4

Lyocell: the production process and market development

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4.1 Overview

Lyocell is the first in a new generation of cellulosic fibres. The development of lyocell was driven by the desire for a cellulosic fibre which exhibited an improved cost/performance profile compared to viscose rayon. The other main driving force was the continuing demands for industrial processes to become more environmentally responsible and utilise renewable resources as their raw materials. The resultant lyocell fibre meets both demands.

Lyocell was originally conceived as a textile fibre. The first commercial samples were produced in 1984 and fibre production has been increasing rapidly ever since. Fabrics made from lyocell can be engineered to produce a wide range of drapes (how the fabric hangs), handles (how the fabric feels) and unique aesthetic effects. It is very versatile and can be fabricated into a wide range of different fabric weights from women's lightweight blouse fabric through to men's suiting.

Other end-uses, such as nonwoven fabrics and papers, are being developed. These non-textile end-uses will become progressively more important as the special properties of lyocell fibres enables products with enhanced performance characteristics to be developed.

Lyocell is a 100% cellulosic fibre derived from wood-pulp produced from sustainable managed forests. The wood-pulp is dissolved in a solution of hot *N*-methyl morpholine oxide (abbreviated to NMMO or amine oxide in this text). The solution is then extruded (spun) into fibres and the solvent extracted as the fibres pass through a washing process. The manufacturing process is designed to recover >99% of the solvent, helping minimise the effluent. The solvent itself is non-toxic and all the effluent produced is non-hazardous.

The direct dissolution of the cellulose in an organic solvent without the formation of an intermediate compound differentiates the new generation of cellulosic fibres, including lyocell, from other cellulosic fibres such as viscose. This has led to the new generic name 'lyocell' being accepted for labelling purposes.

Lyocell has all the benefits of being a cellulosic fibre, in that it is fully biodegradable, it is absorbent and the handle can be changed significantly by the use of enzymes or chemical finishing techniques. It has a relatively high strength in both the wet and dry state which allows for the production of finer yarns and lighter fabrics. The high strength also facilitates its use in various mechanical and chemical finishing treatments both under conventional and extreme conditions. The physical characteristics of lyocell also result in its excellent blending characteristics with fibres such as linen, cashmere, silk and wool.

In common with other highly oriented cellulosic fibres, such as cotton, cuprammonium and polynosic rayon, lyocell fibrillates but its ease of fibrillation is greater. Fibrillation becomes apparent when the fibre is abraded in the wet state and surface fibrils (small fibre-like structures) peel away from the main body of the fibre but remain attached. The fibrillation behaviour of the fibres can be exploited by using a variety of different mechanical, chemical and enzyme treatments to produce a vast range of fabric aesthetics. The control of the fibre's fibrillation behaviour, both to increase and decrease fibrillation, is a major area of continuing research.

4.2 Amine oxide technology – timeline

1939	Patent appears describing the dissolution of cellulose in amine oxide.
1966–1968	D L Johnson of Eastman Kodak Inc. publishes a series of papers discussing a range of compounds, including cellulose, which dissolve in amine oxide.
1969–1979	American Enka/Akzona Inc. work on spinning fibre from a solution of cellulose in amine oxide but decide not to scale up production.
1979	Courtaulds start research on the new cellulosic fibre, which is to become Tencel®.
1983	Pilot plant tow/staple line built in Coventry, UK.
1984	First commercial staple samples produced.
1988	Small commercial plant set up at Grimsby, UK.
1989	BISFA (Bureau International pour la Standardisation de la Rayonne et des Fibres Synthétiques) agrees to new generic name – lyocell.
1992	Full-scale production plant set up at Mobile, USA.
1996	Second stage expansion takes place in Mobile.

1997	Lenzing starts a production plant in Austria.
1998	Courtaulds* starts a new plant in Grimsby, UK.

Lyocell was first made in the laboratory and then on the pilot plant at Coventry during the early 1980s. In 1988 a semi-commercial plant (capacity 30 tonnes/week) was started up at Grimsby, this plant is known as S25 because originally there were 25 spinning ends. Courtaulds invested around £6m in this plant primarily to develop the lyocell process technology. Although the factory is capable of operating at a profit it was never intended that it would recoup the capital investment. The operation of S25 enabled the manufacturing process to be proven and developed and provided enough fibre to initiate full-scale market development. S25 now concentrates on process and product development together with some more speciality lyocell products.

In May/June 1992 the first full-scale commercial factory (SL1 – Spinning Line 1) was commissioned in Mobile, Alabama. Courtaulds invested some £67m in this venture. The process and market scale-up represented a not inconsiderable risk to Courtaulds. However, the success of producing and selling fibre from SL1 convinced the board to invest heavily in the future of lyocell.

Acordis's principal competitor in making lyocell is Lenzing of Austria who commenced production in 1997.

4.2.1 Expansion of production capacity

Courtaulds initial target was to have 150 000 tonnes/year production capacity on stream by the end of 1999. If we consider that SL1 can produce around 16 000 tonnes/year and this took nearly 3 years to design, construct and start up, the magnitude of the expansion plan becomes apparent.

The second plant, SL2, started up alongside SL1 at Mobile in the summer of 1995 with a capacity of around 20 000 tonnes/year, expandable to 30 000 tonnes/year by the construction of a third solution, spinning and fibre line. Approximately £90m was invested in this project.

The third plant was planned for Europe (Grimsby), for start up at the end of 1995 or early 1996. This was to be of a similar design to SL2 and with a capacity of around 20 000 tonnes/year also expandable to 30 000 tonnes/year by the construction of a third solution, spinning and fibre line. A fourth plant was initially planned for somewhere in the Far East.

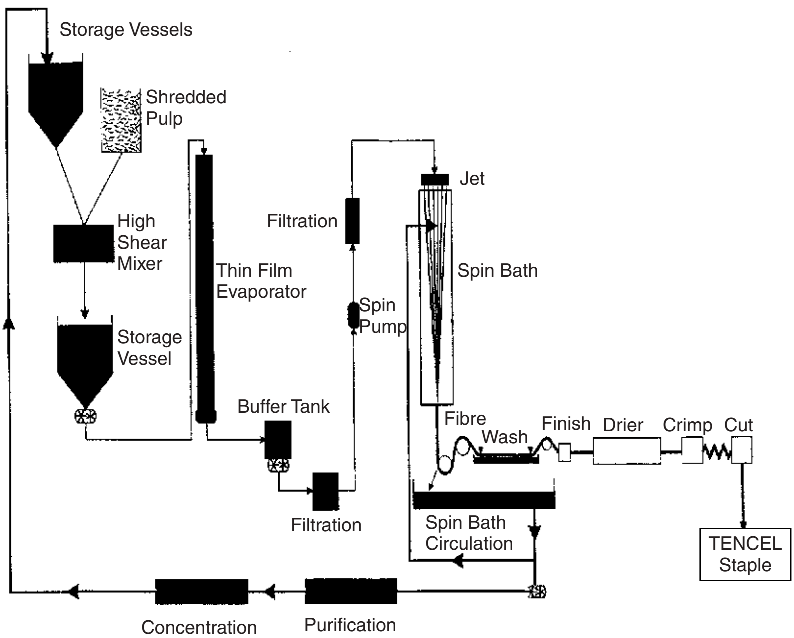
* Courtaulds was taken over by Akzo-Nobel and the combined fibre operations renamed 'Acordis' during the year.

[In the event, the 1997 collapse of Far Eastern markets, and the reduction in demand for denim (see later) resulted in the postponement of the Far East plans and the construction of a bigger Grimsby plant which started in 1998. Ed]

4.3 Process description

This section provides a description of the process steps required for making lyocell. A block diagram of the process is shown in Fig. 4.1.

The principles of the process are simple. The pulp is first wetted out with dilute aqueous amine oxide to penetrate the pulp fibres fully. The subsequent removal of excess water is a very effective way of making a homogenous solution with a minimum of undissolved pulp particles and air bubbles. The solution is highly viscous at its operating temperature (90–120°C) and must be processed in similar high pressure equipment to that used in melt polymer systems. The fibres are formed by spinning into an air gap and then coagulating in a water/amine oxide bath. They are then washed and dried. The wash liquors are recovered, purified, concentrated then recycled.



4.1 The Acordis Tencel® process. (Reproduced by courtesy of Acordis.) NMMO solution recovered from wash and spin-bath liquids is purified and concentrated and returned to the upper storage vessel for re-use in mixing. Unlike both viscose staple and the Lenzing Lyocell process, the fibre is washed, dried and crimped in tow form prior to cutting.

The process description below applies to the two commercial scale operations of Acordis and Lenzing. Variations in detail have been cited in patent applications and the literature but these are at a much smaller scale of operation.

4.3.1 Pulp and premix

Pulp is the principle raw material of the lyocell process in terms of cost and volume. In order to achieve the high quality properties of lyocell fibre, the pulp has to be of a good quality. Typically the degree of polymerisation (DP) of the pulp is in the range 400–1000 units – Tencel® fibres have a DP of 500 to 550.

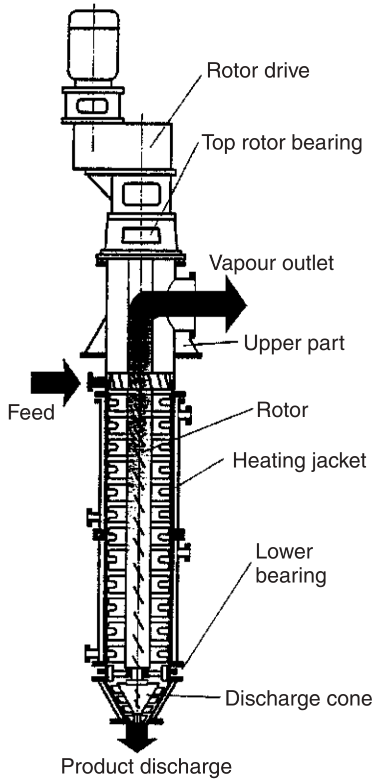
The pulp is pulled from the reels into a shredder which cuts the pulp into small pieces for mixing with the amine oxide solvent. The amount of pulp fed to the mixer has to be measured accurately so that the cellulose content in solution is closely controlled.

The cut pulp is conveyed to vessels where it is mixed with a 76–78% amine oxide solution in water. A small quantity of a degradation inhibitor is also added to the mixer, other additives such as titanium dioxide (for producing matt fibre) can also be added. The mixing is achieved at 70–90°C in a ploughshare mixer that contains a number of high speed refiners to break the pulp down and aid solvent wetting. The resultant slurry consists of swollen pulp fibres and has the consistency of dough. This premix is dropped into an agitated storage hopper from which it is accurately metered to the next stage of the process.

4.3.2 Solution making

Premix is heated under vacuum to remove sufficient water to give a clear dark amber-coloured viscous solution of the cellulose. Typically the solutions contain 10–18% cellulose.

The evaporation of water from premix to make solution is achieved in a wiped thin film evaporator. This is a long vertical cylindrical vessel with steam heating in jackets around the vessel. A shaft down the centre of the vessel with blades attached to its circumference is rotated to smear the material around the heated surface to promote the evaporation process and to transport the solution down the vessel. The position, number, shape, size and type of these blades are chosen to maximise the output of the solution evaporator. The evaporator vessel is operated under vacuum to reduce the temperature (c. 90–120°C) at which the water evaporates, this is important because the amine oxide solvent in solution can undergo an exothermic degradation process if it is overheated. The specific evaporator used is a Filmtruder, shown in Fig. 4.2.



4.2 Thin-wall evaporator (Buss Filmtruder) where the cellulose dissolves as the solvent is concentrated by water evaporation. (Reproduced by courtesy of Acordis.)

4.3.3 Solution transport

The solution leaving the Filmtruder is pumped by a number of specialised pumps in series through the transport system. The transport system consists of a solution cooler and a hydraulic ram buffer tank which feed into the solution primary filters. Owing to the viscous nature of the solution the pressures involved in pumping the solution can be as high as 180 bar.

A complication of the process design is caused by the tendency of the amine oxide in solution to degrade exothermically. Exotherms can be caused by faulty operation of equipment or chemical contamination of the solution. If an exotherm occurs, the temperature of the solution increases rapidly and it decomposes to volatile amines and water. This causes very rapid increase in pressure that would be sufficient to rupture the high pressure equipment with very serious safety implications. To allow for this possibility, bursting discs are provided at strategic positions throughout the plant to give pressure relief in the event of an exotherm. The bursting discs are of a special design to prevent any flow dead spots. The discs vent into disentrainment pots which separate the solid degradation products and allow the gases to be vented to atmosphere. Understanding how and why exotherms occur and the development of a safe way of venting exotherms when they do occur were keys to the scale-up of the whole process.

4.3.4 Solution filtration

Prior to spinning it is necessary to filter various impurities out of the solution. Most of the impurities are introduced with the pulp feedstock, the principle ones being undissolved pulp fibres or inorganic compounds such as sand and ash.

The solution is passed through two stages of filtration. The primary filtration is centrally located and consists of sets of sintered stainless steel media candle filter elements. The secondary stage of filtration is achieved by candle filter elements associated with each spinning machine position.

Filters are washed for re-use by an off-line process involving rinsing with hot amine oxide, chemically decomposing the residual compounds and then ultrasonic washing.

4.3.5 Spinning

For spinning, the solution is split into substreams which serve a number of spinning positions. The solution is then supplied to each jet, via a filter, by a metering pump. It is then extruded and spun through an air gap into a spin bath containing dilute amine oxide solution.

Each jet consists of thousands of tiny holes through which the solution is extruded into fibres. Just below each jet face is a small air gap across which air is blown by the cross-draught system to condition the fibres. After passing through the air gap the fibres, or tow, are pulled down through the spin bath where the cellulose is regenerated in dilute solvent. The fibres are drawn, or stretched, in the air gap by the pull of traction units, or godets.

The design of the spinning assembly proved critical to the successful scale-up of the process and achievement of commercially attractive fibre properties and manufacturing costs.

Spinning the solution directly into an aqueous bath necessitates using very dilute cellulose solutions (producing high costs) and generates fibres with properties that are generally inferior to viscose. Extruding the solution into an air gap enables more economical, higher cellulose solutions to be spun. Furthermore, when the solution is extended or drawn in the air gap it is also oriented so that good strength and elongation properties can be imparted. A draw ratio of between 4 and 20 is typical – within this range fibre properties are similar. At lower ratios fibre tenacities are reduced and at higher ratios spinning stability deteriorates.

When the solution is drawn in the air gap it will readily break unless it is also cooled by means of a gas flow. This tendency to rupture is worse at higher draw ratios and spinning speeds but is reduced if highly viscous solutions are used. This necessitates the use of relatively small spinneret holes relative to the high polymer viscosity so that special jets needed to be designed.

The main limitation of air-gap spinning is the tendency of neighbouring filaments to touch and stick together. This limits the packing density of the spinneret and hence the productivity of the spinning machines. This is compounded by the need to cool all the filaments adequately using an air flow velocity sufficiently low that it does not disrupt the stable flow of the filaments.

The two commercial processes overcome these constraints in different ways. Acordis arranges the spinnerets into rectangular strips whereas Lenzing uses a circular array. Both processes use a controlled flow of gas across the filament arrays to stabilise and control the process.

4.3.6 Fibre washing

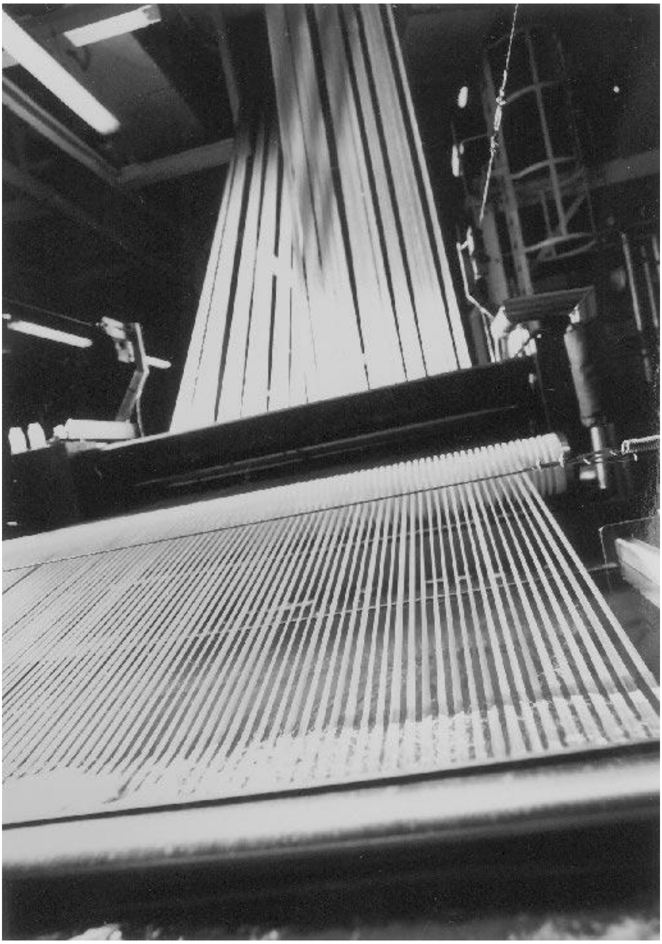
The fibre tows from each end are brought together into one large tow band for processing down the fibre line.

The first process on the fibre line is washing, the solvent is washed from the fibre with hot demineralised water in a series of wash baths. In the Tencel® process the fibre is washed as a single large continuous tow through a series of wash troughs each of which consists of a wide shallow bath containing a number of wedges. These wedges deflect the tow band alternately up and down as it is pulled along the trough, which serves to allow dilute solvent into the tow band where it is then squeezed out. Wash liquor leaving the wash line goes into the spin bath system. The washing water is fed countercurrent to the tow band at a rate to keep the spin bath liquor concentration at the required level (see Fig. 4.3).

4.3.7 Fibre treatments

After washing the fibre is treated in a number of ways:

- The fibre could be bleached if required.
- Soft finish is always applied to make processing easier.
- Antistat is applied.
- Other treatments to give specific fibre properties can also be performed.



4.3 Lyocell tows entering the tow-washing process. (Reproduced by courtesy of Acordis.)

4.3.8 Fibre drying

After washing and finishing the fibre is dried in conventional fibre drum dryers. These consist of a series of perforated drums that the fibre passes over. Steam heated air is sucked through the fibre as it passes over the drums and exhausted to atmosphere (see Fig. 4.4).

4.3.9 Crimping, cutting and baling

Dry fibre is crimped before being fed to a radial blade cutter for cutting into staple and then baled. The bales of staple fibre are then dispatched to customers (see Fig. 4.5).

4.4 Inspecting lyocell tow as it is condensed after drying and prior to crimping. (Reproduced by courtesy of Acordis.)

4.5 Bales of lyocell ready for despatch. (Reproduced by courtesy of Acordis.)

Lenzing use a process where the fibres are cut after spinning and it is then very similar to the wash/dry process used in viscose.

4.3.10 Solvent recovery

Diluted amine oxide solvent from the spin baths on the spinning machine and various other process sections is collected to recover the relatively expensive solvent. Over 99% of the solvent used is recycled and recovered by the process.

The amine oxide will slowly oxidise the cellulose during the process, particularly at the elevated temperatures often used. The reaction will reduce the DP of the cellulose (giving poorer fibre properties) as well as generating coloured compounds that would detract from the whiteness of the fibres. The amine oxide degrades to *N*-methyl morpholine plus other amines. The reaction is strongly catalysed by transition metals such as copper and iron. To control this it is essential that a stabiliser such as propyl gallate is incorporated – this appears to act both as an antioxidant and a chelating agent.

Solvent recovery consists of two main processes, ion exchange of the dilute solvent, then evaporation of the excess water to a concentration required in premixing.

The ion exchange process consists of cation and anion beds which remove various ions that would destabilise the solution and the colour contaminants that would otherwise build up in the solvent. The ion exchange resins and regeneration procedures have been developed especially for amine oxide.

The amine oxide is reconcentrated in a steam heated multiple effect falling thin film evaporator. The tendency of the solvent to degrade exothermically means that the process control of the operation has to be such that the amine oxide can never be overheated. The water overheads can be re-used to wash the fibres so minimising the environmental impact of the process.

4.4 Lyocell conversion

Lyocell is similar in strength to polyester and stronger than cotton and all other man-made staple fibre cellulosics. It also has very high dry and wet moduli for a cellulosic fibre in both the dry and wet states. These properties (see Chapter 8 for details) allow customers great scope for making strong yarns in blend with virtually all the other commercially available staple fibres. They also lead to excellent efficiencies in converting these yarns to woven and knitted fabrics.

All man made cellulosics lose strength and modulus when wetted, but lyocell reduces by much less than the others. This is important in determining how the properties of the fabric are developed during dyeing and finishing. However the fibres do fibrillate during wet abrasion and thus specific techniques are required to achieve the best results – these are discussed later.

Once lyocell fibre has been produced, either as cut staple fibre or continuous tow it will be converted to yarns and fabrics by a range of conventional textile processes. The most common way of using lyocell fibre is as cut staple, with 1.4 and 1.7 dtex fibres being cut to 38 mm and converted into a spun yarn using machinery developed over many years for handling cotton fibres which are similar in linear density and length to lyocell.

The following comments apply to the processing of Tencel® fibres. The Lenzing Lyocell is made by a wet cut route and has very different processing characteristics.

4.4.1 Yarn manufacture

TENCEL® can be processed via established yarn manufacturing routes, using conventional machinery with few major changes to settings or procedures. Its processing performance is influenced by the following properties:

- it possesses a non-durable crimp;
- it has a high modulus;
- there is little fibre entanglement.

Thus TENCEL® will open easily with little nep. In sliver and roving, the fibres pack well together, give high cohesion and require high draft forces. It yields yarns with high tensile strength and few imperfections. It blends well with other fibres, especially other cellulosics. It adds strength to the final yarn and enhances the performance and aesthetic values of final fabrics.

The following comments refer to fibre processed by the cotton system.

4.4.1.1 Blending and carding

The open state of the fibre tufts facilitates ease of bale skimming, bales rise only a small amount on opening, are extremely stable and are consistent in openness. The production of TENCEL® staple fibres ensures that the filaments are not entangled and that removal of foreign matter is not required. Hence minimal opening is required to separate the fibre tufts ready for carding. It should be remembered that each additional machine in the opening line will add nep.

The vast majority of short staple TENCEL® fibre used in yarn manufacture is carded using chute feeds and revolving flat cards. Conventional settings are often employed, but particular attention is needed in chutes and card web take-off mechanisms. Few changes to conventional card settings are required to achieve even webs. Card wire should be suitable for 1.7 dtex and finer fibres.

4.4.2 Lyocell in nonwovens and papers

Although lyocell is primarily used as a textile fibre, it offers significant potential in both nonwoven fabrics and papers owing to its strength, biodegradability and potential for fibrillation. Only very small amounts of lyocell are presently used for nonwovens, but the importance of these end-uses will increase as lyocell capacity grows.

4.4.3 Nonwovens

A nonwoven is broadly defined as a 'textile structure made directly from fibre rather than yarn'. The fabric is usually made by producing a 'web' of fibres which is then strengthened by 'bonding' using various techniques, for example:

- Thermal bonding – bonding carried out by the application of heat
- Hydroentanglement – bonding achieved by entangling the fibres using very fine jets of high pressure water (see Fig. 4.6)
- Needle bonding – the fibres are entangled by a set of barbed needles which are punched through the web.

The key properties of lyocell which make it suitable for nonwovens are:

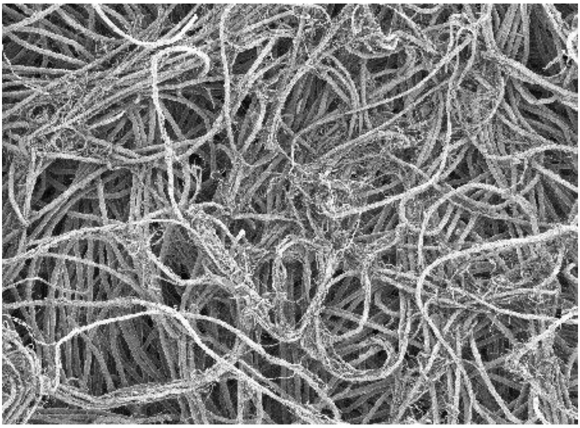
- high strength (dry and wet)
- biodegradability
- easy processing
- absorbency
- potential to fibrillate (in wet processes).

Preparation of the fibre web can be done in many different ways, however two main methods are carding and wet-laying.

Not all webs are suitable for bonding by all methods, however, both wet-laid and carded webs are suitable for hydroentanglement. This is the area in which work has concentrated on lyocell, owing to the fibre's ability to fibrillate in wet processes. Hydroentanglement produces bonding by entangling the fibres together. This is done by passing the web under rows of high pressure water jets (up to 200 bar). As this is a wet abrasive process lyocell can be made to fibrillate at high water pressures, producing submicron fibrils, which enhance filtration properties.

Hydroentanglement gives strong lyocell fabrics, which compare well to polyester and are stronger than viscose.

Lyocell can be bonded using a wide range of other techniques, which



4.6 Surface of a durable wipe made by high pressure entanglement of 100% lyocell. The microfibrils give the surface a chamois-leather like texture.

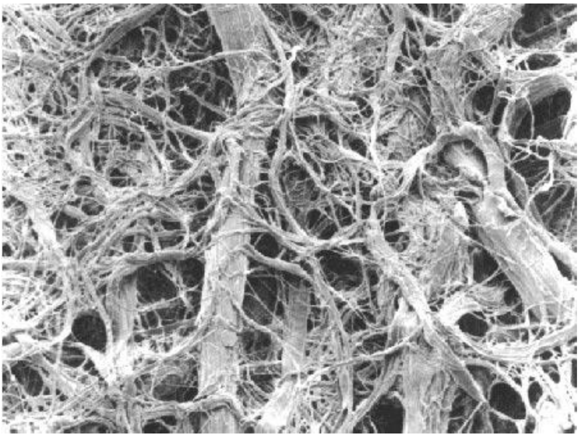
will not be discussed in detail here. It is being trialled and developed in non-wovens for a wide range of end-uses, including:

- surgical swabs, drapes and gowns
- floppy disc liners
- filtration applications
- semi-disposable workwear
- lining materials.

4.4.4 Papers

The ability of lyocell to fibrillate means that it can be processed and made into a paper, like wood pulp (see Fig. 4.7). The main steps in paper making are:

1) Beating and/or refining – these two processes take a dilute suspension (stock) of short fibre (around 5 mm), and mechanically treat the fibre in order to fibrillate it. This is done either by passing it between a plate and a large grooved roller (beating) or pumping it through a pair of rotating grooved discs (refining). The forces exerted on the fibres cause the fibre to fibrillate, and can also cut the fibre (depending on the severity of the treatment).
2) Sheet formation – the refined stock is diluted further and then pumped to the paper machine where it is deposited onto a porous metal belt or 'wire'. The water is sucked through the belt, leaving the fibre on top as a paper sheet. The paper is then passed through a series of drying cylinders and presses before being wound on a roll.



4.7 Surface of 100% lyocell paper held together purely by hydrogen bonding between the high surface area fibrils.

Lyocell can be processed into a strong paper. The properties depend upon the amount of fibrillation generated – increased fibrillation gives more paper strength, and also affects other properties. In general lyocell papers are strong, with good opacity and low air resistance owing to the circular nature of the fibres and fibrils. The end-uses being explored are therefore mainly filtration applications, and also as an additive to improve the properties (e.g. strength) of a standard paper made from wood pulp.

4.5 Dyeing and finishing of lyocell

The dyeing and finishing of lyocell fabrics is the key to their success. There are three characteristics of the fibres that can be manipulated to give fabrics with attractive and differentiated aesthetics – the ease of fibrillation, the high nodulus and the wet swelling characteristics. Fibrillation can yield the characteristic 'peach skin' surface touch of fabrics made from this fibre, but unwanted and uncontrolled fibrillation can also impair the fabric quality. Much of the dyeing and finishing development has been focused on this aspect. The high modulus or stiffness of lyocell contributes to the full handle of lyocell fabrics but allowances must be made in dyeing/finishing procedures to accommodate this property. The high level of wet swelling, particularly in caustic soda, allows fabric to process through dyeing and finishing more readily and yields a full and flexible handle. However, this swelling can cause crease damage to the fabrics if they are not processed correctly. Thus, although lyocell is broadly similar to cotton and viscose in preparation, dyeing and finishing, converters should refer to the manufacturer's current technical information sheets for specific advice on the details.

4.5.1 Dyeing of lyocell

As lyocell is a cellulosic fibre, it can be dyed with colours normally used on cotton. Compared with unmercerised cotton, lyocell, except with a few reactive and vat dyes and a number of direct dyes (pale shades), dyes to a heavier depth by exhaust techniques and therefore many shades can be attained at a lower cost, particularly with reactive colours.

Vat, direct and reactive dyes are the principal classes of dye used on lyocell. The actual choice of the dye class depends, amongst other things, on the extent to which the dyes and dyeing methods are economical, brightness of shade, fastness requirement, machine availability, and processing reliability.

4.5.2 Dyeing mechanism

The dyeing mechanism for most classes of reactive dyes is very similar. First the reactive dye is exhausted on to the cellulose fibre using salt. In the second stage of dyeing, alkali is added to fix the dye.

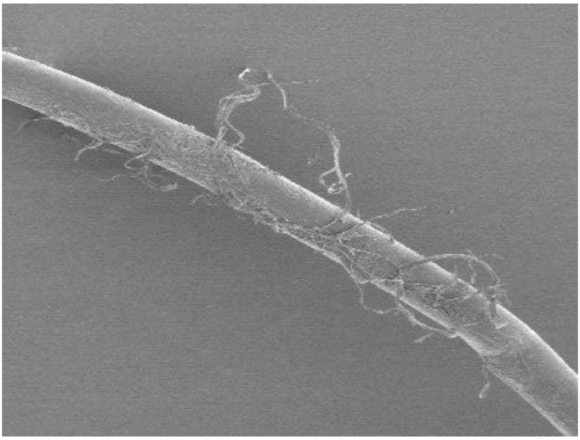
Many of the modern reactive dyestuffs contain two or three reactive groups. A key discovery, made early in the development of lyocell, was that these multifunctional dyestuffs can crosslink the fibre and thereby prevent or inhibit the fibrillation of the fibre. Since manipulation of this fibrillation is critical for the development of the fabric aesthetics (see later), understanding how to control this crosslinking by the dyestuffs is also of critical importance.

This discovery also led to the patenting of ‘colourless dyes’ as a means of preventing fibrillation of lyocell fibres. The critical benefits of this approach are that the good physical properties of the fibre are maintained together with a good dye affinity. Most conventional cellulose crosslinkers, for example easy-care resins, severely inhibit both characteristics.

4.5.3 Control and use of fibrillation

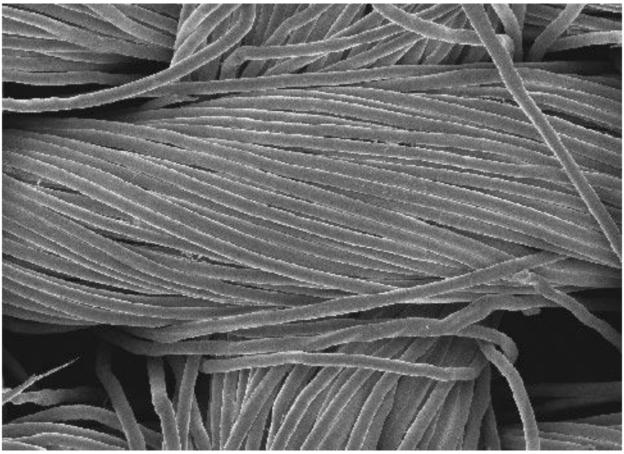
Fibrillation can be defined as the longitudinal splitting of a single fibre filament into microfibres (see Fig. 4.8). The splitting occurs as a result of wet abrasion, particularly against metal. The fibrils formed can be so fine that they become virtually transparent and give a frosty appearance to the finished fabric.

The samples in Fig. 4.9 show an example of a non-fibrillated (a) and a highly fibrillated (b) lyocell fabric. The fibrillated fabric gives a frosty appearance. In cases of extreme fibrillation, the loose fibres on the surface of the fabric fibrillate and then tangle together to form very light coloured pills. The appearance of the fabric then becomes totally unacceptable.



4.8 Micrograph showing fibrillation of a single fibre.

(a)



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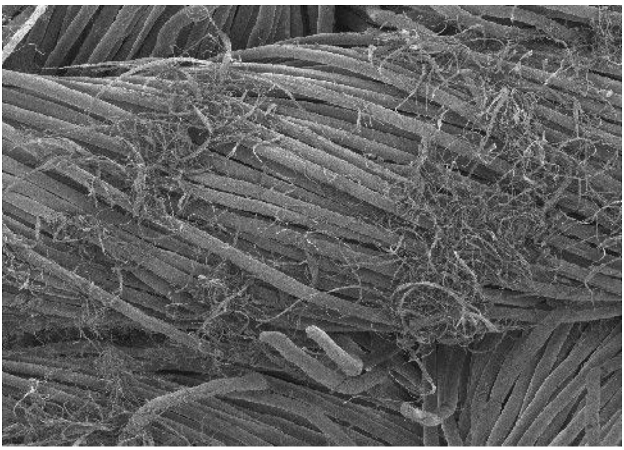


4.9 (a) Woven fabric surface before fibrillation. (b) Woven fabric surface after fibrillation.

4.5.4 What causes fibrillation?

Any process that abrades the fibre in a wet condition will generate some fibrillation. Processing of the fabric in rope dyeing equipment (jets, winches) where the fabric rubs against itself and metal can lead to fibrillation. A number of factors will accelerate fibrillation, such as high pH, high temperature, lack of lubrication, high machine loadings and vigorous machine action. Three methods are being employed to control fibrillation and details of these are given below.

4.5.4.1 Application of enzymes

Once fibrillation has occurred it can be removed by the use of specific cellulase enzymes. These need to be carefully controlled, but are very effective at polishing the fabric surface to remove any unacceptable fibrillation.

Enzymes will not prevent the recurrence of fibrillation of the fibres but, in conjunction with the optimum processing procedures – see later – they can give very attractive and durable fabrics.

4.5.4.2 Application of easy care resins

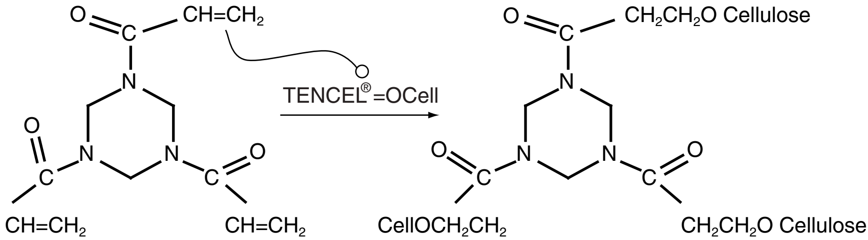
Resins which can crosslink with the fabric are frequently used after dyeing. This embrittles the fibrils and enables any fibrillation occurring during the dyeing process to be easily removed. This process is particularly suited to woven fabrics as these are prepared and dyed open width and so are free of fibrillation before dyeing.

Resin finishing can have a significant influence on the final aesthetic of lyocell fabrics. However, the amount of finish required to give the resistance to fibrillation and easy care performance is lower than for cotton so much of the natural softness of lyocell can be retained.

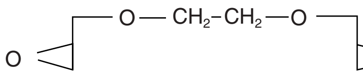
Lyocell fabrics produced by this route have clean bright colouration, a full, soft aesthetic and an excellent performance in use. They have proved very comfortable to wear, are durable and retain their ‘as new’ aesthetic. In blend with polyester they are proving to be excellent for industrial applications, workwear and careerwear.

4.5.4.3 Application of crosslinking chemicals

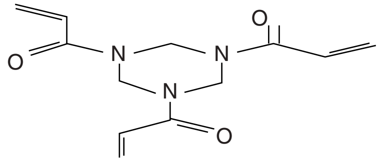
Lyocell’s tendency to fibrillate can also be alleviated by the use of compounds which crosslink with the cellulose chains (see Fig. 4.10). Two chemicals have been marketed as fabric treatments, Axis (from Tencel Ltd) and Ciba 4425 (see Fig. 4.11). Both these compounds will crosslink with lyocell under alkaline conditions either before or after the fabic has been dyed. Treated lyocell is both rope dyeable and durable to laundering and there are no significant changes in fabric properties.



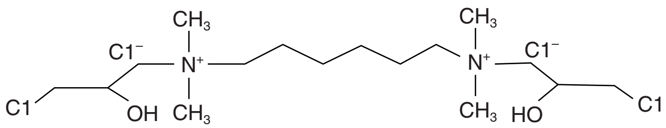
4.10 Crosslinking with triacrylamido-trihydrotrazine. (Reproduced by courtesy of Acordis.) This trifunctional colourless dye crosslinks the fibrillar structure of lyocell and allows fabrics to be processed with minimal fibrillation.



Ethylene glycol-diglycidylether

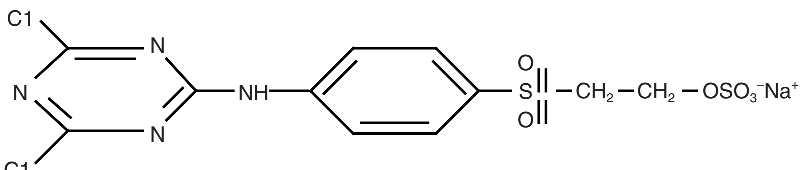


Triacrylamido-trihydrotriazine
AXIS



Bis-hydroxychloropropyl-*N*, *N*-tetramethyl-hexamethylenediamine

CATIONON

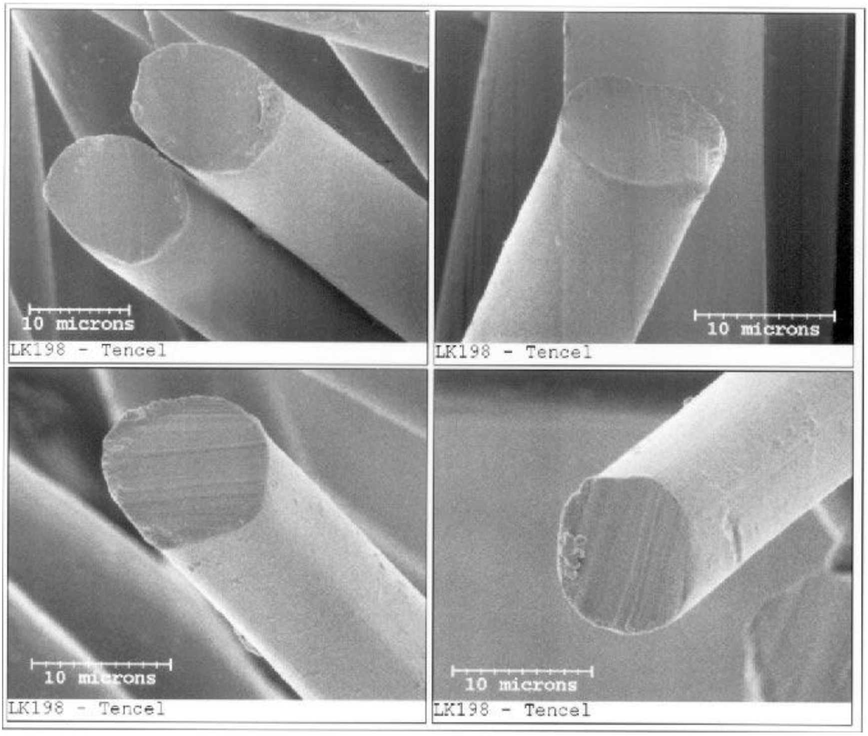


Cibatex AE 4425

4.11 Crosslinking: some of the compounds that can be used to control fibrillation in lyocell processing Axis and Cibatex AE 4425 are used commercially, the other two have been shown to work on a small scale (Reproduced by courtesy of Acordis.)

Figure 4.12 shows lyocell fibre SEMs.

Pre-treatment of the fibre during the manufacturing process has proved the most successful way of using these chemicals. In the A100 process developed by Tencel®, the AXIS chemical is applied to the washed tow in the fibre production line. It is then dried, crimped, cut and baled in the normal



4.12 Lyocell fibre scanning electron microscope (SEM) micrographs.

way. The A100 fibre has similar strength, elongation and modulus properties to the standard fibre and has an enhanced dye uptake that gives more economical dyestuff costs and strong deep colouration. Its resistance to fibrillation means it can be processed on most dyeing machines and the fabrics produced have a good performance in subsequent washing.

Tencel® A100 was launched in 1998 and has proved to be very popular and effective at extending the range of applications for Tencel® fibres. It is particularly beneficial for jersey knit applications because of its excellent colouration, attractive aesthetics and good wash/wear performance. The warm touch of jersey fabrics made from A100 complement the cooler touch of 'peach skin' fabrics made by the enzyme route.

4.5.5 Using fibrillation to create novel hands

The characteristic handle of the first commercially successful lyocell fabrics came from the presence of many fine microfibrils on the surface of the fabric. This gave the so called 'peach skin' aesthetic. Much of the development of dyeing/finishing technology was aimed at achieving this touch without suffering the loss of fabric appearance caused by uncontrolled fibrillation. Additionally there was the challenge of achieving this aesthetic in a commercially economic way.

When a lyocell fabric is first processed in the wet state the loose fibres on the fabric surface fibrillate and form easily seen pills. This is called primary fibrillation and is aesthetically unacceptable.

Primary fibrillation can be removed by enzymes or other treatments so that a fabric with a clean surface can be generated. If this fabric is then agitated in the wet state, short and fine fibrils are formed on the apex of the yarns in the fabric. This secondary fibrillation is only visible as a slight frost on the fabric surface and is aesthetically attractive. Secondary fibrillation also gives the fabric its characteristic 'peach skin' touch, which can be modified to the required aesthetic by the use of a variety of proprietary softeners, eg silicones.

The most attractive fabrics made by this route are given a tumbling process after dyeing. This both generates bulk and softness in the fabric and optimises the surface texture.

4.5.6 Processing routes to making 'peach touch' fabrics

It is important that the fabric produced to give a 'peach touch' is designed to give a good performance in washing. There are a variety of measures that need to be taken to achieve this. Yarn and fabric constructions should be sufficiently tight to prevent the formation of loose fibres that can fibrillate and pill in use. This is more difficult to achieve in knitted fabrics, but singeing of the fabric prior to the primary fibrillation step is very beneficial.

Pre-treatment of the fabric in caustic soda can be effective in improving both fabric performance and aesthetic. In caustic soda, lyocell swells very significantly in diameter but very little in length. The maximum effect is at 10 to 12% soda but stronger mercerisation concentrations can also be used. The swelling of the fibres will increase the diameter of the yarns in the fabric and this will cause the fabric to shrink. This step needs to be carried out with the fabric in open width form and if the fabric is allowed to shrink during caustic treatment its bulk will be significantly enhanced. Furthermore the fibres become set into this new configuration when the fabric is subsequently washed. The fabric then has greater bulk and flexibility – in particular the wet stiffness of the fabric is much reduced. The latter is important for further processing since the fabric is less prone to crease damage marks during the dyeing. The caustic treatment also gives more rapid fibrillation removal in processing and a reduced tendency to fibrillation in domestic use.

When the fabric is subjected to mechanical action in the wet state in preparation and dyeing the surface hairs receive the majority of the abrasive action, therefore fibrillation will occur predominantly on these surface fibres. The fibrils formed are relatively long and are able to become entangled leading to an extremely matted appearance. It is important that the fabric is 'worked' until the surface fibres are fibrillated to their maximum extent. Failure to achieve this will result in incomplete hair removal and an unstable surface appearance in the finished fabric.

A cellulase enzyme is used to clean the fibrillated hairs from the surface of the fabric. Fibrillation of the surface hairs reduces the fibre diameter and this in turn enhances their vulnerability to enzymatic attack. Thus complete removal of the surface hairs can be effected, with minimal effect on the bulk of the fabric. The hydrolytic fibre degradation involved in this process results in a loss of weight, typically 4%, and is accompanied by some reduction in fabric strength. Preliminary local trials are therefore strongly advised. The use of an enzyme on greige fabric is not effective in complete hair removal unless excessive chemical treatment is used which leads to unacceptable loss of fabric strength.

In order to generate a stable finished fabric appearance, it is necessary to fibrillate the fabric a second time. The characteristics of this 'secondary' fibrillation are, however, very different to the 'primary' fibrillation. With the absence of surface hairs, fibrillation is confined to the yarn cross-over points and the high points of the fabric construction. Furthermore, the positioning of these fibrils means that they are physically unable to entangle and therefore pilling does not occur.

Secondary fibrillation produces two effects. A small pile is created on the surface of the fabric which gives the special touch and feel characteristics known as 'Mill Wash' or 'Peach Skin'. The fibrils are only a fraction of the size (diameter) of the fibres, therefore they appear much lighter in colour, even though they contain the same amount of dye. It is this optical effect which gives the dusted mill-wash appearance. Secondary fibrillation can be produced by either a simple washing treatment or by jet-dyeing the fabric and it should be generated to a level that will not increase on subsequent washing.

Dyeing can be carried out either before or after the fibrillation process, but certain dyes can affect the primary fibrillation characteristics and enzyme efficiency. For this reason application of dyes after the fibrillation and enzyme treatment stages is preferred. Softener can be applied to the fabric either by an exhaust or a pad technique, but the best aesthetics are obtained when the fabric is finally tumbled. This treatment maximises fabric bulk and removes any loose fibres and lint from the fabric surface.

The fabric can be finished with a low level of an easy-care resin but care needs to be exercised in selecting the system to be used since some resins can weaken the fibrils so that they fall off during subsequent washing. If this occurs the fabric can lose its 'peach touch'. In order to maximise the peach skin aesthetic, resinated fabrics should be tumble dried before resin curing.

4.5.7 Machinery selection and process methodology

The choice of processing route and the type of equipment used have been of vital importance to the commercialisation of lyocell. These two factors are very much interrelated and this has given a great source of variety (and early frustration!) to the users of lyocell. Lyocell fibre has unusual swelling characteristics: on immersion in water the high degree of swelling causes a pronounced increase in fabric stiffness. This makes the fabric susceptible to creasing in rope processes. The apex of the crease can fibrillate during the mechanical agitation that occurs during most wet processing and this shows up as white lines on the fabric after it is dried. Since this is generally unacceptable, it can be readily appreciated that careful machine selection is important, particularly for heavyweight fabrics. As described above, treatment of the fabric in caustic soda can be very effective in reducing the tendency of the fabric to crease damage.

Peach skin processes rely on surface abrasion and the machinery must be capable of giving evenly distributed abrasion to the fabric surface. Machines vary in severity of physical action, those with a gentle action require more severe chemical and temperature conditions to achieve the desired result.

Garment processing gives an ideal combination of the physical conditions for processing lyocell so this route processed most of the early commercial fabrics. In particular, garment processing of indigo denim was an established industry for tumbling and enzyme treating cellulosic garments. Most machine types designed for garment washing have been found to be suitable.

The early success of lyocell fabrics made by the garment route generated a demand for fabrics to be made by the more economical piece processing route. Simple processing of lyocell fabrics by open width/resination routes gave fabrics with a good technical performance but they did not have the 'peach touch' or the bulk of fabrics made by the garment route. Continuous processes offer advantages in terms of capacity and consistency. Preparation, hair removal, dyeing and tumbling can all be carried out continuously, but the machines used were not sufficiently aggressive to generate fibrillation. More recently, open-width routes have been developed that can generate the required aesthetics by using caustic soda pre-treatments (to generate bulk and softness) and sueding after-treatment (to generate the surface touch). These routes will become increasingly important because of their lower inherent cost.

The first 'large scale' piece-processed fabrics were developed in Japan using Nidom machines: i.e. large scale barrel machines with a similar mechanical action to the garment machines. They generated very attractive fabrics that had both the bulk and 'peach' surface touch of fabrics made in the smaller garment machines. Fabrics made using these Nidom machines were commercially successful in the early days of Tencel® when the products commanded a very high price in the Japanese market. However their relatively low productivity and high labour costs restricted the extent of their adoption.

The most successful jet machines for piece processing woven fabrics are now ‘air-jets’ working on lengths of fabric in rope form. These rope-processing machines must be able to re-orient the fabric frequently to ensure uniform fibrillation across the surface and to prevent crease damage marks appearing. The air jet rope-processors most easily meet these requirements for woven fabrics but knitted fabrics are less prone to damage marks and a wider range of soft flow jets can be used successfully.

In order to achieve the best aesthetics, secondary fibrillation should be enhanced by a final tumbling process. This can be carried out dry, or with water present. Tumbling under wet conditions gives additional bulk to the fabric. Wet tumbling machines can also be utilised for the fibrillation and enzyme processes.

In woven fabrics, the high degree of swelling in water allows the ‘peach skin’ surface, once developed, to be stable to domestic laundering. When the fabric is wetted the fibres swell and are locked into position. Therefore it is very difficult for unfibrillated hairs to be drawn from the yarns within the fabric onto the surface.

In knitted fabrics, the structure is generally more open so that it is more difficult to lock the fibres into the body of the fabric. The fibres can therefore more easily be drawn onto the fabric surface and fibrillate. Good results can be achieved by combinations of the factors normally used to minimise pilling: high quality yarns, high yarn twist levels, tighter stitch length and the use of easy care resins.

Even though peach-skin fabrics in lyocell are inherently stable, their aesthetics and performance can be enhanced by the use of a fabric conditioner during domestic laundering and by tumble drying.

4.5.8 Economical processing routes

Following the success in developing all the principles described above, the next challenge has been to reduce the costs of carrying these out.

Most of the early focus was on achieving high first quality yields in the dye houses since there were high levels of rejects due to white line damage marks. Progress here was rapid and once the key process parameters were recognised and dye houses became experienced, good first quality yields were routinely achieved.

The next steps have been to reduce chemical and process-time costs. Enzyme treatment/fibrillation removal was initially very expensive because it took significant machine time and the enzymes were costly. Combining process stages has significantly reduced the process times. For instance the generation of secondary fibrillation can occur during the dyeing process when an air jet is used. The price of enzymes has declined as the market has grown in scale and competitiveness and the quantities required have been reduced.

Most recently, Tencel Ltd. has developed process routes that combine preparation stages and use oxidative systems instead of enzymes. These novel routes have greatly reduced the dyeing/finish costs of lyocell to the point where it can begin to be cost competitive with the processing on cotton fabrics.

4.6 Lyocell marketing

Following its launch, the development of Tencel® has focused on yarn development, fabric development and latterly, garment development. Garment development is now significant.

4.6.1 Yarn development

Short staple yarns include 100% Tencel® as well as a wide variety of blends with cotton, polyester, silk, cashmere, wool, viscose, linen, and elastane.

Long staple yarns include 100% Tencel® and blends with wool, silk and cashmere.

4.6.2 Fabric development

The first commercially available fabrics in Tencel® emerged with an emphasis on denim, chambrays and piece-dyes. Developments in this field anticipated the so-called denim revival which was given a fillip by the revolutionary Tencel® denim/chambray type – dubbed the 'cashmere of denim' by the press for its extraordinary properties of lustre, drape and softness.

A wider portfolio of fabric types includes velvets, corduroys, colour woven, gabardines, twills, jerseys, prints, voiles, lace, coated fabrics and crêpes, most of which were first shown at Premiere Vision (October 93) by the 27-strong 'partnership' of Tencel® weavers from France, Italy, Germany, Spain, Austria and Switzerland. The commitment of these weavers to the systematic development of individual fabric types, worked, and continues to work, to a unique 'windows of opportunity' matrix which individual customers with a reputation for excellence in a given product area were invited to fill.

The early focus on the premium-branded casual wear market was supplemented by fabric innovation for formal blouse and dresswear, classic shirting and suiting, lingerie and nightwear, rainwear and evening wear and in active sportswear.

4.6.3 Expansion of products and markets

The marketing of Tencel® has followed a carefully planned and controlled strategy. Considering how small the volume of Tencel® production is relative to the whole fibres market, and the investments made by Courtaulds in developing Tencel®, it was essential that Tencel® was initially positioned at the top end of the fibres markets. The properties of Tencel® enable the finished textiles to be of the highest quality thus giving the differentiation required for its market positioning. However, in order to realise the potential for making quality products from Tencel® the textile processing has to be of the highest quality. To ensure this is the case and that no inferior products reach the marketplace with the Tencel® name Tencel® was sold to technically innovative and competent textile manufacturers. As the production of Tencel® increased, it was necessary to move into the middle sector of the textile market and to develop the new dyeing/finishing routes described above, and the new fibres like Tencel® A100. These built on the unique physical characteristics of Tencel® to generate a wide range of attractive and novel fabric aesthetics at economical cost, enabling the textile trade to broaden its range of markets and to successfully meet the price points required by the retail customers.

The combination of comfort and performance of Tencel® fabrics is now enabling them to grow in both household textiles and workwear applications. Attractive towelling fabrics are being marketed that exploit the excellent water absorbency, deep colouration and wet stiffness of Tencel®. The high dry strength of Tencel® woven fabrics makes them desirable as backing for abrasives while in nonwovens its high wet strength is ideal in a wide range of absorbent wipes. The combination of easy fibrillation and wet strength makes short-cut fibre attractive in premium paper and filtration applications. With increased manufacturing capacity this area is important in maintaining a relatively steady sales output. Tencel Ltd. is also looking to exploit other applications in cellulosic films and continuous filament yarns. These are being pursued by joint developments with companies specialising in those fields. (See also carboxymethylated lyocell in Chapter 6).

The very positive environmental characteristics of the Tencel® process underpins the marketing strategy in all applications.