreases in diameter from the outer layers to the inner layers of the cocoon (Akai, 2000). Cocoon size is regulated by complex hormonal interactions during larval development (Akai *et al.*, 1971; Sehnal and Akai, 1990) (Fig. 18.4).

18.3.2 Saturniidae

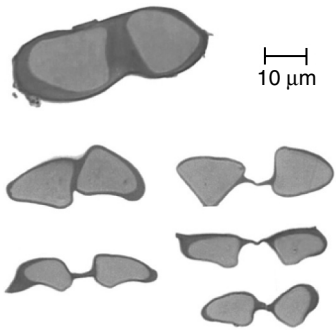
In India, 95% of 'wild silk', or vanya silk, is spun by two species of Saturniidae, *Antheraea mylitta* (tasar) and *Antheraea assamensis* (muga) (Ojha and Panday, 2004). Unlike the compact, macrostructure of silk threads produced



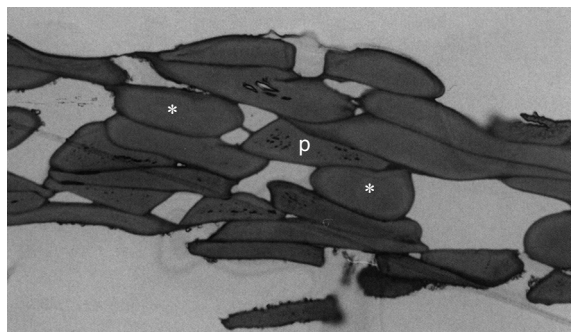
18.3 Comparative fine structure of silk produced by (a) *B. mori* and (b) *Antheraea mylitta*. f: fibroin, s: sericin. Arrows indicate fine tubular structures in fibroin interior in *A. mylitta*. Silk produced by *B. mori* as well as silkworms in the families Lasiocampidae and Thaumetopoeidae are compact. All silkworms in the family Saturniidae produce silk fibers that are porous (i.e. *Antherineae, Cricula*) or that are a mix of porous and non-porous fibrils (*Rothschildia*). (Reprinted from Akai, 2000b.)

by *B. mori*, all Saturniidae species produce fibers that are permeated with fine tubules. A single filament of silk produced by *A. yamamai* contains nearly 1000 fine tubules. The tubules vary widely in number and location and their diameters range from 0.1 to 1.3 µ. Large amounts of sericin cover the column of the filament and fill spaces between the fibrils that make up threads spun into the cocoon shell (Fig. 18.3). Some Saturniidae species also spin porous cocoons.

Fiber porosity results from the release of lysosomes (membrane organelles) into the cell lumen, a holding site for transport of proteins away from the cell prior to silk secretion. Lysosomes break down the silk portein

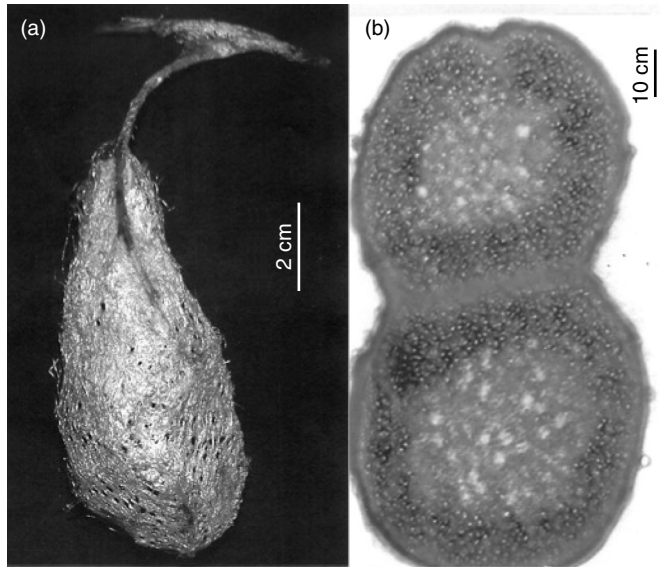


18.4 Cross-section of threads from different cocoon filament sizes of *B. mori.* Fibrils that make up the outer cocoon threads are larger and coated more heavily with sericin than fibrils that make up the inner cocoon layers.



18.5 Mixed porous and compact fibers produced by *Rothschildia*. Thread produced by *Rothshildia* is a mix of porous (p) and compact (*) fibrils. (Modified from Akai, 2001, #231.)

macromolecules into their component nucleic acids, amino acids and sugars. In all other groups of silk producing moths, the cuticular intima (inner lining of the silk gland membrane) acts as a barrier preventing lysosomes from passing into the cell lumen. Only in the Saturniidae does the cuticular intima degrade and allow lysosome secretion (Akai *et al.*, 2007). This finding may imply a unique interaction between the regulatory control of silk production and the initiation of metamorphosis in the Saturniidae silkworms (Craig, 2003; Sumida, 2001). Although all Saturniidae produce porous silk, lysosome secretion may not be continuous. In the case of *Rothschildia*, threads are a mix of porous and compact parts. The number and size of tubules is variable thoughout the silk shell (Fig. 18.5) (Akai, 2001). From a commercial perspective, the number of tubules in the thread reduces fiber weight. Hence the weight of the finished textile woven from Saturniidae silk maybe lighter than textile woven from silk produced by *Bombyx mori.*

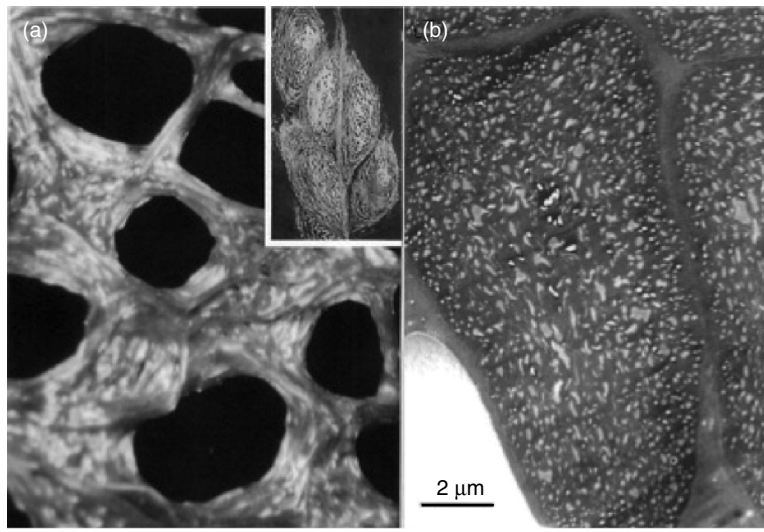


18.6 (a) Silver cocoon of *Argema mittrei*; (b) cross-section of cocoon filament from *A. mittrei.* The cocoons spun by *Argema mittrei* have a metallic, silver-like surface. The fibers that make the cocoons have a complex internal organization where large diameter tubules are clustered in the center of the fibril and smaller tubules are clustered around the outside. (Modified from Akai *et al.*, 2007.)

The comet moth from Madagascar, *Argema mittrei*, spins a very large cocoon that is white, porous, and the outside of the cocoon has a brilliant metallic-like sheen (Fig. 18.6). Individual filaments have a unique and ordered infrastructure and unlike *Rothschildia*, the central region of the fiber contains large tubules and the outer region is filled with fine tubules (Fig. 18.6, from Akai *et al.*, 2007). Thick layers of sericin cover the outside of the filament and fill the spaces between filaments bundles (Akai and Nagashima, 2002).

Cricula trifenestrata (Saturniidae) produces porous cocoons that are golden (Situmorang, 2002) (Fig. 18.7) and each silk filament is made up of more than 1000 extremely fine tubules. Furthermore, unlike the pigments produced by *Bombyx mandriana* the golden pigment produced by *Cricula* is incorporated into the fibroin as well as in the sericin coating the thread. As a result, processed threads retain their golden, shiny aspect (Yamada *et al.*, 2001).

Tasar silk production is a forest-based industry in both temperate (China, India) and tropical regions (India) (Ojha and Panday, 2004). Tasar silk has a lower breaking point than silk spun by *Bombyx mori*, but it is much more flexible. In fact, the mechanical properties of silk spun by *Antheraea* are

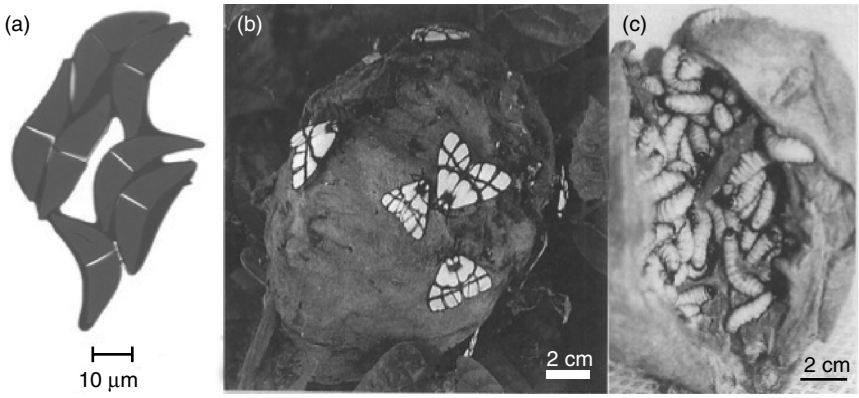


18.7 Cricular trifenestrata silk surface, cocoons and fine structure. (a) Surface of whole cocoon spun by *A. trifenestrata*. (b) Cross-section of cocoon thread shows that each fibril contains 1000s of fine tubules.

more similar to silk produced by the spider *Nephila madagascariensis* than they are to silk produced by *B. mori* (Komatsu, 1994). These differences probably reflect the different molecular organization and composition of silk spun by *Bombyx* and *Antheraea*. The repeat unit that composes the primary protein chain making up silk spun by *B. mori* is GlyAlaGlyAla (Zhou *et al*., 2000). The primary protein chain making up silk spun by *Antheraea* is a polyalanine chain followed by variable regions of amino acid sequence (Sezutsu and Yukuhiro, 2000). The dragline (MA) silk spun by *Nephila* is also polyalanine with similar non-polyaline regions as are found in *Antheraea.* In both *Nephila* and *Antheraea* silk, some of their specific amino acid repeats are considered genetic 'hotspots'. These similarities may suggest that spider *MA* silk and *A. peryni* silk are subject to similar rearrangements of amino acid sequence during replication that affect the silk's functional properties in different environments (Craig and Riekel, 2002). Silk spun by *A. yamamai*, *A. peryni and A. mylitta*, unlike the other wild silks discussed above, can be reeled. (but see Gheysens *et al*., 2011). *A. yamamai* wild silk has four times the value of *B. mori* silk due to its bluish color and shine (Akai, 2000).

18.3.3 Thaumetopoeidae

While most silk moths spin a single cocoon, the bag moths, for example moths in the genus *Anaphe*, aggregate to spin a communal nest. Four hundred or



18.8 Anaphe silk cross-section and nest. (a) Boat-shaped cross-section of cocoon filaments spun by *Anaphe* illustrates effect of spinneret morphology on fiber shape. (b) Outer, paper-like surface of *Anaphe* communal cocoon nest. (c) Cross-section of *Anaphe* communal nest opened to reveal larvae.

more larvae (Akai, 2000; Akai and Nagahima, 1999) spin individual cells into a soft, connective silk network that is bound to an outer, papery shell (Fig. 18.8).

The silk filaments spun by Thaumetopoeidae are compact but differ from other Lepidoptera silks in their crescent-shaped cross-section. Furthermore, the thread is jointed at intervals of 150–170 µ giving it a bamboo-like appearance (Akai and Nagahima, 1999). Recent analysis of the silk's amino acid content suggests that the primary and higher order structure of the protein is intermediate between the polyalanine silks produced by Saturniidae and the Ala-Gly silk produced by the Bombycidae (Tanaka *et al.*, 2008). Analyses of the mechanical properties of the silk show that it is much stiffer and characterized by a higher breaking point than silk spun by either the Bombycidae or Saturniidae. X-ray diffraction shows that the fiber structure is heterogeneous but mainly made up of ß-sheets (Tanaka and Asakura, 2009). Thaumetopoeidae silk is not shiny and hence the textiles spun from it tend to have a matt finish and are very soft (Akai, 2000).

18.3.4 Lasiocampidae

Silkworms in the genera *Gonometa* and *Borocera* (Lasiocampidae) spin compact silk fibrils and that are similar in microstructure to fibrils spun by *B. mori* (Akai, 2000). Unlike the extreme uniformity of *Bombyx* threads, however, silk threads spun by *Gonometa rufobrunnea* are variable in cross-section and can be round, triangular or even flat and ribbon-like (Freddi *et al.*, 1993). Larvae in the genera *Gonometa* and *Borocera* embed what

were, previously, protective body spines in the cocoon surface hence protecting the developing pupae (Peilger, 2004).

Furthermore, variation in cocoon fiber density or thickness may function to protect the chrysalis from desiccation. For example, *Borocera* species found in lowland, humid forests in Madagascar spin thin-shelled, lightweight cocoons while the cocoons spun by drier forest, highland species of *Borocera* are about three times as heavy (pers. obs., Craig). Cocoons spun by *Gonometa* collected in the acacia forest and dry lands of sub-Saharan Africa (Botswana, Kenya, Namibia, South Africa: Akai *et al*., 1997; Fening *et al*., 2010; Ngoka *et al*., 2008; Peilger, 1993, Veldtman *et al*., 2002;) and Kenya are heavier still. Unlike the large cocoons that are spun by Saturniidae that are porous and enhance drying in rainy and humid habitats as well as permit cooling, the seemingly impenetrable cocoons produced by *Gonometa* probably protect developing pupae from desiccation. Furthermore, calcium crystals, defecated during spinning, are packed in the surface of the cocoon shell and in the spaces between the outer and middle layers of cocoons (Akai, 2001). Methods to reel *Gonometa* cocoons have recently been published (Gheysens *et al.*, 2011).

18.3.5 Implications of diverse silk properties

While all of the types of silks we describe have been spun and woven into textiles, the physical properties of the thread and cocoons determine the spinning technologies that are used as well as the silk's potential for new commercial materials. For example, only three species of Saturniidae produce cocoons that have been reeled and hence can use the same processing technologies that are used for *B. mori.* All of the other wild silks discussed above are spun like wool or cotton.

Cocoon weight, when compared among species within a specific genus, is an important determinant of their economic value. For example, cocoons produced by *Borocera* in Madagascar's high central plateau are significantly heavier than cocoons produced by similar species found in lowlands. The weight of three to four cocoons produced by lowland *Borocera* species is equal to the weight of a single cocoon spun by highland *Borocera.* Hence, it would be difficult for lowland farmers to produce enough cocoons to make *Borocera* silk production economically viable (Razafimanantosoa *et al*., 2006). In contrast, wild silk farmers could produce cocoons spun by multi-elevation species, *Argema mittrei* or *Antherina suraka.* The metallic properties of the silk, their UV absorption (Akai *et al*., 1999) and porous fibers could result in new types of textiles as well as more economically competitive products. Understanding the different properties of silks (as well as silkworms and host plants) will allow innovative, wild silk farmers to better leverage their success.

18.4 Wild silk enterprise

The 'wild' silks reviewed here (i.e., non-*Bombyx* silk) all have significantly different protein composition and configuration than silks spun by *Bombyx* (cf. Sezutsu and Yukuhiro, 2000; Zhou *et al*., 2000). Hence, it would seem that they could be candidates for new industrial or biomedical uses that can exploit their specific molecular properties (Tanaka *et al*., 2008). Furthermore, their textures, colors and reflectance make them candidates for unique textiles and papers (Kuroda, 2000).

In India, and in the economically developing world, silk has long been an important cottage industry to backstop seasonal unemployment (Jayaram *et al*., 1998; Rani, 2007; Sahay *et al*., 1997). Currently, India is the largest producer of wild silk (the entire product is used internally) yet despite India's rich diversity of silkworms, only three wild species are used: *Antheraea mylitta*, Tasar silk; *Antheraea assama*, Muga; *Samia ricini*, Eri. Recent concern over habitat degradation and loss, as well as the disappearance of many caterpillar populations, has resulted in a resurgence of research efforts to identify the wild silkworm species and the ecoraces that remain (Kakati and Chutia, 2009; Nurmalitasari and Kuroda, 2002; Reddy *et al*., 2009; Saratchandra and Singh, 2002; Sharma and Sharma, 2006; Thangavelu *et al*., 2002) (Table 18.1).

While not as widely pursued as in Asia, wild silks have been collected in Africa for centuries by the Yoruba people (Gowdey, 1912; McKinney and Eicher, 2009) and since the tenth century in Madagascar (Kusimba, 2004). Recent surveys for additional wild silk moths in sub-Saharan Africa (Kioko, 2000) and Madagascar (Razafimanantosoa *et al*., 2006) have identified new species that may have economic value. In particular, projects in Kenya, Uganda, Namibia, Zimbabwe, South Africa and Madagascar are introducing small-scale, wild silk enterprise as a new livelihood for the rural poor and to promote the conservation of native habitats (e.g. Raina *et al.*, 2009, 2011).

18.4.1 Four wild silk projects with similar long-term goals

We analyze four wild silk projects implemented by Applied Technology India (AT India), International Center for Insect Physiology and Ecology (ICIPE), Ny Tanintsika (NyT) and Conservation through Poverty Alleviation, International (CPALI) designed to address poverty alleviation and conservation goals. Two of the projects make use of silkworms in the family Saturniidae and two of the projects make use of silk spun by silkworms in the family Lasiocampidae. Each of the four projects is organized differently and our goal is to determine whether some methods of implementation are more effective and economical than others. Table 18.2 summarizes the different project strategies, years of operation, mode of operation and funding base. Three of the projects reflect well-established

Table 18.2 Projects studied

	AT India	ICIPE	Ny Tanintsika	CPALI
Location	Uttaranchal India	Mwingi Kenya	Amoron'i Mania Madagascar	Makira Madagascar
Organization goals	Conservation and development	Research and development	Conservation and health care	Conservation
Project approach	Enhanced production	Sustainable resource extraction	Sustainable resource extraction	Enhanced production
Conservation goals	Resource management training, sustainable harvesting, reforestation	Resource management training, sustainable harvesting, reforestation	Sustainable resource extraction, reforestation	Enhance border forest value, avert added slash and burn agriculture
Status	Active	Active	Active	Pilot
Moth	*Antheraea mylitta*	*Gonometa postica*	*Borocera madagascariensis*	*Antherina suraka*
Cocoon size (g)	2	2	0.3	0.3
Value (US$)/ cocoon	0.42		0.0025	0.01
Habitat	Montane woodlands	Savanna woodlands	Highland woodlands	Multi-elevation, rain forest
First stage of project	Egg purchasing	Egg collecting	Cocoon collecting	Egg production
Finishing process	Weaving	Weaving	Weaving	Sewing
Product marketing	Finished products sold in country through AT India	Finished products sold in country through ICIPE	Finished products sold in country and externally to boutiques	Textile sold externally to product designers, product finishers
Silk produced/ year[a]	625 kg/month	416 k cocoons/3 years	83 k/month	2.0 kg/year/farmer (predicted) or 4000 cocoons

Funders[b]	BCN, UNDP, CSB, DOS, IFAD&GOU, USAID, ICEF, SPWD, Ford Foundation	SDC, IFAD, FAO, UNDP-GEF, Toyota, OPEC, CORDAID, Ford Foundation IDB, Biovision, Packard Foundation, World Bank	World Bank, FFEM/AFD Private donors	National Geographic, Fulbright Foundation, IRG, Rufford Small Grants for Conservation, Private donors

[a] Silk production/month: AT India, textile, pers. comm. M. Prakash, 2006; ICIPE, cocoons with pupae, 2005–2008, Raina et al., 2009; Ny Tanintsika, textile, pers. comm. E. Raharisoa, 2007; CPALI, cocoons, no pupae, pers. comm. M. Ratsimbazafy, 2009.

[b] All of funding data above are summarized from organization website or project reports. Funders listed stated to have been involved in silk production implementation and technology development. Nevertheless, funders of AT India and ICIPE projects supported a broad array of development and research initiatives in addition to silk production.

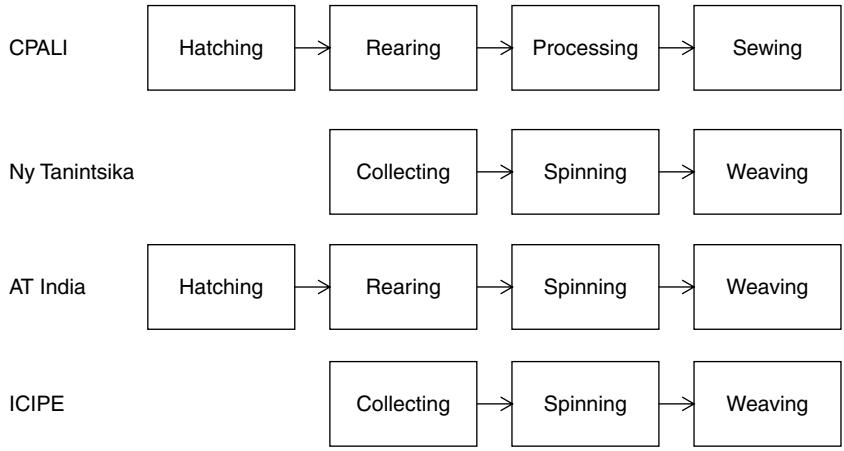
BCN, Biological Conservation Network; CORDAID, Catholic Organization for Relief and Development; CSB, Chemical Safety Board; DOS, Department of State; FAO, Food and Agriculture Organization; FFE/AFD, Fonds Français pour l'Environnement Mondial/Global Environmental Facility; GEF, Global Environmental Facility; IBD, Islamic Development Bank; ICEF, International Cultural & Educational Foundation; IFAD&GOU, International Fund for Agricultural Development/Government of Uganda; IRG, International Resources Group; OPEC, Organization of Petroleum Exporting Countries; SDC, Swiss Agency for Development and Cooperation; SPWDF, Society for the Promotion of Wastelands Development; UNDP, United Nations Development Programme; USAID, United States Aide for International Development.

programs that have been functioning for at least 10 years. The fourth project (CPALI) is still quite small and was first implemented in 2009. While investments across projects vary from thousands of dollars donated by private individuals to millions of dollars donated from international aid organizations (e.g. Swiss Agency for Development and Cooperation, UNDP GEF/IFAD, USAID; World Bank; Rockefeller Foundation), our analysis reveals all of the silk projects return roughly the same income to indigenous people when normalized by the per capita GDP of the host country. Total funds invested do not appear to correlate with environmental effectiveness or numbers of beneficiaries but rather with the proportion of funds devoted to project capital expenses or infrastructure.

18.4.2 Economic comparison methods

Elsewhere (Weber and Craig, in press) we have detailed the techno-economic model on which our analysis is based. Briefly, our model forecasts costs, revenues and benefits normalized by GDP of the host country. Inputs consist of cost of supplies, prices of the products and the biology of the moths (e.g. weight of the cocoons); outputs are the number of workers, amount of land and revenue associated with the scale of the project. We use Simulink®, a modeling environment produced by Mathworks (www.mathworks.com), to code both the overall model and utility routines. Each block in the model calculates the setup costs, yearly operating costs, value added, number of workers and, in the case of the rearing operations, the amount of land required. The value added by each operation is calculated simply as the difference between sales and operating costs, i.e., earnings before interest, taxes, depreciation and amortization. We have assigned values to the transfer costs of materiel between the stages of textile production. We use this model to compare, side by side, the economic and environmental aspects of wild silk projects in Madagascar, India and Kenya. Transfer costs for the intermediate products were scaled from prices of comparable products produced in India where there is a commercial market for cocoons, seed eggs and bulk yarn.

The techno-economic models were based on the sequence of activities required to cultivate the silk and convert it into products that could be exported and sold (Fig. 18.9). The projects, however, employ different sequences of operations, some starting from eggs that were reared industrially (i.e. Central Silk Board, India); others starting with cocoons that were collected from the forest (i.e. Ny Tanintsika). Likewise, there were different middle steps. NyT project participants purchase cocoons from collectors. The ICIPE and CPALI farmers rear larvae on cultivated host plants. Some ICIPE farmers collect cocoons from forest reserves. CPALI farmers are not



18.9 Organization of wild silk model and projects. The techno-economic model is made up of four sectors. Calculations determine how many trees, eggs, larvae must be raised to produce 1000 m^2 of textile and the cost of textile production. Textiles are either spun or sewed. The four projects described in the paper had different starting points for the silk production process. For example, the CPALI projects starts by producing eggs, the ATIndia project starts with rearing larvae and the NyT and ICIPE projects start with cocoon collection.

involved in any kind of forest extraction. CPALI breeders produce pupae and mate emerging moths. CPALI farmers put mated females under nets on host trees on which they lay eggs. AT India workers raise larvae in large forest enclosures on leaves that are collected from surrounding trees. Finally, the finishing technologies also varied. Participants in all but the CPALI project produce yarn that was woven into fabric. CPALI participants produce a non-spun textile based on an innovative, proprietary process of assembling whole cocoons into fabric. The process requires little training and little capital investment in comparison to the investments needed to spin and weave silk.

18.4.3 Model results

To make a quantitative, cross-country comparison that we hope correlates with broader and long-term project effects and implementation approach, we compare farmer earnings as an economic indicator, the number of people engaged as a social indicator and the number of trees planted as an indicator of environmental effect. Nevertheless, we recognize that economic gains are a necessary but not sufficient measure of poverty alleviation (Alkire and Santos, 2010; Sen, 1999) and that the numbers of trees planted is only a tangential measure of conservation impact or environmental restoration. We use a comparison of project capital and operating expenses to determine

Table 18.3 Model results metrics for producing 1000 m^2 of silk

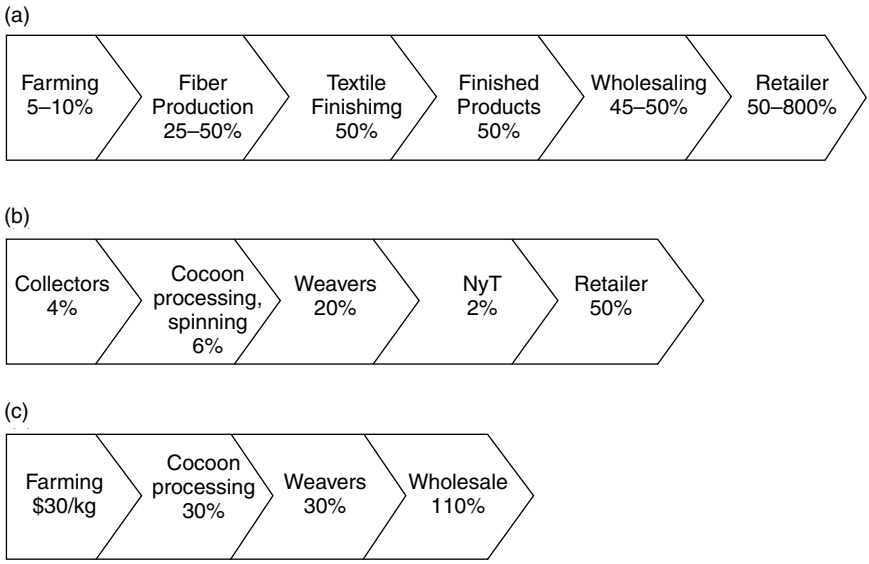
Metric	CPALI, predicted	Ny Tanintsika, measured	AT India, measured	ICIPE, measured
Workers	120	121	54	85
OPEX/USD	5700	1100	540	170
CAPEX/USD[b]	700	1200	5200	4800
Land planted (ha)	8.6	–	9	–
Trees planted[c]	21500	–	22500	–
Net revenue/worker (USD)	76	60	156	130
Net revenue per worker/ per capita (GDP)[a]	24%	24%	23%	22%

[a] Fixed costs depreciated by 20%/year.
[b] Trees planted are tree plantings for cocoon production. Ny Tanintsika has planted 85 000 moth host plants but current textile production costs are based on cocoons purchased from collectors. ICIPE has also planted seedlings to build border forest but cocoons are gathered from forest sites.
[c] GDP values from http://siteresources.worldbank.org/ICPINT/Resources/icp-final-tables.pdf.

how the organization of implementation approaches influences short-term effects on economic, social and environmental outcomes. The results for each production chain are summarized in Table 18.3. Each production chain is normalized to produce 1000 m^2/year fabric, either woven or nonwoven.

The analysis shows that the worker number varies across the projects by a factor of two despite the fact that the projects are normalized to produce the same volume of textile. These differences reflect project objectives, project design and the link on the product value chain where the project is implemented (Fig. 18.10). For example, most of the workers participating in the CPALI project are producing cocoons. CPALI designed its program as an approach to decrease habitat degradation around the Makira Protected Area (MPA), reduce illegal forest extraction and eliminate future slash and burn agriculture in the MPA. Therefore, CPALI targeted the bottom of the silk production chain value chain to develop a program that maximizes returns to farmers who have been most affected by protected area exclusion. Most of the workers in the ICIPE, AT India and NyT program work on silk processing and textile production.

We measure short-term environmental impact on the basis of number of trees planted that are used for silk production. AT India, ICIPE, NyT all have extensive nursery and tree planting programs and all three organizations are engaged in resource management training. However, there is not a direct link between trees planted and silk producer benefits. The CPALI program is designed differently. The project goal is protection of the Makira Protected Area and hence its focus is on farmers that have



18.10 Value chain comparison. Comparison of the value chain for three, wild silk, enterprise projects. The value chains are constructed according to project objectives: (a) AT India: general development; (b) NyT: women's poverty alleviation and (c) CPALI: replacement income for farmers.

been most affected by reserve restrictions. The amount of money that a CPALI farmer earns is in direct proportion to the number of trees planted and hence number of larvae the farmer is able to raise. The CPALI project participants may plant as many trees as the AT India and NyT projects, but unlike those projects, for CPALI farmers, tree survival is directly tied to farmer earnings.

The CPALI project incurs the highest operating expenses because it bears the cost of the entire production chain starting with eggs. Those costs were either assumed to be zero when the cocoons and eggs were collected (Ny Tanintsika and ICIPE) or in the case of India, subsidized by the government's Central Silk Board that provides farmers with disease-free eggs. After the CPALI program is established, village breeders will sell eggs to village farmers.

Finally, the analysis predicts that the capital expenses of the AT India and ICIPE projects are five times greater than the Ny Tanintsika project and an order of magnitude higher than the CPALI project. One reason for these differences may be that both AT India and ICIPE projects invested in infrastructure and equipment not available to NyT or CPALI. In particular, the ICIPE project has incurred the highest capital expense per worker

by building four market and weaving centers, and purchased large, industrial sized looms. In contrast, NyT processes silk locally and most women weave textiles on small, traditional, in-house looms as do participants in the AT India project. CPALI produces a non-spun textile that does not require spinning or weaving and can be made using a needle and thread or sewing machine.

18.4.4 Discussion of projects

The goal of the wild silk projects discussed has been to build development initiatives that are ecologically sustainable, that enable local people to support forest protection, and that allow indigenous populations to assume financial and social independence. Silk production results in many jobs that are associated with host plant care, larval care, silk processing, spinning and weaving. Textile production is relatively inexpensive to set up and can result in high value returns to local farmers and textile workers. The current low volume of commercially available wild silk results in an exclusive niche market where scarcity and naturalness are highly valued (Veldtman *et al.*, 2002). Thus achieving reasonable income returns through wild silk production should be relatively easy.

The returns and effectiveness of enterprise projects to meet conservation and poverty alleviation goals will depend on the stage in the product value chain that is targeted (Fig. 18.10). In the case of the CPALI project, higher returns are afforded to farmers because CPALI has greatly decreased textile production costs by designing a unique textile that circumvents the high equipment costs of spinning and weaving. While most product value chains return 5–10% to the farmer, the CPALI value chain returns 25%. The project goal is to directly affect border forest development through income generation, not just to redirect participants away from non-sustainable harvesting. The NyT project was designed as a rural, poverty alleviation initiative that focuses on women's economic empowerment (see Table 18.2). Therefore, NyT targets enterprise 'higher up' on the silk value chain to maximize returns to silk finishers and weavers. Our model suggests that the net revenue to CPALI farmers is about 10% higher then the net revenue of NyT spinners and weavers, probably due to the capital expenses involved in spinning and weaving. The AT India project was also designed as a rural poverty initiative as well as to protect the existing populations of Himalayan oaks. However, like the NyT program, it is designed to maximize the number of silk processors and weavers that the project employs. Relatively small numbers of workers rear silkworms in forest enclosures and sell their cocoons to spinners and weavers. Finally, the ICIPE program is primarily a research program focused on developing technical information on the natural history of silkworms found in target sites, as well as developing technology to spin wild silk cocoons and

weave textiles on industrial looms. The ICIPE program spends relatively little on operating expenses and invests more heavily in project capital expenses (looms, automated processing and spinning) and research.

18.5 Wild silk enterprise versus alternative conservation and poverty alleviation programs in Madagascar

Not all conservation and poverty alleviation programs are based directly on aid. Payments for Ecosystem Services (PES) are a market-based mechanism for conservation that works well in economically developed countries. PES programs are voluntary transactions where a well-defined environmental service is provided by a seller and purchased by a buyer based on a conditional cash transfer (CCT, Engle *et al*., 2008). Some PES services are user financed and some are government financed.

In countries where the majority of the population is made up of poor subsistence farmers, however, PES strategies can only be supported by long-term donor aid. In these cases, PES becomes a type of 'resource rent'. In Madagascar, the Durrell Foundation implemented a PES program through community-based competitions where cash-based incentives were awarded to communities most effectively managing their resources. While the Durrell program was generally considered a success, program longevity depends on Durrell's ability to continue to raise funds to support the program. The most difficult issues for Durrell to resolve are the continued raising of funds, the equitable distribution of benefits to all community members (Sommerville *et al*., 2010) and targeting those individuals who have the greatest incentive to use the forest.

Funds generated by REDD programs (Reducing Emissions from Deforestation and Degradation) are also considered to be PES programs although in reality first world countries 'rent' forests in developing countries to mitigate pollution. Forests sequester large amounts of carbon produced in the industrialized world. The transaction costs of setting up a carbon credit program including project design, monitoring and verification of emissions reductions, are between $50,000 and $200,000 (Doyle and Erdmann, 2009). Additional financing costs are incurred when governments and developers must register the projects before credits can be delivered. Given these high values, credit value to a landowner is likely to be significantly lower than the credit's market value (Doyle and Erdmann, 2009). Furthermore, landowners most affected by, and most likely to affect, forest conservation are often extremely isolated, making contingent cash payments difficult to deliver, verify and even use without additional development assistance. Finally, some developing country governments are hoping that carbon markets will

be a windfall for funding general fiscal needs making it even more unlikely that indigenous peoples will receive returns (Doyle and Erdmann, 2009).

18.6 Conclusion

We argue that small-scale enterprise may be the best way to affect conservation in politically unstable countries, and could be a first step to establishing better business practices, strategies and operations that will make larger-scale, eco-system approaches to conservation possible. For example, according to economic surveys in the CPALI project area, farmers stated that if they were able to earn $600 added income, they would forgo forest tavy (slash and burn agriculture) as well as all additional extraction of forest resources (Minten, 2003). Wild silk production represents one way that farmers add income to their earnings. CPALI pays farmers $30/kg of cocoons produced by *Antherina suraka*. Textile producers can further increase family incomes by $200–$300/year. Earnings are based on farmer performance and farmers are directly accountable for the returns received.

In the past, conservation and aid organizations have the value chain greatly underestimated the real costs of implementing and maintaining enterprise programs. When programs focus on the production of a commodity product, farmers must compete with well-established markets, (e.g. coffee, spices or domesticated silks; e.g. Lilieholm and Weatherly, 2010) as well as new synthetics that can replace natural products (e.g. vanilla) at much lower prices. We argue that conservation and poverty alleviation enterprise needs to focus on developing new and innovative products for niche markets and adapt products to changing economic conditions. Furthermore, new products need to be targeted to specific sites to reflect local resources. Successful scaling will require: (1) local ownership of the wild silk enterprise; (2) development of innovative products and connection to profitable markets as well local authority over the use of market returns; (3) the organization can insure that returns are distributed fairly. Wild silk enterprise is not a panacea but it is one enterprise that has a 2000+ year track record of success (Reddy, 2010) that can be built upon.

18.7 References

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