Review Article

Development and processing of lyocell

R B Chavan
Department of Textile Technology, Indian Institute of Technology, New Delhi 110 016, India

and

A K Patra
The Technological Institute of Textile & Sciences, Bhiwani 127 021, India

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An account of lyocell, covering the hindsight of its development and available brands has been reported. This wonder fibre surpasses all other cellulosic fibres in terms of properties, aesthetics and quite importantly, ecology in manufacturing. Among the various names with which lyocell is available, Tencel and Tencel A 100 are the prominent and widely used. Besides dealing with the various attributes of lyocell, the options for wet treatment of the fibre with reference to steps of processing, suitability of dyes and process parameters have also been addressed.

Keywords: Fibrillation, Lyocell, Peach-skin effect, Tencel, Tencel A 100

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1 Introduction

There has been a growing demand for absorbent fibres with the need hinging on comfort and fashion. Since cotton production can not go beyond a particular level due to limited land availability, the other obvious options are viscose and the likes. But again, with the increasing awareness of ecofriendly concepts, viscose is not quite highly rated because its manufacturing plants have inherent problem of effluent generation. Carbon disulphide, which is used in significant quantity in viscose manufacturing process, is a source of major environmental problem. Sulphur introduced in this process gets dispersed as sulphur compounds (CS₂, H₂S, COS, SO₂) in the exhaust gases, process baths, solid wastes and product itself. About 15% of the CS₂ used is converted into H₂S in the form of viscose byproduct and sodium trithiocarbonate. Recovery of all the sulphur used in the process is not possible and about 50% CS₂ reclaim is achieved.

Moreover, zinc sulphate, a viscose spin-bath component, is often made at the rayon plant by dissolving metallic zinc in sulphuric acid. This chemical is toxic to the aquatic life. Overall process effluents from viscose plant have high BOD, COD and total and dissolved solids. The viscose rayon manufacturing process is also energy-intensive. Besides this, viscose rayon production has high labour demand, mainly due to the complexity and number of steps involved in converting pulp into rayon fibre.

Among the modified viscose fibres, high wet modulus (HWM) rayon involves relatively simple and economical manufacturing process, but the zinc used in this process is a known pollutant. On the contrary, polynosic fibre does not need zinc but requires formaldehyde for its manufacturing, which again is not ecofriendly. The quantities of some chemicals used in making rayon, HWM and polynosic are shown in Table 1. Utilization of 2,3-hydroxyls in cellulose monomer for complex formation with transition metals was another dimension of development in viscose and cuprammonium rayon manufacturing process. But the toxicity of metals like copper is not quite acceptable.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Regular</th>
<th>HWM</th>
<th>Polynosic</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH, kg/kg</td>
<td>0.64</td>
<td>0.9</td>
<td>0.7</td>
</tr>
<tr>
<td>CS₂, kg/kg</td>
<td>0.296</td>
<td>0.35</td>
<td>0.5</td>
</tr>
<tr>
<td>H₂SO₄, kg/kg</td>
<td>0.95</td>
<td>1.37</td>
<td>1.1</td>
</tr>
<tr>
<td>ZnO, kg/kg</td>
<td>0.004</td>
<td>0.016</td>
<td>0.0001</td>
</tr>
</tbody>
</table>

*To whom all the correspondence should be addressed.
Phone: +91-9812094405; E-mail: arunkpatra@rediffmail.com
As regards various alternative methods proposed for manufacturing viscose, ZnCl₂/water, NaOH/water, dimethyl acetamide/lithium chloride and carbamate are prominent. In ZnCl₂/water, the problem is again with the removal of residual zinc. Moreover, the spinning of fine deniers could not be established. Similarly, in NaOH/water process, strength of the fibres produced was low. In DMAC/LiCl process, the costs involved in pre-activation of cellulose and recovery of solvents proved to be the major bottlenecks. An alternative carbamate process has the problem of high energy cost associated with low temperature requirement for dissolution.

Thus, a good man-made water-absorbent fibre with convenient and ecofriendly manufacturing process was long overdue. After years of research, lyocell fibre was developed and it was claimed to be the first new fibre in 30 years. It provides the comfort of natural fibre coupled with excellent aesthetics. Lyocell is the generic name for a regenerated cellulosic fibre obtained by spinning of dissolved wood pulp in an organic solvent. The solvent spinning technique so adopted is an environmentally responsible process. The fabric made out of this fibre gets very good value addition when processed with requisite care.

1.1 Background

The name lyocell, given in 1989 for solvent-spun fibres, owes its genesis to the Greek word lyein (meaning dissolve) from which comes lye and to cell from cellulose. This name was recognized as the generic name by BISFA (International Bureau for the Standardization of Rayon and Synthetic Fibres, Brussels) and the Federal Trade Commission (USA). This fibre which took 16 years for its development with investment of US $ 500 million is now being produced by different fibre manufacturers under different names.

Prominent among the registered brand names of lyocell are Tencel (Acordis), Lenzing Lyocell (Lenzing) and Newcell (Akzo Nobel). Newcell is a filament while the other two are staple fibres. The two Tencel manufacturing units of Acordis (previously Courtaulds) — one in Mobile, Alabama (USA) and another relatively new one in Grimsby (UK) — have a production capacity of 40,000 tons/year each. Lenzing AG has its lyocell fibre produced by Lenzing Lyocell GmbH in Heiligenkreuz, Austria. It started the manufacturing process after taking a license for the basic patents from Akzo. Akzo Nobel, manufacturer of Newcell (the filament lyocell), is a Dutch multinational and has in fact purchased Courtaulds. The fibre division of both comes under Acordis. Besides these, there is a lyocell plant of Alceru Schwarz GmbH at Rudolstadt, Germany. The company is a joint venture between the engineering company Lurgi Zimmer AG in Frankfurt and the Thuringian Institute for Textile and Plastic Research (TITK) in Rudolstadt. Alceru in fact produces the fibre with brand name ‘Seacell’ using lyocell technology in which seaweed is incorporated. Among the Asian fibre manufacturers, the acrylic fibre producer Hanil Synthetic Fibre Co. in Seoul, South Korea, sells lyocell fibres with the brand name ‘Cocel’. In Taiwan, the viscose fibre producer, Formosa Chemicals & Fibre Corporation (Taipei) started production of lyocell staple fibres but is no more in the business now. In India, the Birla Group, a major viscose fibre producer, had initially shown some interest for developing lyocell fibre, but didn’t pursue it further.

1.1.1 Tencel

Within the lyocell category, Acordis brought out the first commercial product in 1988 and brand named it as ‘Tencel’. The fibre had an initial commercial success in Japan, mainly in indigo denim and specialty niche products. Now, it is of course found in volume markets. The fabric manufacturers have to register with Acordis while using its lyocell fibre. The company gets into partnership with fabric manufacturers using Tencel. The fabric maker buys lyocell fibre from the company and gets technical support from Acordis for developing the requisite fabric. The fabric after being made is sent to Acordis for testing and if it passes the various physical tests, the fabric is branded as Tencel. The fabric maker has to get separate approval for each of the different fabric qualities it makes, if he wishes to label them as Tencel. Same also holds good for processors and the Tencel Quality Number is given to each quality of woven or knitted fabric. Clothings made with Tencel fabrics carry trade mark swing tags and sewn-in-labels issued by Courtaulds (Acordis) as proof of authenticity and quality.

Otherwise, the fabric is called lyocell only. Perhaps that is why it is said that lyocell fibre from Acordis is termed ‘Tencel’ when used for apparel applications and called ‘lyocell’ when used for technical or industrial applications. The trade mark of Tencel is in the form of triple “X”. This is also applicable for blended fabrics, but the blend should have at least 40% Tencel.
Tencel is available in two fibre counts of 1.4 and 1.7 dtex as standard qualities. The staple fibre of 1.1, 2.4 and 3.3 dtex are also available for special applications. Most of the Tencel fibres are bright, but a matt fibre type is also available.

2 Fibre Manufacturing

The starting material for lyocell and viscose are the same, i.e. wood pulp, but the manufacturing processes are different. No cellulose derivative is formed in the former, while viscose rayon manufacturing involves formation of intermediate derivative. Lyocell is manufactured by a direct dissolving process using an organic cyclic polar solvent, namely N-methyl morpholine-N-oxide (NMMO). This solvent is non-toxic and is easily regenerated. NMMO has higher cellulose dissolving capacity than the other organic polar solvents, like DMSO, DMF, DMAC, NDMA, HMPA, etc. The NMMO solution used is a 50:50 (w/w) mixture of solvent and water. The trade name of this solvent is AM and the common name is amine oxide. The melting point of the monohydrate solvent is about 76°C. NMMO can be produced from N-methyl morpholine and hydrogen peroxide as per the following reaction:

\[ \text{H}_2\text{O}_2 \xrightarrow{\text{H}_2\text{O}} \text{O(C}_4\text{H}_6\text{H}_3\text{NCH}_3 \rightarrow \text{O(C}_4\text{H}_6\text{H}_3\text{NOCH}_3 + \text{H}_2\text{O}} \]

Wood pulp is dispersed in concentrated aqueous NMMO and dissolved under exertion of intensive shear forces and simultaneous evaporation of water. The pulp used is an industrial dissolving pulp, having a DP of 750, with 96% cellulose. The starting point of the process is a suspension of approximately 13% cellulose, 20% water and 67% NMMO. Dissolution of cellulose in NMMO is done at 120°C (temperature more than 125-130°C being unsafe for NMMO), resulting in a highly viscous solution. The solution is filtered and then extruded into a water bath through fine jets. As the solvent is washed out, the fibres formed into fine filaments are collected as tow, from which the staple fibre is produced. The surplus water is evaporated off and the remaining concentrated NMMO is recycled into the process. The process cycle, as shown in Fig. 1, is characterized by its closed loop.

2.1 Toxicological Aspect

More than 99% of the solvent is recycled within the process, making the fibre production extremely ecofriendly. Very small quantities of NMMO emitted via waste water are readily degraded in the biological waste water treatment plants. NMMO with its favourable toxicological properties has been found to be less toxic than ethanol. Amine oxides are generally used extensively as active substances of personal hygiene products which normally undergo intensive toxicological examination. Therefore, non-toxicity of the solvent can be assumed for all practical purposes. As per the various scientific tests, it is also not mutagenic. Tencel (the fibre from Acordis), which involves the use of this non-toxic solvent for its manufacturing, is certified for the use of Oeko-Tex, conformance in Textiles mark.

The quantity of chemicals used in the process of manufacturing lyocell does not pose environmental concerns. Waste water when purified by a biological waste water treatment plant contains only small amounts of organic chemicals and salt, mainly sodium sulphate. Thus, the COD per kg of fibre produced comes only 11g and the salt load is found to be 230 g. Emissions into air are of similarly small magnitude.

3 Special Brands

Besides the conventional lyocell qualities, some interesting modifications have also come up with improved properties. These fibres branded with different names are discussed below.
3.1 Tencel A 100

The development of Tencel A 100 is based on the commercial experience\textsuperscript{20-23}. This is a welcome change over the conventional Tencel fibre and has been targeted particularly for the knitwear sector. Normal Tencel has a tendency to fibrillate and is often given chemical cross-linking treatment as a part of wet processing by the dyer. A colourless triazine structured crosslinking chemical (code name Axis) was first supplied by Courtaulds in 1998 for this purpose. The compound has the ability to crosslink the amorphous section of fibres and prevent fibrillation. Axis can be applied to fabric prior to dyeing, during dyeing or in the finishing stage. The resulting fabrics are attractive and have a definite market appeal.

The application process was, however, inconvenient as Axis had lack of substantivity for the fibre, particularly in long liquor ratios, making it expensive to apply and this increases the cycle time. Moreover, the control of process in terms of reproducibility and distribution of Axis chemical contributed to the need for a more appropriate means of producing the unique non-fibrillating characteristics. This has now been achieved by chemically crosslinking the fibre, while it is in tow form after extrusion and curing before being crimped and cut. The process is more economical, controllable and avoids any extra processing step by the dyer. The only place where Tencel A100 is being manufactured is in the Grimsby, UK plant of Acordis and it is available in bright form.

Tencel A 100, normally available in 1.25 and 1.4 dtex, has slightly different fibre properties than that of Tencel. It is about 10% lower in both tenacity and modulus as compared to normal Tencel and has more open structure to give a high water imbibition. Its water retention is about 75% as compared to 65% for normal Tencel, which results in better dyeability. Tencel A 100 also has higher degree of whiteness than Tencel. Quite importantly, because of the non-fibrillating nature of Tencel A 100, there is no need of bio-polishing and hence the process route becomes much simpler. Moreover, it is claimed to have very good suitability for blending with wool because of its compatibility with wool processing.

3.2 Tencel A 200

This is the newest non-fibrillating lyocell fibre currently in the test marketing phase. It differs from Tencel A 100 in that it is totally stable in alkaline solutions and hence can withstand mercerizing treatment, which is important in cotton blends. Its dye affinity is claimed to be similar to that of cotton, thus making it easier to attain solid shades in blends. Finally, Tencel A 200 doesn’t have the potential to release very small amounts of formaldehyde that can be found in Tencel A 100, making the former a more appropriate product for intimate apparel and infant clothing.

3.3 Tencel HS 260

This is the latest variant of the lyocell fibre developed specifically for high speed nonwovens processing. This fibre, which gives efficient high-speed carding performance (250 m/min), has been proven on full-scale commercial carding systems. Tencel HS 260 can be processed via a diverse range of nonwoven manufacturing technologies including spun-lace, needle bonding, latex bonding and in blends by thermal bonding. It has a high crimp level and better crimp retention than standard Tencel\textsuperscript{24}.

3.4 Seacell

Among the other developments in lyocell, Seacell by Zimmer AG is an interesting fibre with seaweed as renewable resource. Since the active ingredients of this fibre originate from sea and cellulose serves as the ‘carrier’ for these substances, it is named seacell. The minerals contained in the seawater accumulate in the seaweed and the seacell fibre made from it has health-promoting effect on the wearer. The interaction between seaweed and skin is in fact anti-inflammatory, anti-viral and anti-bacterial\textsuperscript{15}.

3.5 Hydrocell

Hydrocell\textsuperscript{17-26,27} was introduced by Coutaulds, UK, based on its lyocell technology. This fibre is a very good alternative to calcium alginate used in the advanced wound dressing treatment of chronic wounds, including pressure sores, leg ulcers and burns. Hydrocell is carboxymethyl cellulose product chemically converted from Courtaulds’ lyocell which offers the same advantages as alginate, for example it gels on contact with wound fluid to provide a non-adherent and moist environment. Hydrocell is said to be even more absorbent than alginate (up to 35 times of its own weight) and forms a more coherent gel, thus allowing one-piece removal, reducing nursing time and avoiding any trauma to new tissue growth.

The fibre has, therefore, a potential market in wound care and Cova Tec, a subsidiary of the pharmaceutical firm Bristol Myers Squibb, has taken up the product for worldwide exploitation. Hydrocell is branded as ‘Aquacel’ at Cova Tec’s UK plant.
4 Characteristics, Advantages and Applications of Lyocell

4.1 Physical and Chemical Properties

Some of the physical properties of Tencel are shown in Table 2. In fact, only Tencel brand of lyocell is available in the market in significant quantity and hence the two names are often used as synonymous terms. Cellulose in Tencel fibre has a high degree of orientation and the cellulose crystals are highly parallel in longitudinal direction of fibre. The ratio of crystalline and amorphous area is approximately around 9:1. Tencel fibre has a smooth uniform surface and a circular cross-section. Its structure is not a collapsed structure like that of cotton21.

As regards thermal properties, lyocell is stable below 150°C and the fibre begins to lose strength gradually above 170°C. It begins to decompose more rapidly at 300°C and finally gets ignited at 420°C. Regarding chemical nature, lyocell is inert to most of the organic solvents. However, it degrades in the presence of hot dilute or cold concentrated mineral acid. Alkalies cause swelling of the fibre at first (maximum with 9% NaOH at 25°C) and then ultimately disintegration28-30. When wetted out in water, the lyocell fibres swell laterally and the increase is about 35% over the dry fibre diameter31.

4.2 Fibrillation

It is one of the important properties of lyocell. Fibrillation is the longitudinal splitting of a single wet fibre into microfibres of < 1-4 μm in diameter, caused by mechanical stress5. Fibrillation normally takes place under stress along the long axis in wet condition. The stress is usually due to the wet abrasion against fabric or metal. The microfine surface hairs (or microfibres) generated are so fine that they look almost transparent giving a white or frothy appearance to the fabric. In practice, this problem is encountered when the fabric is processed in rope form in the drum (garment treatment) or in domestic washing. During such treatments, the fibre splits and individual fibrils protrude from the surface of fabric (Fig. 2). The change is both visible and affects the handle. As the fibrils become almost transparent due to their fineness, particularly fibres of medium and dark shades appear lighter and the surface looks grayer.

The unique highly crystalline structure of lyocell fibres is the basis of fibrillation. The crystalline fibres possess a fibrillar structure in which microfibrils are arranged parallel to the fibre axis. They have a tendency to fibrillate because of high orientation and

<table>
<thead>
<tr>
<th>Property</th>
<th>Tencel</th>
<th>Cotton</th>
<th>Viscose</th>
<th>Modal</th>
<th>Polyester</th>
</tr>
</thead>
<tbody>
<tr>
<td>Count, dtex</td>
<td>1.4-1.7</td>
<td>1.5-1.8</td>
<td>1.7</td>
<td>1.7</td>
<td>1.7</td>
</tr>
<tr>
<td>Tensile strength, cN/tex</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dry</td>
<td>38-42</td>
<td>20-34</td>
<td>22-26</td>
<td>34-36</td>
<td>55-60</td>
</tr>
<tr>
<td>Wet</td>
<td>34-38</td>
<td>25-30</td>
<td>19-24</td>
<td>54-58</td>
<td></td>
</tr>
<tr>
<td>Elongation, %</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dry</td>
<td>14-16</td>
<td>7-11</td>
<td>17-25</td>
<td>13-15</td>
<td>25-30</td>
</tr>
<tr>
<td>Wet</td>
<td>16-18</td>
<td>11-14</td>
<td>21-30</td>
<td>13-15</td>
<td>25-30</td>
</tr>
<tr>
<td>Moisture uptake, %</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water-retention capacity, %</td>
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<td>45-55</td>
<td>90-100</td>
<td>75-80</td>
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<td>Initial wet modulus, %</td>
<td>250-270</td>
<td>100-200</td>
<td>40-60</td>
<td>100-120</td>
<td>210</td>
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<tr>
<td>Loop strength, cN/tex</td>
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<td>20-26</td>
<td>6</td>
<td>8</td>
<td>11</td>
</tr>
<tr>
<td>DP value (Staudinger)</td>
<td>500-600</td>
<td>2300-3000</td>
<td>250-350</td>
<td>300-600</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 2 — Fibrillated lyocell fibre
lack of lateral cohesion. When wet, swelling of the porous regions of the fibre breaks the hydrogen bond linking the crystalline units and forces them apart. When this structure is subjected to mechanical action, the outer crystalline region can break and peel away from the main fibre but remains attached like banana peel. These peelings are referred to as fibrils5-9,30.

If fibrillation is not controlled, these microfibres become entangled, giving serious problems of 'pilled' appearance. It also weakens the mother fibre. The fibrillation effect can be advantageous for creating fabrics with an attractive appearance and appealing hand called 'peach-skin effect', but for some other applications, it is desirable to eliminate the fibrils30.

There are two forms of fibrillation – primary and secondary. The first one consists of long and irregular fibrils which can get entangled, leading to an extremely matted appearance. Defibrillation through cellulase enzyme treatment is used to remove this unwanted form. The secondary form, produced deliberately, is responsible for the fabric’s attributes32. These fibrils are short and even, and can not cause pilling. Secondary fibrillation produces change in hand as well as appearance.

Other than the mechanical effect, the factors that increase fibrillation are low yarn twist, open structure, high temperature, alkaline pH, low liquor ratio, etc. On the other hand, the factors that decrease fibrillation include reduced mechanical action, use of crease mark reducing agents, singeing before or after dyeing, cellulase enzymatic treatment and finishing with resins31.

4.3 Advantages

The advantage of lyocell starts right from its manufacturing stage. Compared to the commonly used viscose fibre, lyocell fibre involves less steps and chemicals for its manufacturing. Solvent and water can be recycled in the lyocell process. As against viscose, the lyocell process also starts with pulp, the remaining manufacturing process as discussed is completely new. The environment-friendly production process and the use of renewable raw materials and their biodegradability rate the lyocell above many other regenerated fibres36. Quite importantly, the production method is potentially more cost effective and faster than that used to make viscose rayon. The lyocell process takes three hours to produce fibre compared to 40 hours needed to make viscose rayon staple31. Moreover, the energy and water requirements for manufacturing of lyocell fibre are less19.

Lyocell has the unique characteristics of soft and silky handle, lustre and bulky touch. Fabrics made out of this fibre show very good drape and fluidity that is unexpected for the fabrics of their weight8,27. A rich look stands out as the hallmark of its aesthetics. Interestingly, lyocell has high dry tenacity and modulus. It is the strongest cellulosic fibre when dry, even stronger than cotton or linen. It also retains much of its strength when wet. Its wet tenacity is higher than that of cotton and other cellulosic fibres14,36. Compared to viscose, it is two times stronger when dry and three times when wet. The reason for this is the average degree of polymerization and the number of crystalline zones are greater in lyocell compared to that in conventional viscose rayon, HWM modal or polynosic fibres.

4.3 Speciality and Applications

The important attributes of lyocell are: 100% natural in origin, better dyeability than other cellulosics, softness and drape, luxurious handle, lighter and finer fabrics, fluidity and movement, breathability, lustre, high strength both in dry and wet state, good moisture retention and hence wearing comfort, and good dimensional stability. Added to these, it has also moderate resiliency and hence doesn’t crease as badly as cotton and rayon.

Notably, Tencel fibre blends well with various natural and synthetic fibres, like cotton, linen, rayon, polyester, lycra, nylon, silk and wool. The stress-strain characteristics of Tencel make it an ideal partner with the various textile fibres. The blends give good performance and quality with luxurious appearance. On blending with wool, Tencel introduces new fluidity and drape while on blending with cotton and linen, it increases suppleness and lustre4,14. Also, high tenacity of the fibre enables blended yarns to be produced in finer counts8.

As regards applications of the fibre, the range includes sportswear, casual wear (denim, chino, chambray, etc), fashionable ladies garments, mens shirts and luxurious peach skin finished jersey and knitwear. Besides apparels, lyocell can also be used for home products, including bath towels, sheets, pillowcases, etc. It also has industrial applications in making wipes, medical swabs and gauzes, filters, bicomposites, battery separators, etc37,38.

5 Wet Processing of Lyocell

5.1 Pretreatments

The processing of a greige lyocell fabric, like other
cellulosic fabrics, also begins with the cleaning process. However, the impurities in lyocell are far too less than that in cotton. The impurities present in it are predominantly those introduced during fabric making. Hence, for woven fabrics, it is mainly size while for knits, yarn lubricants and knitting oil comprise the impurities. In 100% lyocell fabrics of woven qualities, PVA or a mixture of PVA and polyacrylates are used as sizing material. Hence, the removal of these water-soluble added impurities does not require much of chemicals. But woven fabrics of lyocell/cotton blend are often sized with PVA, polyacrylates and starch. In such cases, desizing is done with enzymes to ensure removal of starch. However, lyocell/cotton knits don't require any desizing treatment for obvious reasons. The starch containing lyocell blends are desized with amylase in a process similar to any of the conventional techniques.

Prior to this, singeing of the fabric may be done which of course is optional. This process, normally carried out in gas singeing machine, has an important contribution in removing the hair from the fabric surface.

As regards scouring and bleaching, lyocell doesn't require any rigorous scouring. It can however be bleached, if required. A single-stage scouring and peroxide bleaching can be conveniently done. In case of Tencel A 100, the non-fibrillating brand, the effect of increasing levels of sodium carbonate and sodium hydroxide has been assessed. Scouring with soda ash at levels up to 20 gpl at 80°C and 95°C causes no loss in anti-fibrillation performance of Tencel A 100. However, if 10 gpl and 5 gpl caustic soda is used respectively at 80°C and 95°C, the fibrillation occurs on subsequent washing and tumbling. Hence, the conditions less severe than this should be used to ensure protection of the anti-fibrillation property of Tencel A 100. Regarding bleaching, although Tencel A 100 is slightly whiter than normal Tencel, it may still be necessary to bleach for pastel shades and optical whites. The recommendation for its bleaching is to use 7.5 ml/litre hydrogen peroxide (35%) at 85°C with 0.5 gpl sodium hydroxide or 1 gpl NaOH with 5 ml/litre hydrogen peroxide at 80°C. For the non-fibrillating lyocell brand, 4 gpl caustic and 25 gpl peroxide are recommended for pad-batch application while 3 gpl caustic and 15 gpl peroxide are suggested for pad-steam bleaching.

Besides these petreatments, causticization of lyocell can also be done to improve its dye absorption capacity. Lateral swelling of fibre in NaOH solution is greater than that in water with diameter increasing by over 200% at optimum concentration. The lateral expansion in the fibre leads to fabric shrinkage, i.e. reduced planar dimension and increased fibre thickness. Lyocell fibre can be causticized in long liquor. Studies on the effect of mercerization on dyeing and physical properties of tencel have also given reasonably encouraging results.

5.2 Pre-fibrillation

All processes that abrade the lyocell fibre in wet condition generate some fibrillation. Before the surface is cleaned (defibrillated) from all fibrills, it is essential to initially fibrillate the fabric to maximum extent. This fibrillation process is known as pre-fibrillation or primary fibrillation. For a 100% lyocell fabric, this is carried out by running the material in a jet dyeing machine at 80-120°C for 60-90 min using a solution of 5 gpl soda ash and 2-4 gpl lubricant. Lubricant used here in fact decreases the fibrillation, but is recommended in order to minimize creasing. The chemical treatment should be followed by draining and rinsing.

5.3 Dyeing

Lyocell/Tencel is a cellulosic fibre and therefore can be dyed with any class of dyestuff suitable for other cellulosics. In fact, it is observed that the dye yield on tencel is found to be greater than that on cotton, modal and viscose. Investigations carried out by Ciba, Clariant, Dyestar and Sumitomo on dyeing of Tencel conform to this fact for reactive dyes of different types and even for direct dyes. It is also suggested that with appropriate dyes, a one-bath simultaneous scouring-dyeing process is also possible as there are few contaminants in Tencel to interfere with the dyeing process. Since Tencel is manufactured from an amine oxide solvent spinning process, there is no risk of sulphur contamination as in case of conventional viscose fibres spinning process. Sulphur contamination is well known for possible dye reduction effects.

It is now an established fact that the reactive dyes have higher natural affinity for lyocell than for cotton. Hence, reactive dyes with good migration properties will be best suited for lyocell/Tencel, providing level dyeing effect. Thus, the electrolyte recommendations can also be significantly reduced, as compared to the dyeing to the same depth of shade on cotton. Consequently, there is reduction in chemical cost, handling...
and total dissolved solids. Lower electrolyte also facilitates quicker removal of unfixed hydrolysed dye from the fibre in washing stage after dyeing, thus reducing the process cycle time and hence the cost. Reactive dyes are in fact seen as the major dye class used on Tencel today.

Procion H-EXL dyes of Dyestar (previously of BASF, Zeneca) are often recommended for dyeing of Tencel. These are dyes with high fixation percentage (85%), and the specific dyeing process is suggested for Tencel which has to be diligently followed to get good results. The process has also been endorsed by Acordis for dyeing its Tencel fibre. The detailed process is shown in Fig. 3. For dyeing with Procion H-EXL dyes by exhaust method, the dye bath is first set at 50°C with electrolyte, auxiliaries like sequestrant, lubricant (in case of knits), etc. Starting pH should be 5.5-6.5. Then dye is added over 20 min and temperature is raised to 95°C. After continuing exhaustion at that temperature for 30 min, the bath temperature is brought down to 80°C. After 10 min, the alkali (soda ash) is dosed over 30 min. This addition should be progressive and follow Curve C3 (30% progression) or Curve C5 (50% progression). After that, the process may be continued for 45-60 min at the same fixation temperature of 80°C. Subsequently, the washing off should be done (Fig 4). In the wash-off cycle, soaping with Dekol SN is recommended to accelerate the removal of unfixed and hydrolysed dye from the fabric.

With a view to reduce production cost, BASF has also come up with a novel range of deactivated polyfunctional reactive dyes—the Procion XL+ dye range. These dyes have high tinctorial strength due to incorporation of at least two chromophores and two reactive groups into the molecule. This means that less dye will be required to get a given depth. This further reduces the electrolyte requirement, which is 35% less than that used for cotton and therefore shortens the process cycle, saving water and energy. These dyes also offer good robustness to process variation, particularly the material-to-liquor ratio. As compared to normal Tencel, high colour yields are obtained on Tencel A 100. Colour fastness on Tencel A 100 is at least equal to those obtained on other fibres, and in many cases it is superior.

Among other reactive dyes, Sumifix HF range of dyes from Sumitomo Chemicals Ltd is claimed to suit exhaust dyeing of Tencel. The manufacturer says that these high fixation modified vinyl sulphone dyes are based on 'high fidelity' concept. High fidelity mainly refers to low sensitivity to process variables, thereby giving good batch-to-batch reproducibility, high wet fastness without post-fixation treatment and high fixation with low amount of salt, leading to easy wash-off.

Moreover, cold pad-batch dyeing of lyocell has also been suggested. Using suitable Levafix and Remazol dyes, the fabric can be dyed in open width, getting an advantage in terms of low batch-to-batch variation, low energy and water requirement, no fibrillation during the process and never-the-less reduced crease marks. In fact, in exhaust dyeing in rope form, crease marks are often a serious problem despite using a crease inhibitor. However, pad-batch dyeing of lyocell has some disadvantages also. The required secondary fibrillation for peach-skin effect is not possible in this. Besides this, correcting faulty shades is relatively difficult in this case and sometimes a moiré effect may be observed on lyocell fibres.

The possibilities of dyeing lyocell with sulphur dyes have also been explored although not commercially done. Dyeing trials have been taken by some vat dye manufacturers.

Specific multifunctional reactive dyes are reported to have favourable effect on fibrillation behaviour of...
lyocell fibre. A study has shown that the crosslinking of reactive groups of these dyes with adjacent cellulose chains provides an opportunity to reduce fibrillation during wet processing.\textsuperscript{45} Extensive tests have shown that certain reactive dyes, which have at least two reactive centers, can form a covalent bond with two adjacent cellulose molecules\textsuperscript{46}. It is also believed that the presence of several reactive groups is not alone sufficient to produce this effect, but very specific molecular constitution and properties are also required to get the said effect. Those features of the dye include stearic orientation of the reactive groups, degree of reactivity, diffusion properties, number and size of chromophores and flexibility of dye molecule (bridge).

Cibacron LS dyes, the low-salt dyes from Ciba, are claimed to largely fulfill such conditions in exhaust application and thus induce crosslinking. Claims have also been made for Cibacron C dyes to promote such effect in pad application, especially cold pad-batch. Although diffusion is considered to be an important criterion to uniform crosslinking, Solophenyl dyes (direct dyes by Ciba) which have a high standard of penetration, do not exhibit any crosslinking property\textsuperscript{45,46}.

Among the blends of lyocell, the blend with cotton is relatively common. Both the components can be dyed with the same dye in one bath and in one step. But, usually lyocell dyes deeper than cotton due to its high dye uptake. Hence, the dyed fabric gets an uneven dyeing effect and appears like a mélange fabric. Although this uneven dyeing may be considered as a sort of fashion, but getting an even shade on such blends is still a problem waiting for solution. One of the possible ways could be a preferential treatment of cotton in fibre stage, so that the dye uptake of both the cellulosics can be brought to par. But this is only an idea, which has to be worked out.

As regards other blends, Schäfer\textsuperscript{47} has worked on dyeing of lyocell/wool blends with direct dye/acid dye and direct dye/2:1 metal complex dye combinations. Satisfactory results in terms of levelness, shade depth, tone-in-tone effects and wet fastness have been reported. The blend was dyed optimally using a one-bath procedure.

5.4 Defibrillation

Defibrillation usually precedes dyeing when a peach-skin effect is desired. However, for a totally clean surface, defibrillation is done after the dyeing stage. The primary fibrillation, often referred as pre-fibrillation or meta-fibrillation, is understandably inevitable in normal fibrillating type of lyocell fibres. Removal of the fibrils so generated during this process is absolutely imperative and this is done by using a cellulase enzyme. Defibrillation is often done in the same machine in which pre-fibrillation occurs. For cleaning the fibrillated hair from the fabric surface, acid type of cellulase is preferred to neutral ones\textsuperscript{48}. A pH of 4.5-5.0 and a temperature of 55°C are maintained during this enzyme treatment. After the enzyme wash, the enzyme is denatured by increasing the temperature and pH before rinsing the fabric. The enzyme degrades the protruding fibrils along the surface of the fibre, softens the fabric, removes fluff and finally gives an even effect. The mild hydrophilic fibre degradation involved in the process results in loss in weight of the fabric, typically around 4%.

Later on, the desired secondary fibrillation takes place during dyeing (if dyeing is done later), which is quite different from the primary fibrillation, giving the special touch and feel characteristics known as ‘mill-wash’ or ‘soft-touch’ or ‘peach-skin’ effect.

5.5 Finishing

In general, finishes which are suitable for use on other cellulosic fibres are also applicable to Tencel. For example, softener finish can be applied on Tencel fabric either by padding or by exhaust technique. But the amount of finish required to give the desired hand may be lower for Tencel than that for cotton due to the natural softness of the regenerated fibre. Lower amounts of soft finishes are also required on Tencel blends with synthetic fibres, such as polyester and nylon. Some of the softening systems suitable for the fibre include silicone emulsions, fatty acid macromulsions, polyethylenes, polyurethanes, silicone elastomers and acrylic copolymers.

In fibrillating type of lyocell, if clean surface is required instead of peach-skin effect, the secondary fibrillation step should be left out and the surface appearance should be stabilized with resin finish. The crosslinking agents used for this finish control fibrillation. A suitable low formaldehyde resin may be used for this purpose. Use of ultra-low formaldehyde resin, like Fixapret ECO of BASF, is recommended and practiced. This modified DMDHEU resin also fulfils the requirements of Öko-Tex Standard 100 for free formaldehyde and gives formaldehyde values of less than 75 ppm without afterwash\textsuperscript{49}. It has been found by practical application that the easy care properties of Tencel in terms of both dry and wet crease
recovery angles improve substantially on resination. The residual shrinkage is much less in this case compared to that in the case of other cellulosics like modal and viscose with similar finish application.

Fluorocarbon finishes wherever required may be added to impart oil and stain repellencies to the fabric.

6 Conclusion

Lyocell has come up as a fibre for the future and has many advantages over other cellulosic fibres in respect of fibre properties as well as from fashion and aesthetics points of view. The fibre is, however, quite expensive and an economical way of producing it perhaps holds the key to further popularize it. However, blending it with other fibres brings down the fabric cost significantly, maintaining the required properties. Most of the initial bottlenecks in wet processing of the lyocell fabrics have also been overcome by continuous process developments.

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