# **TEXTILE FINISHING**

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# **Chapter 1 Introduction to Finishing**

#### 1.1 Introduction

The operation that is performed for improving the appearance or usefulness of a fabric after it leaves the loom or knitting machine can be considered a finishing step. Finishing is the last step in fabric manufacturing and is done to impart the final fabric properties that are developed intentionally and purposefully.

The term 'finishing', in its widest sense, covers all processes which fabrics undergo after their manufacture in looms or knitted machines. However, in a more restricted sense, it is the third and final stage of processing after bleaching and dyeing. Even this definition does not hold well in some cases where the fabric is not bleached and/ or dyed. A simple definition of finishing is the sequence of operations, other than scouring, bleaching and coloration, to which the fabrics are subjected after leaving the loom or knitting machine. Most finishes are applied to woven, nonwoven and knit fabrics. But finishing is also done in yarn form (e.g., silicone finishing on sewing yarn) or garment form. Finishing is mostly done in fabric form rather than in yarn form. However, sewing threads made from mercerized cotton, linen and their blends with synthetic fibres as well as some silk yarns require finishing in yarn form.

A fabric's finish can be either chemicals that change the fabric's aesthetic and/or physical properties or changes in texture or surface characteristics brought about by physically manipulating the fabric with mechanical devices; it can also be a combination of the two.

Textile finishing gives a textile its final technical character with regard to appearance, shine, handle, drape, fullness, usability, etc. Nearly all textiles are finished. When finishing takes place in a wet state, it is called wet finishing, and while finishing in a dry state, it is called dry finishing. The finishing auxiliaries are applied using finishing machines, padders or mangles with one- or two-sided action or by impregnation or exhaustion. Altering the composition, rheology and viscosity of the finish applied can vary the effects obtained by finishing.

The operations carried out in a textile finishing mill include:

- (1) Preparation, in which the goods are scoured and bleached in readiness for finishing;
- (2) Dyeing or printing;
- (3) Mechanical finishing procedures, usually performed on dry material;
- (4) Thermal processes including drying and heat setting;

(5) Wet finishing processes in which the goods are treated with solutions of appropriate chemicals and subsequently dried.

Textile finishing involves treating a textile material in such a way that the product has the desired aesthetic and functional properties required for its intended use and therefore has greater market value. The desired properties may include the fabric's dimensions and their stability, its weight, drape, appearance, softness and handle, as well as any required functional properties such as resistance to creasing, flames, water, oil, dirt or bacteria. Textile finishing is therefore an extremely diverse field involving an extensive range of chemicals. This chapter will only provide a brief survey.

Much of this diversity arises from the many different properties that are possible using various finishing techniques, even for the same fabric. The degree of permanency of a particular finish for a fabric depends on its intended use. For example, a crease-resist finish for a cotton/polyester shirting fabric must obviously have a much higher resistance to repeated washing than a flameproof finish used for theatre curtains. Some temporary finishes are, however, useful for facilitating the assembly of a material into a final article. The chemicals used may weight the fabric to make it easy to slide and handle, or allow smooth penetration of a sewing needle to minimize needle breakages.

The majority of chemical finishing methods are used for fabrics containing cellulosic fibres. Before 1950, washing cotton fabrics invariably resulted in severe creasing and shrinkage. Hot pressing or ironing of the washed article, under damp conditions with stretching, produced a smoother appearance and the desired shape. Materials made from synthetic fibres, on the other hand, can be washed and spun or drip dried with virtually no creasing or change in dimensions. At worst, a light ironing might be needed for a completely smooth appearance. The development of new chemical finishes for crease-resist cotton began in the 1950s, once the new synthetic fibres began their rapid ascent in the marketplace. Cotton has remained the major textile fibre because of these chemical finishing techniques, its success aided by blending it with polyester staple to produce fabrics with even better performance. Anyone involved in textile coloration must be aware of the influence of preparation processes on the dyeing behaviour of the fibres. Poorly prepared goods will be difficult to wet and to colour evenly. Equally important, the colourist must also know how the finishing methods will influence the final colour of the goods and their colour fastness properties. Without this information, the colourist will not be able to satisfy a client's colour and fastness specifications.

# 1.2 Object of Finishing

The object of finishing is to improve the attractiveness and/or serviceability of fabric. There is a wide variation of techniques among different fabrics and different production units. In fact, many of them are business secrets; that is why many details have not been published. There are actually very few published works available except about functional finishes, for which specific chemicals serve specific functions.

The variations of finishing depend on the following factors:

- 1. The type of fibre and its arrangement in yarn and fabric
- 2. The physical properties of fibres such as swelling capacity and behaviour when pressure or friction is applied
- 3. The capacity of fibres to absorb chemicals.
- 2 Principles of Textile Finishing
- 4. The susceptibility of the materials to chemical modification.
- 5. The most important factor, the desirable properties of the material during its use

If the inherent property of the material is excellent, such as lustre of silk, little finishing is necessary. The materials made of worsted yarn require less finishing than those made of woollen yarn. The materials prepared from cotton need a variety of finishing techniques, as it has diversified uses.

## 1.3 Classification Of Finishes

The finishing processes may be broadly classified into two groups:

- (a) Physical or mechanical
- (b) Chemical.

The physical or mechanical processes encompass simple processes like drying on a steamheated cylinder to various type of calenders, raising for soft effects on the surface of the fabric and breaking the finishing of filled goods for comfortable feel.

Most of the mechanical finishes are known from ancient times and few changes have occurred in their method of operations. Some physical properties, such as dimensional stability, can be improved with chemical finishing. Mechanical finishing or 'dry finishing' uses mainly physical (especially mechanical) means to change fabric properties and usually alters the fabric's appearance as well. The mechanical finishes include calendering, emerising, compressive shrinkage, raising, brushing and shearing or cropping. The mechanical finishes for wool fabrics are milling, pressing and setting with crabbing and decatising. Mechanical finishing also encompasses thermal processes such as heat setting (i.e., thermal finishing).

Mechanical finishing is considered a dry operation even though moisture and chemicals are often needed to successfully process the fabric. Chemical finishing or 'wet finishing' involves the addition of chemicals to textiles to achieve a desired result. In chemical finishing, water is used as the medium for applying the chemicals. Heat is used to drive off the water and to activate the chemicals. The chemical methods have changed with time remarkably, and the newer finishes have been developed continually. Many chemical methods are combined with mechanical methods, such as calendering, to improve the effect. Typically, the appearance of the textile is unchanged after chemical finishing. Some finishes combine mechanical processes along with the application of chemicals. Some mechanical finishes need an application of chemicals; for example, milling agents are needed for the fulling process or reductive and fixation agents for shrink proofing wool fabrics. On the other hand, chemical finishing is impossible without mechanical assistance, such as fabric transport and product application. The assignment to mechanical or chemical finishing depends on the circumstance; that is, whether the major component of the fabric's improvement step is more mechanical or chemical. Mechanical devices are used in both categories; the major distinction between the two is what caused the desired fabric change, the chemical or the machine?

Another method of classification is to classify finishes as temporary and permanent finishes. In fact, no finish stands permanently till the material is serviceable, hence a more accurate classification would be temporary or durable. Some of the temporary finishes are:

- (a) Mechanical: calender, schreinering, embossing, glazing, breaking, stretching, etc.
- (b) Filling: starch, china clay and other mineral fillers
- (c) Surface application: oil, different softeners and other finishing agents.

Some of the durable finishes are:

(a) Mechanical: compressive shrinkage, milling of wool, raising and cutting processes, permanent 'setting, etc.

- (b) Deposition: synthetic resins—both internal and external, rubber latex, laminating, etc.
- (c) Chemical: mercerization, parchmentising, cross-linking agents, water repellent finish, fire-resistant and fireproofing finishes, shrink proofing of wool, etc.

It should be noted that any such classification is arbitrary. Accurate classification is difficult because durability depends on several factors. Durability can be varied, and it is not possible to draw any borderline between temporary and durable finishes.

Finishing processes are so varied that it is difficult to classify them. For cotton, several finishing processes are used widely, but they are so varied in technique that it is difficult to group them together. For many years, the dispersion processes, namely mercerization and parchmentisation, were the only permanent finishes on cotton, and they still remain of great importance today. The common chemicals used in these finishes are caustic soda and sulphuric acid, respectively, in a moderately concentrated form.

# 1.4 Physical finishing

Physical finishing methods for textiles include optical finishing, brushing and napping, softening, shearing and compacting of the textile structure.

# 1.4.1 Optical finishes

Lustre may be imparted to a fabric by physical means. The techniques basically involve flattening or smoothing the surface yarns using pressure. Beating the fabric surface or passing the fabric between hard calendering rolls under pressure and with some friction will tend to flatten out the yarns and lower light scattering by the fabric surface, thereby improving reflectance and lustre. Lustre may be improved further if the calendering rolls are scribed with closely spaced lines which will be imprinted on the fabric to reinforce light striking and reflecting from the fibre surface. Similar techniques can be used to impart optical light interference patterns on the fabric. Thermoplastic fibres which can deform under heat and pressure can most readily be modified to impart lustre.

# 1.4.2 Brushing and Napping

Physical de-lustering of a fabric, as well as bulking and lofting of the fabric can be achieved by treatments which roughen the fibre surface or raise fibres to the surface. Fibre raising processes, such as brushing and napping, involve the use of wires or brushes which catch yarns in the textile structure and pull individual fibres partly from the yarn structure. The resulting fabric is warmer, softer and more comfortable.

During calendering or beating of a fabric interaction between individual fibres within yarns may be lessened and the textile structure softened. Also, when a smooth textile structure free of raised surface fibres or hairiness is desired, the fabric may be sheared by passing the fabric over sharp moving blades or by passing the fabric over a series of small gas jets which singe and burn away raised fibres.

# 1.4.3 Compacting

During the fabric formation processes, tremendous stresses are applied on textile materials. Such stresses can be controlled by drying the finished fabric with or without tension on a stenter frame, which controls the width of the fabric and the tension on the fabric during the drying process. A second method involves compression of the fabric structure, as in the Sanforizing process. In this process, the fabric and backing blanket (rubber or wool) is fed between a feed roller and a curved braking shoe, with the blanket kept under some tension. The tension on the blanket is released after passing the fabric and blanket between the roller and braking shoe. The net result is the compaction of the fabric. Such a simple technique permits garment making with finished textile goods to be without fear of excessive shrinkage on laundering. Protein hair fibres, such as wool, and thermoplastic fibres, such as polyester, can also be compacted. The scale structures on protein fibres entangle and stick on agitation, particularly in the presence of moisture. The resulting 'ratcheting' effect causes the fibres to compact and felt. Many processes for wool take advantage of this effect, and nonwoven felt structures are produced by this method.

Compaction of the thermoplastic structure occurs when the fibres are raised to near their softening point. At a sufficiently high temperature, the fibres shrink and contract and achieve a stable structure, causing compaction of the textile structure.

#### 1.5 Functional Finishes

Various functional fabric properties may be improved by using suitable chemical and/ or physiochemical techniques. The latter includes coating and exposure to high-energy sources and are gradually superseding conventional wet chemical methods. The use of polymers instead of simple chemicals is increasing in order to improve multiple functional properties simultaneously. The properties of fabrics and fibrous materials are altered to improve their performance with regard to various physical, chemical and/ or biological agents and influences. Such property modifications include: resistance to wrinkling, fire, soils and stains, water, microorganisms and insects, light, heat and cold, shrinkage, air pollutants and chemical agents,

mechanical changes caused by abrasion, pilling and various types of deformation and build-up of static charge. A few finishing processes which improve functional textile properties are listed below along with applicability or demand for specific fibre types:

- 1. Wrinkle resistance or resiliency—for cellulosic fibres and their blends with synthetics
- 2. Flame retardancy—for most natural and synthetic fibres
- 3. Absorbency—usually to impart hydrophilicity to synthetic fibres
- 4. Soil release—primarily for synthetic fibres and their blends
- 5. Repellency (soil and stain)—primarily for synthetic fibres
- 6. Repellency (water)—primarily for cellulosic fibres
- 7. Resistance to microorganisms—primarily for cellulosic fibres, all fibres for medical purposes
- 8. Resistance to insects—mostly for wool fibres
- 9. Shrink proofing—primarily for cellulosic and wool fibres
- 10. Resistance to static charges—primarily for synthetic fibres
- 11. Resistance to pilling—high tenacity synthetic fibres and their blends
- 12. Abrasion and wear resistance—primarily for cellulosic fibres and their blends
- 13. Resistance to UV light, heat and pollutants—for most natural and synthetic fibres, especially polyamide fibres
- 14. Thermal conductivity (hot or cold, thermal comfort)—all natural and synthetic fibres

The physicochemical or chemical methods are employed for the application of functional finishes on textile materials. The former includes application or irradiation of high energy, coating, insolubilisation or deposition and microencapsulation. Chemical methods include polymerization, cross-linking and resin treatment, covalent formation and ion-Exchange/Chelation.

#### 1.6 Chemical Finishes

The proper formulation of chemical finishes is not easy. Several important factors are to be considered before the finalization of a formulation; a few are as follows:

- 1. The type of textile (fibre composition of the fabric and its construction)
- 2. The performance requirements (extent of effect and durability)
- 3. The economics of the formulation
- 4. Availability of machinery and associated process restrictions
- 5. Procedure requirements
- 6. Environmental consideration
- 7. Compatibility and interactions of finishing components.

Chemical finishes should meet the following requirements:

- 1. Low-cost product and process
- 2. Stable during storage and application in terms of pH, temperature and mechanical stress
- 3. Compatible with other finishes
- 4. Adaptation to customer requirement and substrate variation
- 5. Suitable for all kind of fibres and all textile forms such as yarn, woven or knit fabric, garment, nonwovens, etc.
- 6. Satisfactory stability during washing and dry cleaning
- 7. Should not hamper important textile qualities
- 8. On application should be distributed evenly on the fabric and fibre surface
- 9. No yellowing of white goods or colour change of dyed goods.
- 10. Easy correction of finishing faults
- 11. Nontoxic and ecofriendly
- 12. No release of volatile organic compounds
- 13. Biodegradable

Usually, several types of finishes are combined mostly in one bath (only one application and drying process) for economic reasons. This is often the hardest challenge of chemical finishing. First, all components of the finish bath must be compatible. Precipitations of anionic with

cationic products should be avoided. Most of the finishes are marketed in the form of emulsions. The emulsion stability of different products may be reduced by product interactions. The inherent natures or the effect imposed on the substrate of two mixed components may be similar or opposite. Some components assist each other; for example, silicone elastomers may enhance water repellency, softeners may bring additional antistatic effects and antistatic finishes can soften material further. On the other hand, some agents may impart opposite effects; for example, hydrophobic finishes and hydrophilic antistatic finishes, or stiffening and elastomeric finishes, or stiffening and softening finishes.

# 1.7 Plasma Finishing

The coupling of electromagnetic power into a process gas volume generates the plasma medium comprising a dynamic mix of ions, electrons, neutrons, photons, free radicals, metastable excited species and molecular and polymeric fragments, with the system overall being at room temperature. This allows for the surface functionalization of fibres and textiles without affecting their bulk properties.

In the textile field, significant research has been done since the early 1980s in various laboratories across the world. The researchers mostly dealt with low-pressure plasma treatments of a variety of fibrous materials. Such works showed very promising results regarding the improvements in various functional properties in plasma-treated textiles. A variety of commercial low-pressure plasma machines, mostly in prototype form, have been offered for batch/in-line processing of textiles for more than 15 years.

In recent times, some companies have also started to offer commercial systems for atmospheric-pressure plasma processing of textiles, both off-line and on-line. The potential use of plasma treatments of fibres, yarns and fabrics are promising for various types of functionalization; examples are listed below:

- 1. Anti-felting/shrink resistance of woollen fabrics
- 2. Hydrophilicity enhancement for improving wetting and dyeing
- 3. Hydrophobic enhancement of water and oil-repellent textiles
- 4. Removal of the surface hairiness in yarn
- 5. Antibacterial finish
- 6. Room-temperature sterilization of medical textiles

- 7. Flame-retardant coating using monomer vapour (halogen and/or phosphorus) in combination with nitrogen and/or silicone
- 8. Silicone coating of airbag fabrics using cross-linked silicones (poly-organo-siloxanes)
- 9. Durable antistatic properties using PU-resin and plasma processing
- 10. Shrink resistance of animal hair textiles using urethane-based resin and plasma processing
- 11. Electro-conductivity of textile yarns surface. The plasma treatment improves wettability and soil release properties of polyester.

#### 1.8 Coated Fabric

The coated fabrics are becoming more popular day by day primarily for technical textiles as water repellency, air permeability, etc. Coating can be applied on any fibrous substances including glass, polyethylene and polyethylene in woven, knitted or nonwoven form. Woven coated fabrics are known for high strength, while knitted coated fabrics have high elongation properties. Insolubilisation of chromium compounds inside the textile materials can impart resistance to UV light or sunlight. Antimicrobial properties can be improved by microencapsulation with quaternary ammonium salts.

# 1.9 Application Of Chemical Finish

Chemical finishes can be applied by a number of methods including exhaust (running batch wise in finish liquor after dyeing), padding and curing (immersion in the treatment solution followed by squeezing to remove excess and heat treatment), spraying, printing, foam application or vapour techniques. In addition, the finish can be added to the spinning bath prior to formation of manmade fibres.

In the exhaust method, after the dyeing process in a winch, jigger or jet dyeing machine, the liquor is drained and the textile material is thoroughly washed. Fresh water is added to the finishing liquor and the material is run for a specific time. After a specified time, the material is sent for drying without washing. This is known as the batch process.

The most popular method is the padding method. In a padding machine, the material is continuously dipped in liquor and squeezed to a certain degree (called percent pickup or percent expression) by passing between a pair of rollers. After padding, the fabric must be dried (i.e., water is removed) and cured (i.e., heated to cause a chemical reaction) in a separate machine before chemical finishing is complete. In the continuous method, the fabric after padding is

continuously passed through a cylinder drier, curing machine or a stenter. The process is often referred to as pad-dry-cure. Each part of the process can influence the outcome of the treatment. The other method is to pad the fabric and to roll it on a roller for batching for a specific time. However, his semi continuous pad-batch process is popular for dyeing but not for finishing. Wetting of the fibre by the finish solution and spreading of the finish evenly over the fibre surface is critical in most cases to get the desired effect. The location of the finish on the surface or within the fibre is important, depending on the finish and its function.

# 1.10 Padding Mangle

The padding mangle (Fig. 1.1) is perhaps the most familiar and universal of all textile dyeing and finishing machinery. It gained popularity for the application of dyes and chemicals in continuous and semi continuous dyeing and finishing processes. The satisfactory performance of the padding mangle is absolutely essential for the success of such processes.

The padding process is suitable for applying dyes and chemicals having low or no affinity for the fibre. If the chemicals used in the padding mangle have a high affinity, their concentration in the trough decreases with time, resulting in a tailing effect. The effect can be minimized by using feeding liquor with a higher concentration than the solution initially fed to the trough. The padding mangle is not a complete machine by itself; other machines are necessary for fixation of chemicals in the padded fabric. The padding operation consists of two steps. First, the fabric, usually woven, is immersed in the liquor to achieve thorough impregnation. Second, the fabric is passed between two rollers to squeeze out air and to force dye-liquor inside the material, with the excess liquor being sent back along the fabric. The former step is known as dip, while the contact between the squeeze rollers, as well as passing between the rollers, is known as nip. For one dip and one nip padding process, the fabric is immersed in the trough once and passed through the nip once. For finishing, most fabrics at speeds of up to 50 m/min, single-dip and single-squeeze processes are sufficient. For higher speeds (e.g., 120 m/min) or for heavy fabrics, the double-dip and double-squeeze process using a three-bowl padding mangle is preferred. The liquor retained in the fabric after padding is expressed by weight as a percent of the weight of dry fabric and is known as percent pickup or percent expression. Thus, 80% expression means the fabric weight is increased by 80% of dry weight after padding. The expression less than 60% is difficult to obtain in a padding mangle. Increased pressure at the nip results in a lower percentage expression, but at the same time, the penetration inside the fabric is better. The fabric is subsequently passed through various machines for the fixation process.

Mangle width for textile applications may be as high as 4–5 m, but widths of 1.7–2 m are more common for the continuous dyeing and finishing of woven fabrics. In old mangles, one of the rollers is covered with rubber, while the other one, the driving roller, is made of stainless steel, brass or ebonite. However, such types of mangle may give face to back difference on the fabric. Now both rollers of the mangle are of similar hardness. Most of the padding bowl mangles have a steel mandrel covered with about 15-mm deep hard rubber with a comparatively soft rubber surface having softness of 55–70° shore hardness. The softer the rubber, the more readily it deforms under pressure to provide a greater area of contact and thereby reduce the pickup of fairly open fabrics. However, such rollers are also more readily damaged, especially at the selvedges, and so a compromise must be established. The physical strains may cause unevenness, particularly for wider padders, and regrinding may be needed from time to time.

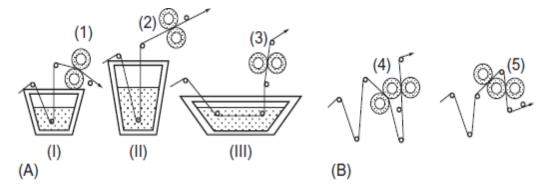


Fig. 1.1 (A) Two-bowl padding mangles with varying troughs and bowl arrangements.(B) Three-bowl padding mangles with varying bowl arrangements.

The mangles should be equal in diameter, usually 30–40 cm, but larger diameter rollers are also used for high-speed running. With larger diameter rollers, the revolution per minute for a given fabric speed and the deflection under pressure are less, but they require larger bearings and a heavier foundation. Depending on the pressure applied, higher diameter rollers have a larger area of contact between two rollers. The chemical composition of the rubber surface should be chosen carefully so that it is unaffected by the liquor containing finishing agents. For cross-linking finishing, it should be impervious to formaldehyde condensates. Immediately after a run, the rollers should be washed thoroughly to avoid hardening or fixing of finishing agents on the rubber surface.

In most of the two-bowl padding mangles, the lower bowl is in fixed bearings and the upper bowl is separated by about two centimetres when the mangle is not operating. The bearings of the top roller are mounted on arms pivoted at the side of the mangle and actuated by pneumatic cylinders attached to each side of the machine. Just before the padding operation, the upper roller is moved to press against the lower roller by pneumatic pressure created by a small compressor. The pressure is applied to the mandrel at the end of the roller, the maximum pressure being about 400 N/cm (50 kg/cm) of bowl width or about 10 tonnes across the total width, 5 tonnes at each end. In the three-bowl padding mangle, the centre bowl is in fixed bearings, and the two outer rollers are pressed against the centre. When the mangle is not operating, the top rollers should be detached from the fixed rollers in an open position. The roller in fixed bearings is driven from one side of the machine; the drive system depends on the maximum speed of the mangle, which may vary from 25 to 120 m/min. The sides of the padding mangle must be robust in order to withstand the forces applied. Applying pressure to the ends of a roller causes some deflection of the roller providing lower pressures (and consequently a higher pickup) in the middle of the pad and higher pressures (and lower pickup) at the sides. The deflection can be measured by using the Shirley Bowl Deflection Indicator. To counteract the effect of the bowl deflection, the surface of one or both bowls may be tapered slightly towards each end.

The problem of bowl deflection can also be tackled by using a special mangle bowl, such as swimming roll mangle of Küsters. The roll has a stationary mandrel, around which the roller shell rotates in bearings. The space between the mandrel and the shell is divided by pressure seals in two compartments. The smaller compartment, pressurized by oil, is always towards the nip. Oil is constantly pumped into the compartment, with some passing the seals and circulating back to the reservoir. When the oil pressure in the compartment is increased, the surface pressure at the middle of the roll is raised, and therefore the deflection of the mandrel is compensated. By controlling the internal oil pressure (maximum 500 kPa or 71 lbf/in.2) and hydraulic pressure at the ends of the mandrel, the configuration of the bowl can be adjusted to have a uniform linear pressure at the surface. The swimming rolls may be used in pairs or in combination with another conventional roller. The Artos stabilized roller system consists of three components, and the load is applied at the tube shaft at the centre. Although the inner shaft deflects under pressure, the force is transmitted to the outer sleeve via the inner sleeve covering only the middle section of the roller. To minimize oxidation of the rubber, the rollers should be covered during storage and protected from sunlight, moisture, excessive heat or cold. After prolonged storage, the oxidized surface is to be ground for better results. The pressure should be applied only when the rollers are running and they should be cleaned thoroughly after use.

Situated below or at the front of the squeeze rollers is the padding box or trough, having a freerunning roller at the bottom which guides the fabric through the trough. The box may be Vshaped, deep immersion type or shallow type (shown in Fig. 1.1A as (I), (II) and (III), respectively) having a width slightly more than maximum width of the fabric to be processed. The volume of the pad box should be as small as practicable (about 2 gal for a two-bowl padding mangle with 60-in. bowl width) so that the waste of the dye-liquor is minimal at the end of the run. It is made small in order to avoid a tailing effect, a problem that arises from the preferential pickup of dye or water by the fabric, resulting in a variation in composition of the liquor as the run continues. The finish liquor may be fed to the trough manually or with the help of a pump through a perforated pipe running across the back of the box. The perforations are pointed down and are away from the fabric. A constant level of dye liquor in the box can be maintained with the help of a float switch. For a very wide fabric, a liquor circulation system is often recommended, but the advantages are questionable. A drain plug is fitted at the bottom of the box for rapid draining at the end of run. The interior of the pad box should be smooth and easy to clean. Usually, impregnation is done at room temperature. However, some pad boxes are fitted with an external heating jacket for the circulation of hot water or oil. Hot impregnation may be required to achieve rapid and uniform wetting-out and high pickup of dyes. When two pad boxes are fitted in tandem, the second is kept at a slightly higher level so that the liquor overflows from the second to the first.

The mangles may be arranged vertically opposite each other in ascending or descending order or as horizontally opposite each other. Such three arrangements with Artos two-bowl padder are shown in Fig. 1.1A as (1), (2) and (3), respectively. For three-bowl padders, all three may be inclined with a steady central bowl, while the top and bottom bowls press themselves against the central steady bowl with the action of levers and pneumatic pressure. There may be a combination of ascending and horizontal arrangement with two-dip (same or different bath)-wo-nip (4) or descending and horizontal arrangement with one-dip-two-nip (5) arrangements, as shown in Fig. 1.1B. The ascending-bowl system gives a better observation of the nip, while in a descending-bowl system, the fabric path does not change much. With a horizontally opposed mangle, the fabric rises vertically from the padding box directly to the nip and straight through a dryer mounted above the nip. This arrangement is widely used for the Thermo-sol dyeing process for polyester and its blends. In a three-bowl arrangement, maximum impregnation occurs at the first dip-squeeze and maximum uniformity at the second squeeze. After impregnation and before entering the nip, it is necessary to take the fabric over a free-

running smooth roller. The rollers should be positioned so that the fabric path leaving the pad box is nearly vertical, and upon leaving the rollers, the fabric enters straight to the nip. Nip guards covering both the entry zone and the sides may be fitted. Upon leaving the nip, the fabric should leave both rollers simultaneously, otherwise face-back differences may appear. The number of rollers following padding should be less so that the squeezed wet fabric has fewer points of contact. The fabric is fed from the plaited form to the pad bath after skying or floating in air over guide roller. An end piece is to be attached to the tail end if it is run from a large roll. Alternately, the fabric may be run through an accumulator, which will continue to deliver to the padding mangle. The fabric must be dry but readily absorbent, as the immersion time in the mangle is very short, about 0.5 s. It should also be free from loose impurities, waxes, oils, etc. Polyester-cellulosic blended fabrics are to be heat-set before padding. These fabrics, which are slow in wetting-out, may have to be treated with a rewetting agent beforehand. The fabric must be flat and crease-free. The knitted fabrics require low lengthways tension and the presence of scroll rollers and edge uncurlers. The fabric must be centered accurately on the padding mangle and run under moderate lengthways tension. Excessive tension gives poor absorbency and low liquor pickup.

# 1.11 Low-Pickup Padding

In order to reduce the volume of dye bath and to stop draining back of the liquor after preferential absorption, trough less mangles such as Fibre padding mangles (Benninger Engineering Co.) have been developed. In such machines (Fig. 1.2), two sets of horizontally opposed mangles are placed one above the other, so that each of the four bowls makes contact with two others and there are four nips. These bowls are provided with to-and-fro as well as up-and-down motions, and no separate trough is used. The geometry of the rollers provides some space between the four rollers where the liquor is held. Two stainless steel plates covered with rubbers sheets about 3 in. thick on the inner sides are fixed at the ends of the rollers to prevent the padding liquor licking through the ends. A perforated pipe (shown above the guide rollers in the figure), entering the liquor space through one of the steel end plates, feeds the pad liquor. The liquor is fed at the same rate at which the liquor is taken up by the fabric during padding and is circulated by a centrifugal pump. Only about  $2\frac{1}{2}$  gal of liquor is to be held in the space in 36-in. width machine.

The fabric enters between the nips of the topmost rollers, which are under considerable pressure. The high pressure removes much of adsorbed air from the fabric so that it is wetted very quickly. It then passes over a guide roller inside the trough and horizontally through a nip.

It passes around two guide rollers outside and reenters the trough through the nip of two bottom rollers. The fabric then passes around a guide roller in the trough and goes out horizontally through the nip of two vertical rollers. However, the wedge nip, as it is sometimes called, is restricted to padding lightweight fabrics. The thicker fabrics suffer from liquor seepage at the selvedge. There are difficulties in wetting-out in such a short time, and the continuous expulsion of air may cause frothing. The setting of end plates has to be adjusted very accurately. The wedge nip is normally used as the second stage in combination with the conventional dipsqueeze unit. The presence of occluded air in the fabric structure prevents the ready absorption of dye liquor during the padding process. It was observed that a roll of fabric when put under vacuum is readily saturated with liquor. Farmer Norton designed a machine for continuous vacuum impregnation. The fabric is carried on a rubber belt underneath a perforated rotary screen, in the first half of which suction is applied to the fabric, while in the second half, dye liquor is fed at atmospheric pressure. The fabric is then passed to a mangle to remove excess liquor, which may be drained or recirculated. Good absorption may be achieved even on loomstate fabric containing sizes. However, such a machine is not commercially popular as adequate impregnation may be achieved on most of the fabrics in conventional padding mangle by a two-dip-two-nip process. Moreover, very high penetration in thick fabric necessitates an increased quantity of liquor.

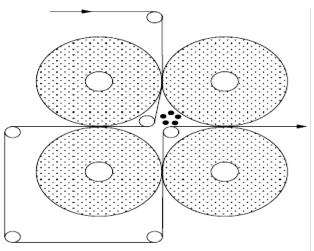


Fig. 1.2 Fibe troughless padding mangle.

For conservation of energy, lower liquor retention during padding is necessary. The relatively high liquor retention from normal padding techniques is expensive in terms of energy requirements, because drying is one of the most expensive stages of processing. Furthermore, migration is a more serious problem when the fabric contains excess liquor. To minimize liquor retention, the pad-transfer technique has been suggested, in which the padded and squeezed

fabric encounters dry fabric. Owing to the initial oversaturation, however, the amount of liquor remaining is still typical of that achieved by a good conventional system.

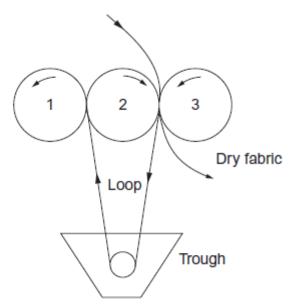


Fig. 1.3 Loop-transfer system with three-bowl padding mangle.

In the three-bowl loop-transfer technique (Fig. 1.3), the fabric is not directly dipped into the trough. Instead, a loop of fabric dipped into the trough encounters the dry fabric while passing through a nip. The pickup of liquor at the transferring loop can be controlled by varying both squeezing pressure (between Bowls 1 and 2) and transferring pressure (between Bowls 2 and 3) independently. Reproducibility and levelness are better at higher transfer pressure. Fixation values for fabric by either loop-transfer or conventional padding showed close agreement. The methods designed for low pickup values in the range of 5–40% region are of considerable interest for the application of finishing agents. The use of a coated fabric with a spongy surface as a backing fabric or an endless conveyor backing to give a more effective squeezing of heavy fabrics in continuous dyeing and finishing.

# 1.12 Vacuum Slot or Suction Hydroextractor

If a vacuum slot is added in line with a padder, the vacuum will extract the liquid residing in the fabric capillaries and open spaces, therefore reducing the wet pickup. Water in the capillaries and spaces between fibres and yarns is not bound as strongly as water in the interior of the fibre. The amount of water in the pores and spaces can be reduced by passing the fabric over a vacuum slot. Water extraction from such fabrics can be safely carried out in open width by passing over a vacuum slot or suction hydro extractor (Fig. 1.4), which is often located in front of a stenter or cylinder dryer.

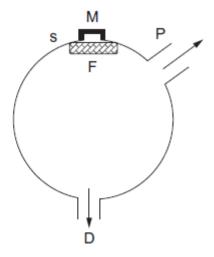


Fig. 1.4 Suction hydroextractor.

The slot (S) is cut along the top of a horizontal cylinder of about 20 cm in diameter, coupled at the bottom to a large-diameter pipe leading to a gauze filter (F) (to trap any loose yarn), a water separator (D) and a vacuum pump (P). The machine draws the air through the fabric into the slot, and the rush of air sweeps out extraneous water from the fabric. Obviously air leakage is to be minimized, so the lengths of the slot at either side of the fabric are covered with rubber or plastic sleeves or masks (M). The fabric must be centered before passing over the slot, and then around a pair of draw rollers. Water is removed in the form of droplets driven from the fabric by air flowing into the vacuum system. Some evaporation may take place, but within the fabric, the removal is almost wholly mechanical. The rate of removal is so rapid that a 2–3 mm wide slot may give satisfactory suction drying at a speed of 60 m/min with contact time of 2–3 µs. Except for very dense fabrics, a vacuum of 40 kPa is adequate.

The important variables in suction drying are the vacuum achieved, the fabric speed and the slot width. When the vacuum pump runs at a fixed rate, the vacuum achieved will depend on the slot width and the density of the fabric. At a constant vacuum, the slot width alone has a very little effect on water retention. With a given slot width, retention does not change much when the fabric speed is increased. Investigation showed that most of the water removal occurs in regions close to the slot edges, and the central region contributing very little, although it does influence the airflow into the vacuum chamber. For the highest possible vacuum, the slot should be narrow, and multiple slots may be advantageous. The achievement of an adequate level of vacuum is the main practical problem in suction drying. Open structured fabrics lower the vacuum, thereby obstructing easy water removal. Suction drying requires a separate mechanism for fabric transport, and the power requirement of the vacuum pump is more than

double that required for mangles. Nevertheless, the suction process is less severe and has a useful role in mechanical drying. The advantages of vacuum slots are:

- 1. They are relative simple devices and are easily fitted onto existing finishing ranges.
- 2. The extracted solution can be saved and fed back to the pad after the lint has been filtered out.
- 3. The entire fabric is exposed to the treating solution, therefore the solution is uniform throughout the fabric cross section. Face-to-back uniformity is good.
- 4. Units have been designed to have movable slot openings to accommodate fabrics of different widths.

The Evac-Nip by Eduard Kusters Maschinenfabrik GmbH, Germany, is a prototype machine that combines chemical application with a wet pickup reduction. A patented vacuum assembly is used to draw hot air through the wetted fabric after immersion prior to the fabric contacting a heated cylinder. The exiting fabric has a low moisture level and can be processed more efficiently in a subsequent drying/curing operation. The Evac-Nip seems to be well suited for finishing applications and provides a distinct advantage over conventional vacuum slot applicators, since additional moisture is removed prior to final drying and curing. This lower moisture level should assist in preventing finish migration during drying. Morrison Textile Machinery, USA, presented the Micro-Stat, a combined fabric saturator and chemical-dosing system. As the fabric passes through a double-immersion, low-volume pad, a microprocessor with touchscreen controls calculates the necessary feed rate of the replenishing chemical solution based on the desired add-on and actual fabric flow. Very consistent add-ons can be realized with minimal chemical waste. This approach addresses only one aspect of finish application; the problems associated with high wet pickups, finish migration and longer drying times still remain.

# 1.13 Drying and Curing

The water dispersed in a textile material by a chemical—physical process is generally eliminated by the action of hot air, which makes the water evaporate. During the drying process, it is very important to carefully consider the way heat is directed on the fabric.

The drying process can be carried out by:

• Heat convection

- Contact with heated metal surfaces
- Infrared radiation
- Microwaves or high-frequency waves
- Combustion

Yarns and textile materials in bulk are generally dried inside hot air compartments. In finishing, water is used to distribute the chemicals evenly in the surface and interior of the fibres and fabrics. After the application of finishes, water is evaporated to make the fabric dry. Though the concept of drying is simple, it can be the source of unsuspected problems too. It is also a high energy-consuming step and in these days of energy crisis, this significantly affects the cost of finishing. For drying to take place, the water must be converted to vapour, and the vapour must be moved away from the surface. Air with a low relative humidity has a greater capacity for water vapour than air with a high relative humidity. When the relative humidity nears the saturation value, liquid water is in equilibrium with water vapour, and so evaporation is slowed. The absolute amount of water vapour in a volume of air is a function of temperature. For example, at any given relative humidity, the actual amount of water vapour is much greater at high temperatures than at low temperatures. The rate at which water evaporates from fabric depends on the temperature of the fabric and the volume of air that passes through it. Evaporation of water occurs rapidly at its boiling point. When wet fabric is heated with hot air (regardless of the air's temperature), the temperature of the fabric will not rise above water's boiling point until all the liquid water is gone. As water evaporates, the fabric is cooled by evaporative cooling. Some of the factors affecting drying rate are air temperature, relative humidity of drying air and volume of air passing over fabric (air flow). Liquid held in the capillaries is responsible for the phenomenon called migration. If a fabric is heated from one side, the dissolved or suspended chemicals will concentrate at that side. The movement will continue until the moisture level in the fabric is reduced below that required to sustain the filled capillaries. For cellulosic fabrics, this level is about 30–40%. To prevent migration, dryers must be designed to evenly remove water from both sides of the fabric. Once the level of moisture is below a critical point, the source of heat becomes less critical.

Curing or fixation is also a thermal treatment, generally at a temperature much higher than that of drying. The same equipment used to dry fabric is also used for curing, provided the equipment is capable of reaching curing temperatures. In many finishing procedures, drying

and curing are done in two successive steps. Each step will have its own individual specified conditions. Sometimes however, no delineation between the two is made; wet fabric enters the machine and cured fabric exits. The type of dryer used will affect fabric properties. The cylinder dryer, in which the fabric is passed over hot metallic cylinders, was once very popular. But some fabrics will develop a surface sheen when they come in contact with heated surfaces, and some will shrink unless the fabric is restrained. Tension can cause the fabric to become stiff. The main limitation of a conventional cylinder dryer is that there is no control of fabric width, and a final finish is often given on a stenter to ensure consistent and specific width of the final fabric. Additionally, tension can distort fragile fabrics.

Choosing the right equipment to handle the fabric is an important matter when one is trying to create specific fabric properties. Loop ovens differ from roller ovens in that the rollers move horizontally from the entry side of the chamber to the exit side, carrying a loop of fabric along the way. The loop is formed by forcing the fabric between adjacent rollers, sometimes called sticks. The ends of the sticks are attached to a rotating mechanism that constantly propels them through the chamber. Hot air circulates through the chamber, and the loops are free to flap around as they dry so that the fabric can dry in a relaxed state. However, this may result in huge shrinkage.

# 1.14 Stenter or Tenter

Setters are the most expensive and most important machines in a dye house and finishing works. However, the stenter is the only drying machine that provides adjustment and control of fabric width during drying. In addition to drying, the stenter performs several other functions, namely:

- 1. Heat setting of fabrics made of synthetic fibres and blends.
- 2. Applying and fixation of several finishing agents.
- 3. Imparting a particular mechanical finish affecting appearance and feel, commonly known as stenter finish.

A tender or stenter is a transport device that carries fabric from one point to another continuously in open width with precise width control. The stenters are of two types, pin stenters or clip stenters, depending on the means of holding th1e fabric onto the chains. In clip stenters, the clips are in two sections, upper and lower; the upper being in either in an open or closed position. At the entry point of the fabric, the upper jaw descends and grips the edge of

the fabric. At the delivery point, the jaw opens and releases the fabric. The clip stenter requires a specific edge, preferably a selvedge, to grip and is usually confined to woven fabrics. The more common pin stenters have pins about 5 mm long, mounted in two rows, about 3–4 pins/cm, along the base plate. The pins are thicker near the base to prevent direct contact of the fabric with the hot base plate. Clip stenters leave no pin marks in the selvedges, but customers will accept pinholes in the selvedges associated with pin stenters.

The speed of stenter ranges from 10 m/min for heavyweight to 100 m/min for lightweight fabrics. The actual speed also depends on the particular operation (e.g., drying, heat setting, weft stretching or finishing) being carried out. The higher the speed, the more vital the need for a good entry system to ensure that the fabric is properly centered and the edges are fed onto the pins at the correct speed. Two endless chains equipped with either clips or pins grasp the selvedges and move in synchrony, carrying the fabric between them. The width between the two chains can be automatically adjusted by means of a motor-driven screw. The entry end is equipped with edge sensors, which signal the screw to move the chains in or out to assure that the edges have been engaged. The width along the length of the chain can be varied by additional independently driven screws located downstream. Sections of the chain can be set to different widths. For example, the width may be narrow at the entry point and be pulled out at a subsequent section to make it wider, or the final width may be set narrower and allowed to shrink. The stenter also has a provision for length control. The fabric either can be stretched or overfed as it enters onto the pins or clips. The yardage yield may be increased by stretching. Pin stenters allow the overfeeding of wet fabric onto the pins by a separate drive mechanism. The fabric will shrink when it enters in the heated zones. As a result, residual shrinkage of the fabric is low but yardage yield also diminishes. The modern stenters consist of multiple closed heated chambers, each about 3 m long. The schematic diagram of a modern four-chamber stenter is shown in Fig. 1.5. A variety of methods are used to heat the air circulating in stenters via the heat exchanger, namely:

- 1. High-pressure steam is released from the boiler, but this will only provide a maximum air temperature of 165°C—adequate for drying and curing, but inadequate for the heat setting and thermo fixation of dyes.
- 2. A thermo-stable oil called a thermic fluid is heated to a temperature above 250°C. A separate thermic fluid boiler is the best choice, but the provision of a separate boiler and other accessories increases the capital and maintenance costs.

- 3. Separate oil burners discharge into one side of the heat exchanger of each compartment, but the arrangement may cause oil spots and soot due to incomplete combustion.
- 4. Burnt gas fumes may be fed directly into the stenter chamber. To avoid any build-up of unburned gas in the stenter, the exhaust fans must be operating for 30–60 s before the gas supply is turned on. Indirect firing through a heat exchanger can be used for gas, but the major disadvantages are additional capital cost, together with a lower thermal efficiency (only about 60% against direct firing).
- 5. Setters may be built with electrical heating, but the electrical energy requirement and the operating cost will be very high.

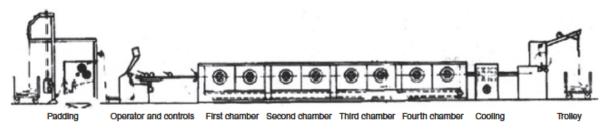


Fig. 1.5 Schematic diagram of a modern four-chamber stenter.

The temperature in each zone can be independently set to a profile of any drying and curing conditions desired. The chambers are equipped with hot-air distribution heads that blow hot air directly onto the top and bottom of the fabric. Hot air is forced into pairs of tapered ducts extending across the width of the fabric and is discharged on it. In some stenters, the ducts have nozzles running the full width to provide uniform air distribution onto the fabric. Some other designs have circular nozzles arranged to strike the fabric surface at an angle. It is most important, particularly during heat setting prior to dyeing, that hot-air distribution is uniform across the full width of the fabric. A reasonable balance of air distribution is to be maintained between the top and bottom ducts. During drying, slightly more air is forced into the bottom ducts to support some of the fabric's weight and also because the bottom of the fabric is wetter than the top. This helps to prevent excessive sagging, particularly in knitted fabrics, but it should not be overdone; otherwise, the fabric may be lifted off the pins. The hot air, after being forced against the fabric surface, makes its way between the ducts to the sides of the stenter, where it is collected by a fan and recirculated. At the top of the compartment is a damper that regulates the rate at which exhaust gases, including steam, are removed into an exhaust duct. Either slots or tubes direct the air onto the fabric. As soon as all the water is evaporated, the fabric temperature reaches the temperature of the heated air. The time it takes to travel from this point to the exit end is the actual time (curing time) the fabric is exposed to the higher temperature. Some modern frames are equipped with fabric temperature sensors located along the length of the frame as a better way of controlling the process. By knowing the point within the frame when the fabric is dry, the frame could be programmed to give it the time necessary for curing to take place.

# 1.15 Mechanical Finishing Methods

Most mechanical finishing processes modify the appearance and handle of a fabric. Table 1.1 lists some typical mechanical finishing operations and their objectives. These processes are used on a variety of fabrics containing different types of fibres. Their major effect is to modify the fabric surface, usually either to make it smoother, or to raise a pile. Both effects can cause a change in the perceived colour of the material because they modify the reflection of light from the fabric surface.

Napping, or raising, and calendering are the most common mechanical finishing methods for fabrics. In napping or raising, the fabric passes over rapidly rotating rollers covered with thousands of fine steel wires projecting from their surface. The points or small hooks on the wire tips break fibres in the yarn surface and pull out their ends, thus producing the characteristic soft fluffy surface or nap. This gives a thicker, warmer material since the trapped air is a good thermal insulator. The penetration of the pins into the surface yarns must be limited to avoid excessive abrasion and weakening of the yarns. The presence of a lubricant helps to minimize fibre damage. Other related mechanical processes used on fabrics with a nap or pile include brushing and shearing (Table 1.1).

Table 1.1 Mechanical finishing processes and their objectives

Process	Objective
Calendering Napping or raising Brushing Shearing Compressive shrinking	Pressing and smoothing the surface of the fabric Breaking fibres in the yarn surface and raising a pile Laying the pile in the same direction Cutting the pile fibres all to the same length Compressing the fabric to the dimensions it would have after shrinkage caused by washing

In calendering, the fabric passes between heavily loaded rollers. At the nip, the high pressure exerted on the fabric flattens and smoothens its surface and loosens any yarns stuck together with finishing chemicals. The fabric becomes softer and more flexible. By wrapping the fabric through a series of calender rollers, the face in contact with a roller alternates from one roller

to the next. The rollers may have identical or different surfaces, such as all steel, or alternating steel and cotton-covered rollers. In some cases, the metal rollers may be heated. To increase the lustre of a fabric, the linear speed of the polished steel rollers may be much higher than that of the fabric generating a polished surface by friction. To enhance this glazing effect, the fabric may be friction-calendered after applying a solution of starch or gum and drying. These chemicals simply fill in the natural undulations of the fabric surface, increasing the smoothness and lustre. Often, relatively inexpensive natural polymers are used simply to increase the weight and decrease the flexibility of a fabric to facilitate sewing up into garments. Heated engraved rollers are used in processes similar to calendering for embossing the fabric surface. These types of finishes are often not resistant to washing unless carried out in combination with a reactive polymer such as those used for crease-resist finishing. One desirable characteristic of a textile material is dimensional stability. This means that the material will not easily stretch or shrink during normal use. The elasticity and easy deformation of many knitted materials often lead to poor dimensional stability. Knitted fabrics require careful manipulation during processing to avoid their over-extension. When cotton fibres absorb water, they swell and the fibre diameter may increase by as much as 18%, much more than the relative increase in fibre length. Viscose fibres swell even more. Water absorbed by these cellulosic fibres acts as a plasticizer. Any strains introduced during manufacture or use will invariably result in shrinkage on wetting because the absorbed water allows the cellulose chains to move to relatively strainfree positions. To avoid this, some cotton fabrics undergo a treatment called compressive shrinkage (e.g. Sanforising). The material is compressed to the dimensions it would have after relaxation by washing. In one method, the humid fabric is firmly held against the outer surface of a thick rubber belt which has been stretched by passing it around a small diameter roller. As the rubber belt leaves the roller, the stretched surface relaxes and compresses the fabric held against it in the warp direction. The degree of compressive shrinkage is greater, the smaller the diameter of the roller stretching the belt surface.

## 1.16 Thermal Finishing Processes

Drying is by far the major thermal process for all textile goods. During manufacture of a fabric, there may be as many as three drying operations. These contribute significantly to the total cost of production. For fabrics, drying in a tender frame is common. The fabric selvages are either held by clips, or set onto small vertically oriented pins, mounted on horizontal continuous chains running alongside the selvages. The chains run continuously down each side of a hot air drying oven and back to the start. By increasing the distance between the chains, the two edges

of the fabric can be gradually pulled apart so that the fabric dries at the required final width. When using pins, it is possible to overfeed the fabric onto the pins at a speed slightly above that of the chains. In this way, some lengthways shrinkage is possible without producing undue tension in the fabric. The hot air for drying is usually generated by heat transfer from steamheated pipes, or from the combustion of gas. To improve the efficiency of heat transfer to the wet web, the circulating hot air often impinges on the fabric surface at relatively high speeds from appropriately placed slots or jets. A tender frame usually has a series of independently operated sections so that the temperature in each section can be controlled. Fabrics are also dried by passage over a series of smooth, steam-heated metal cylinders. The fabric face in contact with the heated surface alternates from one cylinder to the next. The initial cylinders often have a lower temperature than the later ones to provide more gradual heating and drying of the goods. This avoids undue migration of unfixed chemicals and their deposition on the heated metal surface. Drying on hot cans smoothens the fabric surface giving a just-pressed appearance. In the hot flue dryer, the fabric passes in vertical loops through a hot air chamber guided by a series of rollers. Again, to increase drying efficiency, hot air from jets impinges on the fabric surface at high speed. The festoon dryer is useful when fabric must not be under tension. The fabric passes through the hot air chamber in long loops that hang from a series of moving bars. The drying of yarn packages in hot air is particularly inefficient unless the air passes through the packages. Despite their expense, microwave and radio frequency dryers are becoming more popular. The electromagnetic radiation is able to penetrate into the wet package interior to effect uniform drying and the rate of heating drops sharply once all the water has evaporated so that over-drying is avoided.

Thermal drying of a dyed material may cause a slight change in colour related to changes in the fabric surface and possibly to some thermal instability of the dyes present. The perceived colour is often a function of the final humidity and temperature of the goods. Hot air for drying is frequently generated by burning gas and circulating the combustion gases into the drying chamber. A gas flame is sufficiently hot that a low concentration of nitrogen oxides can develop from reaction of oxygen and nitrogen in the air. Oxides of nitrogen are effective oxidizing and diazotizing agents that rapidly fade the colours of sensitive dyes and modify finishing chemicals. Heat setting is the other major thermal process in finishing. It gives fabrics of improved dimensional stability during subsequent washing or heating. Heat setting of synthetic fibre materials is usually carried out using a tender frame. It may be possible to dry the fabric

in the first sections of the frame, and then to heat set it in the latter sections operating at higher temperature.

#### 1.17 Chemical Finishing of Fabrics from Cellulosic Fibres

Most chemical finishing methods are for fabrics containing cellulosic fibres. They give fabrics containing cotton and viscose the easy-care properties usually associated with fabrics made from synthetic fibres such as nylon and polyester. In particular, the use of crease-resist and durable-press finishes for fabrics containing cellulosic fibres is widespread. The fabrics produced have good crease resistance and dimensional stability.

Some chemical finishing methods are quite simple, such as drying a material after rinsing it in a dilute solution of a softening agent. Others are much more complex, such as the continuous pad-cure finishing of a cotton fabric to give it wash-fast flame resistance. Almost all chemical finishes give the material characteristics that it would not otherwise have. Good fastness to washing of a finish usually involves combining the functional chemicals with polymer precursors. These undergo further polymerization in the fibre, or react with the cellulose, and by so doing anchor the functional chemicals in place. Many such finishes are produced by the same series of operations. Firstly, continuous padding of the dry fabric impregnates the fibres with a solution or dispersion of the required chemicals. The padding solution invariably contains a wetting agent and often a softening agent. Newer processes involve the add-on of the minimum amount of solution to minimize migration of chemicals during drying and the cost of drying. Low add-on techniques include the superficial application of the chemicals as a thin layer of foam or liquid. Alternatively, after padding of a woven fabric, vacuum slot extraction decreases the amount of solution in the material, the extract being recycled. This technique has had a considerable impact in the crease-resist finishing of cotton/polyester fabrics. Vacuum extraction leaves sufficient chemical solution in the wetted cotton fibres to promote the desired crease-resist effects but removes it from the polyester fibre surfaces where it has no beneficial influence. After padding, the fabric is dried under conditions that minimize migration of unfixed chemicals to the yarn surfaces where water is evaporating. Accumulation of chemicals at the yarn surfaces produces a stiffer fabric of harsh handle and inferior performance. Finally, curing of the dried fabric promotes the reaction of the chemicals together or with the cellulose. Stringent performance requirements often make the finishing operation the key step in the production of a material with a particular functionality, such as water repellence. Many chemical finishing methods cause visible changes in the colour of a dyed fabric, or modify some of the colour fastness properties. Such effects must be known in advance to avoid off-shade and sub-standard products unacceptable to the customer.

#### 1.17.1 Crease-Resist Finishes

Increase-resist finishing of cotton, the first step is padding the material with a solution containing a condensation polymer precursor and a suitable polymerization catalyst. Drying and curing in a tender frame follow this. On heating, the polymer precursor either reacts with hydroxyl groups in the cellulose to form crosslinks between adjacent polymer chains, or it polymerizes in the amorphous regions of the fibres. Crosslinks between the polymer chains considerably limit the movement of the cellulose molecules so that creasing is more difficult. Such reinforcement of the fibre network improves the dimensional stability of the cotton during washing.

Crease formation in a fabric is complex. It depends on the strength of intermolecular bonds between the polymer chains of the fibre. If these are strong, as in wool, the chains will extend on bending or stretching the fibres but the strong intermolecular bonds do not break. They will pull the polymer chains back to approximately their original positions once the fibres relax. The helical molecular structure of keratin imparts a natural elasticity to wool fibres that is also significant. Cotton fibres are much more rigid fibres. The hydrogen bonds between cellulose chains are relatively weak, particularly in the amorphous regions. On folding a cotton fabric, the hydrogen bonds break easily as the cellulose chains are stressed by bending of the fibres. New hydrogen bonds then form with the chains in their new positions and the new crease is stabilized. This is particularly easy if the cotton is wet. The crosslinks between the polymer chains, introduced during finishing, reinforce the cotton fibres and prevent the permanent displacement of the polymer chains when the fibres are stressed. It is therefore much more difficult for creases to form, or for the fabric to shrink on washing. The types of chemicals used for crease-resist finishing of cotton are condensation products of the urea-formaldehyde type capable of forming network polymers (Figure 1.6). Urea and formaldehyde react together to form a series of hydroxyl-methyl-ureas with up to four hydroxyl-methyl or methylol groups per urea molecule. When these types of functional compound are heated in the presence of an acid catalyst, they undergo condensation to a network polymer. The two main condensation reactions involved in polymer formation are shown in Scheme 1.1.

Figure 1.6 Condensation polymerisation of urea and formaldehyde

$$R-NHCH_2OH + R-NH_2 \longrightarrow R-NHCH_2NH-R + H_2O$$

$$2R-NHCH_2OH \longrightarrow R-NHCH_2OCH_2NH-R + H_2O$$

## Scheme 1.1

Urea-formaldehyde and the similar melamine-formaldehyde products do not give extensive crosslinking of the cellulose. They are still used today in the finishing of viscose materials and for some cotton goods. The thermal curing of these chemicals with an acidic catalyst considerably weakens the cellulose and makes the fabric more rigid. The decreased tear strength and the lower resistance to abrasion of treated fabrics are quite significant. (Table 1.2) and depend on the quantity of chemicals used and the severity of the curing operation. The

incorporation of polyester staple fibres into the yarns, along with the cotton, compensates somewhat for this loss of mechanical resistance.

Table 1.2 Influence of crease-resist finishing on fabric properties

	Untreated	5% Urea – CH <sub>2</sub> O 1:2/ Zn(NO <sub>3</sub> ) <sub>2</sub>	5% DMDHEU* MgCl <sub>2</sub>
Crease recovery angle (dry) ** % Loss of abrasion resistance % Loss of weft tear strength % Loss of weft tear strength after	150	260 42 38	265 25 22
chlorine bleaching and ironing		59	25

- \* See Figure 1.7
- \*\* Average for creasing across the warp and weft directions

Figure 1.7 Typical crosslinking agents for crease-resist fabrics of cellulosic fibres

If the crosslinking of the cotton is carried out under moist or even wet conditions, by a padbatch procedure followed by washing and drying, the tear and abrasion resistance and the wet crease recovery are higher than when using thermal curing, but the dry crease recovery is somewhat less. When crease-resist fabrics are bleached with hypo-chlorites, chlorine reacts with any residual amino groups in the condensation polymer. The chloro-amino groups produced will decompose on heating under humid conditions, for example on ironing, liberating hypochlorous acid, a strong oxidizing agent. Aldehyde groups in the cellulose reduce this to give hydrochloric acid. The result is severe de-polymerisation of the cellulose, yellowing and weakening of the fabric (Scheme 1.2).

Resin—NH
$$_2$$
 + Cl $_2$  — Resin—NHCl + HCl

Resin—NHCl + H $_2$ O — Resin—NH $_2$  + HOCl

2HOCl + R—CHO — Cl $_2$  + R—CO $_2$ H + H $_2$ O

Cl $_2$  + H $_2$ O — HOCl + HCl

#### Scheme 1.2

The newer types of methylol-amino crease-resist chemicals for cotton are mixtures of cyclic methylol-urea derivatives. They are mainly bifunctional compounds that give increased crosslinking of the cellulose and less polymer formation in the voids of the fibres. They have much lower chlorine retention, their tertiary amino groups being less nucleophilic. Considerable care, however, is still required to avoid excessive weakening of the cellulose fibres. Figure 1.7 shows the structures of some modern crease-resist reactants.

Table 1.3 gives the recipe of a typical crease-resist finishing formula for padding a 50/50 cotton/ polyester fabric indicating the role of each particular chemical. Many of these finishing treatments give fabrics that are noticeably more rigid. The solutions used therefore often contain softening agents and lubricants, as well as additives to minimize the decrease in tear strength and abrasion resistance. Another preoccupation has been the tendency of treated cotton to liberate formaldehyde during use. This arises from the hydrolysis of the polymer crosslinks and the decomposition of residual methylol-amino groups (Scheme 1.3).

Table 1.3 Typical composition of a crease-resist finishing bath

Chemical	Amount in the bath solution %	Function
DMDHEU	6.0	Crosslinking agent
MgCl <sub>2</sub> .6H <sub>2</sub> O	2.0	Acid catalyst for DMDHEU
Ethyl silicate ester	0.5	Increases resistance to abrasion and tearing
Polyethylene glycol	0.5	Softening agent
Non-ionic surfactant	0.1	Wetting agent

### Scheme 1.3

For durable-press finishing, the fabric is impregnated with the same types of polymer or crosslinking precursors and dried without curing. Once the finished article has been assembled, it is hot pressed in the required shape, when crosslinking and polymerization occur stabilizing the form of the article. Formaldehyde generation is sometimes a problem if articles are stored between drying and curing. The crease-resist finishing of cotton and cotton/polyester fabrics is therefore often a compromise involving maximizing the desirable properties of crease resistance and recovery and dimensional stability, while maintaining adequate mechanical and tactile properties of the fabric along with minimum chlorine retention and formaldehyde release. Some effects of crease resist finishing on fabric properties are given in Table 1.2. Soil-release, flame-retardant and water-repellent chemicals for fabrics containing cellulosic fibres are invariably combined with the crosslinking agents used for crease resistance. The padding bath compositions are then quite complex. The greater the number of chemicals in the bath, the higher the risk of their incompatibility.

## 1.17.2 Water-Repellent Finishes

A film of wax such as paraffin, or of a hydrophobic insoluble soap such as aluminium stearate, on the surface of a fabric containing hydrophilic fibres such as cotton will make it water-repellent. This can be achieved by treating the fabric with an emulsion of paraffin. If the wax particles in the emulsion have an anionic emulsifying agent adsorbed onto their surface, addition of a polyvalent cation such as Al3+ or Zr3+ helps to reduce the cotton's negative

surface charge so that the paraffin particles are not repelled by it. The resistance of the water-repellent finish to repeated washing is improved by combination of the paraffin with a crease-resist resin. Insoluble soaps are usually applied by firstly impregnating the fabric with a soluble sodium soap. Treatment in a second bath containing a solution of an appropriate polyvalent metal salt such as Al3+ precipitates the hydrophobic soap on the fibre surfaces. A number of relatively permanent hydrophobic finishes involve reaction of the cellulose hydroxyl groups with acid chlorides, isocyanates, pyridinium salt derivatives (1, in Figure 1.8) or N-methylolamides of fatty acids (2). Some of these reactive chemicals need an organic solvent; others can be used in aqueous dispersion. These reactive chemicals give finishes that are resistant to washing and dry cleaning. Again, for cotton, they are often combined with crease-resist chemicals.

$$C_{17}H_{35}CONHCH_2$$
  $C_{17}H_{35}CONHCH_2OH$ 

Figure 1.8

A film of hydrophobic chemical on the fibre surfaces provides water repellence, but the fabric still retains a reasonable degree of air permeability as the yarn interstices are still not completely filled. Total water impermeability requires a coating of rubber or polyvinyl chloride on one face of the fabric. This drastically reduces the air permeability. Because of this, water impermeable coatings are used mainly for industrial applications rather than for clothing. The types of fabrics used for water-repellent finishes must have a smooth, compact construction with little space between the yarns. The fabric must be also be free of hydrophilic surface active agents that decrease the adhesion of the hydrophobic finish to the fibre surfaces. Water repellence increases with an increase in the proportion of hydrophobic fibres in the fabric but the use of cotton is not excluded. In fact, cotton/nylon and cotton/polyester blends are popular for rainwear fabrics. The swelling of the cotton fibres that occurs on water absorption helps to close up the yarn assemblies and prevents water penetration.

## 1.17.3 Silicone Finishes

Modern water-repellent chemicals are often emulsions of poly-dimethyl-slogans. These chemicals spread rapidly all over the fibre surface during impregnation and, on curing, form a layer of resistant hydrophobic liquid polymer on the fibre surfaces. These chemicals not only promote water repellence but also give the fabric a soft, smooth handle.

Hydrolysis of chloro-methyl-silanes and polymerization of the silanols produced gives polyslogans (Figure 1.9). Dichloro-dimethyl-silane hydrolyses to a diol that gives a linear condensation polymer, whereas trichloro-methyl-silane gives a triol and thus leads to a network polymer. Trimethyl-silanol from chlorotrimethyl-silane will not polymerize but will block a growing poly-siloxane chain. By varying the relative proportions of these three chemicals, the molecular weight and properties of the silicone polymer produced can be varied. The amount of methyl-silanetriol controls the degree of polymer crosslinking while that of the trimethyl-silanol controls the chain length. Polysiloxanes, commonly called silicones, are available as liquids, rubbers and solids. They have many industrial uses as lubricants and elastomers.

Figure 1.9 Polymerisation of methylsilanols

The silicones used in textile finishing are partially polymerized methylsilanols in an organic solvent or aqueous dispersion. They contain an organometallic catalyst to promote additional polymerization on the fibre surface during curing. The water repellence of the treated fabric depends on the orientation of the polysiloxane molecule on the fibre surface. The polar oxygen atoms along the silicone chain are oriented towards the polar fibre and the methyl groups away from it. This is assisted by the incorporation of zirconium or titanium compounds. In this way,

the surface of the fibres presents an array of hydrophobic methyl groups that provide the water repellence and smooth handle. The adhesion of the silicone depends on the fibre surface being free of surface-active wetting agents so that good rinsing of the fabric after the previous treatment is essential. Since the liquid polysiloxane film does not have strong adhesion to the surface, the finish is not very resistant to laundering and dry cleaning. Reactive silicone finishes that undergo more extensive poly-condensation to a cross-linked polymer are more durable. One type of reactive polysiloxane has methyl-hydroxy-siloxane units (Figure 1.10). The reactive silicon–hydrogen bond reacts with water to produce hydrogen and a silanol group that then undergoes further poly-condensation resulting in chain crosslinking. Because hydrogen is generated, good ventilation is necessary during use and for storage of drums containing the silicone emulsion. A mixture of the silicone dispersion and organometallic catalyst is padded onto the fabric. This is then cured to cause the desired reactions. The resulting network polymer film on the fibre surface is resistant to laundering and dry cleaning while imparting water repellence and a soft handle to the fabric.

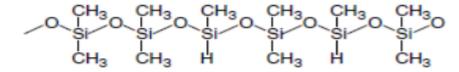


Figure 1.10 Reactive polysiloxanes

Another type of reactive silicone finish depends on crosslinking a polysiloxane by means of reaction with methylsilanetriol generated by hydrolysis of its methyl ether. This type of crosslink is longer than those formed by condensation of hydroxyl groups in a polysiloxane. Polysiloxanes with short crosslinks are used mainly for water-repellent finishes. The more elastic types, with longer methylsilanetriol generated crosslinks, are softeners. In addition, a range of polysiloxanes with other functional groups is now available (Figure 1.11). These include polysiloxanes with amino or epoxy groups, the numbers of such groups depending upon the particular effect required. Softening agents. Polysiloxanes with a grafted polyoxyethylene side chain are relatively hydrophilic. If the polyoxyethylene chain is long enough, the polymer can even be water-soluble. When used with a methylolamino crease-resist chemical, the polyoxyethylene chain can bond to the fabric and imparts a soft hydrophilic surface.

Figure 1.11 Functionalised polysiloxanes

The stability of the polysiloxane emulsions is critical. Separation of the polymer may cause uneven water-repellent deposits on the fabric surface and equipment. This also applies to other types of polymer emulsions. Polysiloxane emulsion technology has developed to the point that some micro-emulsions of these products can even be applied in a jet dyeing machine where the shear forces generated would rapidly crack most polymer emulsions. The application of any type of hydrophobic finish on the fibre surface that requires thermal curing may result in decreased rubbing fastness of fabric containing polyester fibres dyed with disperse dyes. On curing, the disperse dyes migrate from the polyester fibres into the layer of hydrophobic chemical on the fibre surface, in which they are very soluble. The concentration of dye in this surface layer can become high enough that it is easily transferred to white or pale coloured adjacent fabric by abrasion.

# 1.17.4 Anti-Soil and Soil-Release Finishes

Soil-release finishes allow the easy removal of dirt and oil picked up by a fabric during use. Most types of dirt are hydrophobic and adhere well to hydrophobic synthetic fibres such as polyester and nylon. The ready accumulation of static electric charge by synthetic fibres also leads to more soiling than for more hydrophilic fibres. Static charge is more readily dissipated by cotton because it absorbs more water from the atmosphere than a typical synthetic fibre. Stain or soil repellent finishes invariably contain a fluoro-polymer, possibly in combination with a silicone polymer. The 'Scotch guard' finish is a typical example. The chemicals are applied as an emulsion followed by drying and curing. They leave a thin film of polymer on the fibre surfaces of very low adhesive capability so that dirt does not stick to the film. This is a key characteristic of fluoro-polymers. Polytetrafluoroethylene exposes an array of fluorine atoms along the polymer chain. The fluorine atom is the most electronegative atom known and other atoms do not form even weak bonds with it.

Soil-release and soil anti-re-deposition chemicals facilitate the release of dirt already on the fibre surface during washing. They are usually hydrophilic anionic polymers often based on poly-acrylic acid derivatives. A film of such a hydrophilic polymer on the fibre surface does not strongly attract hydrophobic dirt so that it is more readily emulsified by the detergent solution during washing. The negative charges of the carboxylate groups in the poly-acrylic acid film repel the dirt particles and prevent any re-deposition (Figure 1.12). This occurs because the dirt particles also have a negative charge from the detergent molecules adsorbed on their surface.

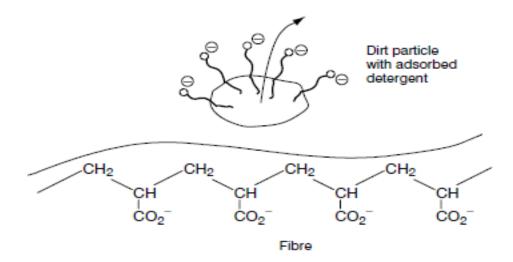


Figure 1.12 Function of a polyacrylate soil-release finish

### 1.17.5 Flame-retardant finishes

The majority of organic polymers are sensitive to heat. At high temperatures, in the range 100 to 300 °C, they will either melt or decompose. Decomposition will usually lead to the formation of flammable gases. Below the decomposition temperature, the majority of synthetic fibres will first melt on exposure to heat.

Drops of molten polymer can cause serious burns. For some uses, such as theatre curtains, protective clothing and children's nightwear, a fabric must be nonflammable. Cellulose chars and burns quite readily. Cotton or regenerated cellulose materials pose a serious fire hazard because the flammable gases generated on combustion permit rapid propagation of flames. Because of more stringent government regulations, the use of flame-retardant finishes is now common for cotton fabrics for children's nightwear. The processes used to decrease the flammability of cellulose are very similar to those used for crease-resist finishes but include other chemicals based on phosphorous or halogenated derivatives that impart the required flame resistance along with crease resistance. Careful selection of chemicals and curing

conditions is required to avoid excessive losses in fabric strength and a harsh handle. Minimizing flammability involves three major principles:

- (1) Decomposition of the chemical on heating to give a non-combustible gas that envelops the fibres and excludes oxygen. Ammonium salts that release ammonia on heating are typical examples, but they are water-soluble and not resistant to washing;
- (2) Decomposition on heating to produce a deposit on the fibre surface that blocks the release of combustible gases. They may also catalyze the dehydration of cellulose and minimize gas release. Many of the phosphorous or boron-based finishes probably work on this principle;
- (3) Decomposition to generate free radicals that combine with the free radicals needed for flame propagation. Organo-bromine compounds decompose to give free bromine atoms that are effective in this way.

For decreasing the flammability of cellulosic fibres, it is usual to apply a chemical finish to the material. Some artificially-made fibres contain additives that reduce their flammability. These are either reactive chemicals incorporated into the polymer chains during polymerization, or chemicals added to the polymer liquid or solution before filament spinning. There are, of course, a number of artificially made fibres with outstanding heat and flame resistance such as poly-aramids (Kevlar, Nomex) and polybenz-imidazoles.

When flame-resistant material will not be washed during use, adequate protection is possible by impregnating with a soluble salt solution and drying. Common chemicals include ammonium carbonate and borax. Ammonium carbonate decomposes on heating to form ammonia, water and carbon dioxide.

The release of these gases prevents oxygen from reaching the fibres. Borax coats the fibres with boron oxide and reduces the evolution of combustible gases. To increase washing fastness, various insoluble salts such as metal oxides can be precipitated in the fibres. Modern finishes for flame-resistant cotton and viscose use reactive chemicals that are combined with crease-resist resins. A solution or dispersion of the mixture of chemicals is padded onto the material. This is then dried, cured and possibly washed. This type of chemical finishing of cellulosic goods is extremely severe and invariably has an influence on the dyes present in the fibres. Both the colour and the colour fastness properties may be affected. These types of finishes often give products with a harsh handle and the use of softening agents is common. Alternatively, to avoid stiffening of the fabric, the chemicals can be applied using a cold pad—

batch method. They give a flame resistant finish with good washing fastness. Many are known by their commercial names (Figure 1.13)

Figure 1.13 Typical flame-retardant chemicals

## 1.18 Other Types of Finishing Chemicals

Bacteria or fungi (mildew) will grow on the surface of many fibres particularly under warm damp conditions. This can cause coloured spots that are extremely difficult to remove and has an unpleasant, mouldy odour. The bacteria may even feed on natural polymers causing considerable damage to the fibres. When the fabric will be stored for more than a few days, it will normally be rinsed with a dilute solution of an anti-bacterial agent before drying. These agents are usually organic quaternary ammonium salts, such as hexa-decyl-pyridinium bromide (3, in Figure 1.14, also commonly called acetyl pyridinium bromide), or toxic organo-halogen compounds such as chloro-phenols (4). Permanent moth-proofing of wool involves finishing by a dyeing process with colourless anionic organo-halogen compounds that behave like acid dyes.

Figure 1.14

Softening agents are widely used in textile finishing. The goods are usually rinsed in a solution of the chemical and then dried. If not polysiloxanes, they are often cheaper surface active chemicals, or closely related derivatives, whose molecules have long hydrocarbon segments. They may be anionic, cationic or nonionic. They lubricate the fibre surface and reduce friction. In addition, many such ionic chemicals minimize build-up of static electricity.

During the finishing of many fabrics, solutions or dispersions of chemicals may be dried into the material simply to increase its weight, improving the draping characteristics. The classic example of this is weighted silk. Their presence often increases the fabric stiffness. The objectives of applying such chemicals can be quite varied and the finish is often not permanent.

### 1.19 Conclusion

Chemical finishing is always an important component of textile processing because it makes textile materials marketable and user-friendly. In recent years, there has been a growing trend towards 'high-tech' textile products. As the use of high performance textiles has grown, the need for chemical finishes to provide the fabric properties required in these special applications has grown accordingly.

One strong future trend is the continuing incorporation of a user-friendly computer control into textile finishing equipment. Touchscreen controls with easy-to-understand icons have been installed on many machines, and more versatile microprocessors are being utilized, not only to monitor machine and process parameters, but also to control the process through closed feedback loops. A feedback loop is the path by which some of the output of a circuit, system, or device is returned to the input. Additionally, production data are being recorded and stored so that they can be recalled for later use. A major trend is to provide improved and more consistent quality in finished yams, fabrics, and garments. Machinery manufacturers recognize the need for ongoing competitiveness, and they are supplying their customers with the means to increase productivity while reducing overhead costs. The growing popularity of short manufacturing runs requires finishing machinery that can provide the desired fabric properties over a wider range of fabrics. Softening agents. Polysiloxanes with a grafted polyoxyethylene side chain are relatively hydrophilic. If the polyoxyethylene chain is long enough, the polymer can even be water-soluble. When used with a methylolamino crease-resist chemical, the polyoxyethylene chain can bond to the fabric and imparts a soft hydrophilic surface.

The stability of the polysiloxane emulsions is critical. Separation of the polymer may cause uneven water-repellent deposits on the fabric surface and equipment. This also applies to other

types of polymer emulsions. Polysiloxane emulsion technology has developed to the point that some micro-emulsions of these products can even be applied in a jet dyeing machine where the shear forces generated would rapidly crack most polymer emulsions. The application of any type of hydrophobic finish on the fibre surface that requires thermal curing may result in decreased rubbing fastness of fabric containing polyester fibres dyed with disperse dyes. On curing, the disperse dyes migrate from the polyester fibres into the layer of hydrophobic chemical on the fibre surface, in which they are very soluble. The concentration of dye in this surface layer can become high enough that it is easily transferred to white or pale coloured adjacent fabric by abrasion.

# **Chapter 2 Mechanical Finishing**

### 2.1 Introduction

The principal object of finishing fabric is to confer a more pleasing appearance and handle or to render the fabric more suitable for a particular end use. It has long been known that simple physical or mechanical treatments can change the appearance and properties of textile fabrics significantly. As little or no water is used during the processes, mechanical finishes are often termed as 'dry finish'. The mechanical treatments are significantly affected by the extent of heat and pressure applied, the moisture content of the material during the treatments and on the pretreatment of the fabric with gum and starchy products. The traditional batch wise mechanical finishes have now been replaced by continuous treatments capable of finishing at high speed. Moreover, better control of machine parameters is possible in continuous state-of-the art finishing machinery and they assure that the fabrics being finished are consistently to close tolerances. The surface characteristics of fabrics can be altered by a variety of techniques. The surface modifications aim to improve smoothness, roughness, lustre, adhesion, dyeability and wettability, in addition to removing creases and wrinkles.

#### 2.2 Calenders

Calendering is a type of mechanical levelling and segmenting process for 'finishing' fabrics or webs to produce a special effect, namely flattening, lustre, compacting, glazing, moiré, Schreiner, smoothing, texturing and other embossed patterns by passing in open width between two adjacent rollers kept under pressure. The passage of material (full-width piece goods) between two rollers is called nip. The use of calenders to produce flat, compact and polished fabric is very still popular. From the beginning of fabric manufacturing, it was observed that the application of pressure by a simple iron or press could alter the properties of fabric. The alteration in properties depends on the ability of the fabric to be mechanically changed. The use of calender is an important technique in finishing of cotton, linen rayon and silk materials. On the other hand, synthetic fibres react to mechanical deformation, but they require the presence of heat to defeat the physical and thermal memory of the fibres. The object of normal calendering is to flatten yarns, to close interlacements in fabric and to impart a lustrous, smooth feel to the fabric.

## 2.2.1 Rolling Calender

The normal type of calender is sometimes termed a rolling calender or roller calender. The function of a rolling calender is to provide a smooth or glossy fabric surface as well as to

improve the handle. The basic mechanical action of this type of calender is to cause the fibres inside the textile material not only to reshape but also possibly to flatten or deform. It also causes the fibres to nestle or more tightly stack around one another. Fundamentally, the calenders consist of a series of heavy rollers or bowls mounted vertically in a robust frame. Calenders with 3, 4, 5, 6, 7, 10 or 11 may be used according to the output and type of finish required. Three bowl calenders and seven-bowl universal calenders are the most popular. Normally, the unit comes as a three-roll calender with alternate steel and filled rolls, although it may also come with two, four or five rolls. The main roll, which is the top steel roll, is driven by a variable speed motor, either directly or through a roller chain drive, while the intermediate filled roll can be driven with off nip drive. When required, the steel roll can be heated by gas, hot oil, electric or steam up to 210°C. The metal roll is made of fine cast iron or high-quality steel, which is often case hardened and highly polished. Chrome or nickel-plated steel bowls are used where a high-gloss finish is required. When fabric is passed between two metal rolls, the fabric may be damaged, particularly in the case of thin woven fabric. Therefore most calenders consist of a hard metal roller and a softer roller. The softer nonmetal bowl is usually termed as 'calender bowl'. The calender bowls are made out of variety of materials, with each bowl manufacturer offering a specialized bowl covering. The usual substances used are cotton paper, wool paper or a mixture of the two. These machines are often heavily engineered in reinforced concrete to a depth of 4 ft. The torque and loading at a high power (e.g. 60 hp) with a high ratio of speed reduction are considerable.

One major problem is the potential damage of the calender bowl due to improper sewing techniques where more than one layer of fabric present. The hardness of the calender bowl should be high enough so that the required finish is achieved without excessive pressure loading, too high temperature or low production speed (high nip dwell). The surface hardness is normally kept at 75–90 degrees Shore hardness. Calendering is a high-speed ironing process that primarily imparts lustre and is usually the final treatment for the fabrics in the finishing sequence. The basic principle of calendering is to expose the cloth to the combined effect of moisture, heat and pressure until the fabric acquires a very smooth and light-reflecting surface with a good lustre. The calendering effect on the fabric is usually temporary and disappears after the first washing. Semi-permanent lustre is sometimes achieved by padding fabric in a sparingly soluble polyvinyl acetate emulsion before calendering, where the solution acts as a binding agent. After scouring, bleaching, mercerizing and printing, the fabric is dried so that it can transform into its true shape and dimensions. However, it is likely to end up in a form

which is less lustrous, because the threads are left highly crimped or wavy so that the fabric surface is not smooth and flat. To become highly lustrous, the fabric surface should be perfectly flat with the individual fibres as parallel to each other as possible.

To increase its lustre, the fabric must be flattened and smoothed. This process is usually referred to as Schreiner calender (Section 2.2.4) when substantial improvement in lustre is obtained by passing the fabric between rollers. In ordinary calendering, the two bowls are run together at the same peripheral speeds so that there is no slippage between them. Under these conditions, the surface of the fabric is simply flattened to the extent that the pressure between the bowls and the plasticity of the cotton fibres will allow. This can give a considerable increase in lustre. However, this increase can be made even greater by first impregnating the fabric with waxy and starchy substances, which can assist by filling up inequalities in the fabric surface and by promoting a certain degree of polishing. When the maximum increase of lustre is desired, it is arranged by suitable gearing for one bowl to rotate somewhat faster than the other. This is called friction calendering, and under such circumstances, there is a slip between the bowl and the fabric which is equivalent to an ironing action. If the steel bowl is quite hot and the fabric suitably damp, there is also a very high increase in lustre. The polishing action closes up the pores of the fabric to make it appear more solid and highly lustrous. By simple and friction calendering, the cotton fabric can achieve a greater lustre than that obtained by mercerization. In mercerized fabric, the threads appear clearly and are round and spaced evenly according to the weave. On the other hand, calendering results in the flattening of threads and reducing space between them. In spite of a somewhat lower lustre, however, mercerized fabric does appear to be of higher quality. Of course, mercerized cotton fabrics can be calendered to give them a higher lustre, and it is possible through calendering to control the increase of lustre more accurately.

A defect of a lustrous finish obtained by calendering is that it is not fast to washing. When the fabric is moistened, the cotton fibres swell and lose their flattened state so that during drying, the fabric reverts to its original appearance, with most of the added lustre having disappeared. By contrast, the lustre produced by mercerization is permanent and is unaffected by washing. The lustre produced by the calendering of a moderately moist fabric can be readily removed by light washing. However, a wash fast lustre is produced by the very hot calendering of the wet fabric so that drying takes place during the process. The lustre of the fabric is greatly increased if the individual fibres showing on the surface are parallel to each other. However, it is impossible to weave a fabric in which all of the fibres run parallel to each other and there is

no subsequent means to make them parallel. It would be possible to emboss the surface of the fabric with parallel lines spaced a distance apart that is about equal to the thickness of the cotton fibre and more or less running in the same direction as the majority of the fibres showing on the fabric surface. Such embossing would mostly cover up the irregular alignment of the fibres, resulting in a fabric surface where the numerous, closely spaced embossed lines would appear as a fabric with all of its fibres ideally parallel; thus the produced fabric would be one of very high lustre. The Schreinering process evolved and the Schreiner calender was designed on this principle. In order to fully flatten and smooth cotton fabric, it is necessary for the cotton fibres to be brought into a fairly plastic state. Cotton fibres are more plastic when they are hot and moist, thus the most favourable conditions for calendering are achieved by first conditioning or dampening the fabric and then using a calender with hot pressurized rollers. Prior to calendering, it is often customary to stretch the conditioned fabric to bring it to the required width and to remove any wrinkles and creases. Fundamentally, the calender consists of a series of heavy rollers or bowls mounted vertically in a robust frame. Many types of calenders are available, featuring anywhere from 2 to 11 bowls or rollers. With a simple two-bowl calender, the fabric can pass through only one nip in its passage, but with a multiwall calendar, the fabric can pass in succession through several nips and is more completely treated as a result. The bowls are made of polished steel or compressed paper or cotton. The metal bowls are usually made of chilled iron with a hard and highly polished surface. The bowls are generally hollow and often are made of close-grained cast iron, allowing them to be heated. For each pair of rollers running against each other, one should be softer than the other so that if there is any inequality of thickness in the fabric, the soft bowl will compress to accommodate this extra thickness. If two steel bowls run together, the fabric may be damaged or cut at the extra thick portion, thus it is usual to run a steel bowl against a hard roller made of rubber, paper or fabric.

If the calendering is to be carried out hot, then obviously the soft bowl must be of paper or cotton and not of rubber or any other material adversely affected by high temperatures. In most of the cases, all metal bowls are geared together in order to prevent slippage. The three mechanical operating parameters of speed (or dwell time in nip), temperature and pressure on the adjacent rolls (pounds per lineal inch, or PLI) must be controlled.

When a loading force is applied to the end of the calender bowls, they tend to deflect, resulting in a decreased nip pressure in the middle. The obvious remedy is to have a bowl whose diameter varies across the length with a greater diameter in the middle. However, the correct setting only applies to one specific loading. Another solution to bowl deflection involves 'skew' positioning

of the bowl. The temperature can vary from ambient for a light smoothing finish to 190°C for a full lustrous finish. The heating of the metal bowl may be achieved by gas, electric, thermic fluid or steam. Gas and electric heating need very accurate control, whereas steam and thermic fluid give level heating profiles. The production speed of the calender depends on many factors. The slower the speed, the longer the dwell of fabric in the nip, which will consequently have a greater effect on the fabric. Speed generally varies from very slow at about 5 m/min to very fast at a maximum of 75 m/min. It is difficult to specify all of the parameters involved in calendering, as there are many. A few important parameters are listed below:

- (1) Fabric structure—a more open structured fabric is affected more easily by calendering.
- (2) Nature of the fibre/yarn—a softer fibre or softer spun yarn is affected more easily.
- (3) Finish—pretreating the fabric with starch, gums and/or softeners distinctly enhance the finishing effect.
- (4) Moisture content—dry fabric will be less affected than conditioned fabric.
- (5) Roller temperature—synthetic fibres react to mechanical deformation in the presence of heat, and mechanical deformation of natural fibres are also enhanced by heat.
- (6) Calender speed—this determines dwell time in nip and consequently the extent of deformation.
- (7) Differential bowl speed—this determines the friction on the fabric's surface and consequently the fabric's lustre.
- (8) Nip pressure—high pressure gives a papery finish while reduced pressure results in a soft and thready appearance of the finished fabric.
- (9) Roller composition—the extent of the hardness and its variation on the roller surface decides the uniformity of a surface finish.

There are eight types of calendar finishing:

- (1) Simple finishing or swizzing
- (2) Chasing
- (3) Friction
- (4) Schreiner calender

- (5) Embossing calender
- (6) Moiré calender
- (7) Felt calender
- (8) Ciré calender

The seven-bowl universal calender is the most popular. A universal calender may be used to produce swizzing, chasing and friction finishes in the same machine, as shown in Fig. 2.1A—C, respectively. In the seven-bowl calender machine, the constructions of the bowls are different. The first bowl at the bottom (the fabric entry point) is made of close-grained iron. Bowls 3 and 6 from the bottom are made from chilled iron with a hard polished surface, while bowls 2, 4, 5 and 7 are made from compressed cotton. Friction calendaring may be done in a separate machine, too.

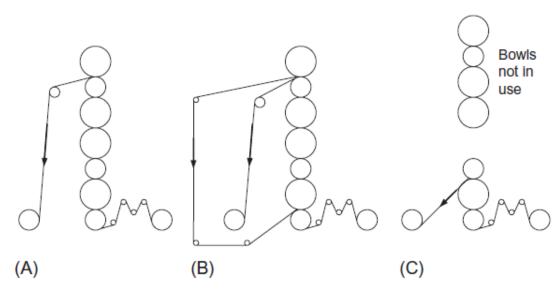


Fig. 2.1 Finishing with seven bowl calender. (A) Swizzing. (B) Chasing. (C) Friction finishing.

## 2.2.1.1 Swizzing or Swissing Finish

In this finishing technique, the fabric runs through the nips of several bowls with the same surface speed. As a result, the fabric achieves a lustrous paper-like finish depending on the number and composition of the bowls.

## 2.2.1.2 Chasing Finish

The fabric is passed through the nips of the calender, over the external rollers and back into the bottom nip of the calender. Multiple layers of fabric run through the nip resulting in a thready appearance with soft handle. This is mostly done for linen fabric.

### 2.2.1.3 Friction Finish

Generally, a three-bowl calender is used. One heated polished metal bowl runs at a surface speed of 1.5–2.0 times faster than the other two, resulting in a highly polished fabric surface with a high gloss. The bottom bowl is usually made of close-grained cast iron, and the middle one is made of cotton and has a greater diameter than the others to allow for wear. The seven-bowl calender may be used by detaching or lifting the top four rollers.

### 2.2.2 Friction Calender

For a maximum surface change, a friction or glazing calender is used. For an improved friction finish, a three-bowl friction calender may be used. The machine is generally heavier, and a gear system is used to drive the smooth metal bowl faster than the softer calender bowl. The main function of a friction calender is to polish cotton fabric surfaces to a very high gloss. The basic mechanical action of this type of calender is similar to that of a rolling calender, with the addition of a polishing action by a smooth heated metal roll passing across the fabric as it is being held against a fibre-type roll under moderate pressure. It is normally used on apparel fabrics of natural yarns, such as cotton, that are woven, as well as on linen drawing cloths. These fabrics must be very strong in order to withstand tremendous tension in the calendering nip.

A friction calender consists of a three-roll calender, and the intermediate roll has a filling of 100% heat-resistant cotton. The top roll is driven by a mechanical variable speed motor and roller chain, while the auxiliary roll is driven by a roller chain drive from top to bottom, usually with a 2.1 rotating in the same direction. A friction calender can operate at speeds up to 35 yards per minute; it has a nip loading of up to 2500 PLI, with the average being 1500 PLI.

Both the top and bottom roll are heated; the top roll is heated to 175–215°C and the bottom roll is heated to a maximum of 65–120°C. The unit consists of a jaw clutch from top to bottom roll to disengage the drive when the machine is used as a rolling calender.

### 2.2.3 Silk Calender

The silk finishing calender provides a smooth fabric surface, light lustre and improved handle of silk and other delicate fabrics. The basic mechanical action for this type of calender can be

the same as for the normal roller calender with the exception of special fibre rolls that are comparatively soft. The 78 Shore D fibre rolls will create a very wide nip between the rolls when loaded against a smaller diameter steel. This type of calender can process all types of fabrics, but it is used mostly for high content cotton and silk fabric. The calender speeds for silk finishing can reach up to 100 yards per minute, with a nip loading of 400–700 PLI. The wide nip flexes and releases the fabric, causing a disruption between the bond points, serving to soften the fabric as well as strengthen it by enhancing inter-fibre cohesion

## 2.2.4 Schreiner Calender

A silk-like appearance, especially with mercerized fabric, is achieved by passing over a metal bowl with engraved inclined lines (i.e. angles with warp or weft) of about 125–500/in. under very high pressure (about 10 tons). In order to obtain a silk-like lustre, it is necessary to produce a very large number of small reflecting surfaces distributed in several planes. The production of lustre by the Schreiner finish is therefore a method of embossing.



Fig. 2.2 Schreiner roller.

Though friction calender can produce lustrous, smooth and drapeable fabric, it gives a papery thin handle. Schreinering is a special type of embossing where a heated metal roller engraved with fine diagonal lines comes in contact with the fabric and presses those fine lines into its surface. With the correct cloth construction and the correct line direction of the engraving, a soft lustrous handle can be achieved because of the regular reflection from the fabric surface along the embossed lines. This calendering is quite popular for sateen fabric. When the fabric is passed in contact with such rollers under pressure, the fabric texture is changed to a desired soft, lustrous and translucent due to changed light reflectance from fabric surface. The Schreiner roller is usually engraved with 260 lines per inch at a 26.5 degree angle and only 0.001 in. deep (Fig. 2.2). This pattern physically can consolidate the fibres by as much as 4–16%. Highly lustrous fabrics can be obtained by engraving 500 lines per inch at 20 degrees to the weft as recommended elsewhere. In principle, the angle of engraving should follow the approximate angle of the line of the twist of the yarn and also the direction of twist (for S and Z twists, the lines of engraving should incline to the left and right, respectively). Sometimes

vertical lines (i.e. perpendicular to the roller axis) are used for warp-faced fabrics and plain

square-weave fabrics irrespective of the S or Z twist. Such calenders can be used for natural,

synthetic and blended or coated fabrics that are both knit and woven or nonwoven. For knits, a

Schreiner calender provides improved handle and surface texture as well as more cover, while

for woven goods it offers more texture and drape. A Schreiner calender can operate at speeds

up to 30 yards a minute with a nip load normally at about 1200-1500 PLI with 1500 PLI

maximum.

If cotton is subjected to slight pressure, a low degree of lustre is obtained, whereas if the

pressure is great, the numerous small surfaces are merged and give no satisfactory lustre, only

a specular reflection, as with a mirror. In weft sateen, it is common to engrave the lines at an

angle of about 20 degrees to the weft in the direction of the twist in the yarn. For plain weave

fabric, the engraving must be coarser and will average about 150-200 lines per inch. The

number of lines determines the lustre and appearance of the finished fabric. They may be

engraved so as to have a rounded or sharp outline. Their depth in the bowl surface may be

limited because if they are too deep and sharp, they may cut the fibre and reduce the strength

of the fabric. For a Schreiner calender, the suggested process conditions are as follows:

(1) Moisture content of fabric: not less than standard regain, 9–15% for cotton, usually ensured

by pre-damping

(2) Temperature: 120–160°C

(3) Nip pressure: 3.5–5.0 bar

(4) Speed: 2-10 m/min

A Schreiner calender is usually a two- or three-roll machine, with the third roll added to smooth

the filled roll and reduce true crowning requirements. The fabric is passed through the nip

between the heated engraved roller and a filled roller. The engraving is so fine that the filled

bowl is not usually positively driven and will rotate only when it comes in contact with the

metal roll. A filled bowl can be used against different Schreiner lines. However, in true

embossing, a filled bowl, once impressed with a specific embossing design, can only be used

with that particular design.

The usual Schreiner calender is built with two bowls in an open frame. The upper bowl is made

from a special quality steel of high carbon content and engraved with the necessary number of

lines. When a large output is required, a three-bowl Schreiner calender is used; the engraved

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bowl is placed in the middle and two sets of fabric are passed through the upper and lower nips simultaneously.

The fabric to be finished must be thoroughly pretreated, meaning that it must first be properly singed, bleached and dyed, followed by mild softening and drying processes. The effect produced by Schreinering is not wash fast, as the impression of the fine line is disturbed and destroyed when fibres are swollen with water. Most schreinered materials are used for linings, which are not washed.

# 2.2.5 Embossing Calender

An embossing calender usually consists of two bowls; the top metal bowl is engraved with a suitable design and the softer composition bowl has a surface that accepts the embossing pattern. Embossing produces a raised relief design which is permanent on thermoplastic fibre but temporary on cotton.

For proper embossing, the filled bowl is first impressed with a specific, deeper, reversed image of the design on the steel roller. For the proper registration of an impression, the filled bowl should be driven at the same peripheral speed during the aforementioned process. These bowls are specially made with super resilient properties, and both rollers are often heated. The function of an embossing calender is to impart a texture or pattern on the surface of the fabric. It can be accomplished on all types of fabrics including woven, nonwoven and knitted cloth. The mechanical action is the same as that of the rolling calender, but with the addition of a pattern engraved upon the steel roll. A Schreiner calender is a specific kind of an embossing calender distinguished by its pattern. Embossing calenders can operate at speeds up to 50 yards per minute and have a maximum nip loading of 1500 PLI. Cotton and some cotton/synthetic nonwoven fabrics require that the pattern be such that it will 'break the memory' (the remaining effects of previous treatments) of the fibre through the use of pressure and a pattern that will displace the fibre into the desired shape. The embossing calenders are designed to reproduce certain effects for book cloth, imitation leather, furnishing cloths, etc. They are generally made with two or three bowls. Embossing calenders are two-roll machines using a forged steel top roll and a filled bottom roll with the filling of wool felt paper or resilient wool/cotton. A much lighter embossing calender has been devised for finishing of crêpes or pebbles. These types of fabrics are made from a highly twisted yarn and buckle or distort on swelling, producing a disturbance to the surface of the fabric that gives a broken appearance to the fabric. However, crêping is irregular, but the problem is solved by embossing or moulding the fabric in such a manner that a pattern is formed into which the contracting yarn is directed. A good specimen of pebbles from real silk crêpes are photographed and engraved on metal bowls; these patterns may be transferred to rayon or synthetic fabrics by the embossing process. Moisture, heat and pressure impart the pebble or crêpe pattern to the fabric as it passed between the engraved metal bowl and the large (nearly double in size) paper or cotton bowl. The embossing calenders were originally developed to produce imitation leather cloth and book cloth. However, the embossing bowls are still quite expensive and the embossing process is quite slow.

## 2.2.6 Moiré Calender

In moiré finishing, the roller is engraved with a watermark or wood grain pattern. The moiré effect resembles a watermarking effect; it is produced when the weave structure of a tightly woven fabric is distorted by the movement of very fine yarns due to surface pressure. This is possible only if the fibre being treated is capable of deform. The moiré style is in demand for cotton and dyed synthetic fibres used for curtains and wall coverings. Acetate and viscose fibres are more capable of showing the moiré effect than other fibres.

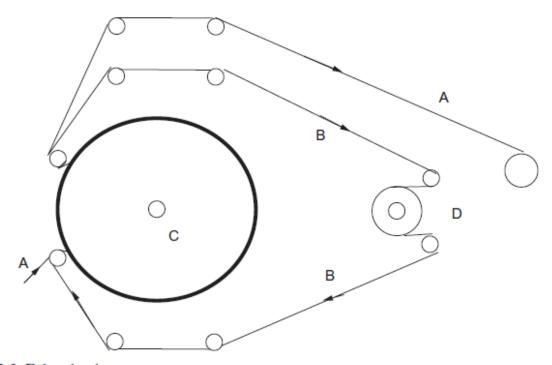


Fig. 2.3 Felt calender.

#### 2.2.7 Felt Calender

This is used for delicate fabrics like cotton mulmul, voiles, silks and synthetics, as well as knitted fabrics, especially tubular fabrics. The combined drying and finishing process is achieved by using an iron or stainless steel cylinder with a diameter of about six and a half feet,

moving together with an endless blanket while the fabric passes in between. Very little tension

is put on the fabric. Fig. 2.3 shows the line diagram of a felt calender. In the figure, A, B, C

and D represent the fabric, blanket, large heated cylinder and small cylinder, respectively, to

control tension in the blanket.

2.2.8 Ciré Calender

Ciréing provides a highly polished surface to fabric, making it appear wet. The friction roller

runs faster than that in the case of friction finishing. The ciré calender is used for glazing and

glossing fabric surfaces using a high temperature of 220°C and pressure as high as 1500 PLI.

Some porosity reduction and compaction is also obtained through this process. All types of

fabrics can be processed, but they are usually made of 100% synthetic fibres or high 75%

synthetic fibre content blends.

The units can operate between 30 and 35 yards a minute and have a maximum nip loading of

1500–3000 PLI. Depending on the width of the calender face with the rolls of steel on filled

roll and the filling is usually cotton, although nylon or urethane shells are also used at times.

The top or main steel roll is driven through a variable speed motor and a roller chain with an

optional drive to the auxiliary roll, which can be driven off the nip drive. The steel rolls can be

heated by gas or hot oil to a surface temperature between 175°C and 260°C. The approximate

production rates during the various calender finish processes are as follows:

For a swizzing and chasing finish: 3-bowl calender—60 yards per minute

5-bowl calender—70 yards per minute

7-bowl calender—90 yards per minute

For a friction calender: 35 yards per minute

For a Schreiner calender: 15–30 yards per minute

For a felt calender: 30–50 yards per minute

2.3 Sueding or Emerising

Suede is a type of leather. It is made from the underside of the skin, primarily lamb, although

goat, calf and deer are commonly used. Splits from thick hides of cow and deer are also sueded.

Its softness, thinness and pliability make suede suitable for clothing and other delicate uses; it

was originally used for women's gloves. Suede is also popular in making upholstery, shoes,

bags and other accessories, as well as linings for other leather products. Due to its textured

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nature and open pores, suede may become dirty and quickly absorb liquids. The term originates in France in around 1860, where the term 'gants de Suède' was used to refer to a particular type of soft gloves imported from Sweden. Over time, the word 'suede' came to refer to any type of soft leather with a smooth nap finish.

Fabrics are often manufactured with a brushed or napped finish to resemble suede. These products often provide a similar look and feel to suede, but have other advantages, such as increased liquid or stain resistance, and may appeal to consumers who prefer a non-animal product. Sueded silk, sueded cotton and similar sueded fabrics are brushed, sanded or chemically treated for extra softness. 'Suede' yarns are generally thick and plush.

The sueding or emerising process aims to recreate the natural suede feel using other materials, making them ideal for certain clothing applications such as ladies' bras and shoes as well as children's apparel. This process is also used to make upholstery, table and wall coverings. Emerising, also known as sueding or sanding, is a process in which fabric in open width is passed over one or more rotating emery-covered rollers to produce a suede like finish. Woven, knitted and laminated fabrics can be emerised; the surface appearance, texture and handle of the treated fabric will be modified according to the emerising conditions. The handle is much softer after emerising and much more enhanced in the case of fine micro-fibres (<1 dtex per filament). Cotton denim fabric can be given a suede-like finish, while amino-silicone-treated polyester and nylon microfilament fabrics can take up a soft, peach-like finish. The selection of fabric for emerising is very important. The weft yarn contributes the most towards surface fibre development. A tight plan weave is difficult to suede than a 2/1 twill or 3/1 twill, and so long weft float is helpful. Finer microfilament fabrics produce a high surface cover, and the handle is softer due to the lower resiliency of the fibre. Tight grey fabrics as well as dyed ones can be subjected to the sueding process; the cloth to be emerised must be completely free of any finishing resin (unless otherwise required) or adhesive substance remaining on the fabric surface after de-sizing. Emerising a dyed fabric can result in slight unevenness and stripiness, and the colour dust generated by emerising may cause contamination. The sueding process reduces the mechanic and dynamometric resistance of the fabric, thus making it more subject to tearing and seaming. The sueding machine consists of rotating rollers coated with abrasive paper, which emerise the cloth and produce somewhat of a marked effect depending upon the pressure exerted on the fabric by the abrasive rollers. The emery-covered rollers function as a cutting tool, trimming the protruding fibres to produce a very short velvet-like pile or nap. The effect on the fabric may be finer or coarser depending on the emery grade (or grain) size, which

is determined by the average grit size glued to the backing. Common sizes are, from coarse to fine: 40, 46, 54, 60, 70, 80, 90, 100, 120, 180, 220, 320, F and FF. A 46- or 54-grade cloth is used on roughly filed work, while 220- to 320-grit cloth will give a good polish (Oberg et al., 2000). The relatively coarse grades of 80–100 allow weft yarns to be caught and lifted by the emery-covered rollers. The coarse grain produces long, dense piles. A grain size of 280–320 may produce a short dense nap on lightweight ladies' outerwear of 100–180 g/m2. For polyester and nylon micro-fibres, the grain size may be increased to 400–600 for emerising. A higher grain size may give a polishing finish instead of emerising. The abrasive paper used can vary according to the desired sueding degree and must be replaced after a given number of operating hours, or when it stops sueding properly. In some cases, it is possible to also use metal rollers, with their surfaces coated with uneven and rough grains, or pumice rollers to perform an excellent sueding action on either dry or wet fabrics. For a very superficial sueding, the natural abrasive power of pumice can be applied with successful results. There are two types of emerising machines: single roller and multi-roller.

# 2.3.1 Single-Roller Emerising

A single-roller emerising machine consists of one metal roller with an abrasive covering (occasionally cooled by water circulation to prevent expansion by heat created during abrasion) and one rubber-covered pressure roller; the latter is micro-adjustable. The abrasion and cutting takes place in the nip formed between the pressure roller and the abrasive roller. Single-roller machines are less productive, typically moving at 7.5 m/min on micro-fibre fabric. Fabric imperfections like slubs or knots can lead to fabric damage and holes. Thicker selvedges are over-treated on single-roller machines, but run they can run on a multi-roller machine without problem. The directional pile effect may be developed only on a single-roller machine. A rubber-coated pressure roller may be replaced by an engraved roller to produce patterns on the sueded fabric. The machine also incorporates a self-cleaning dust exhaust and collection system.

# 2.3.2 Multi Roller Emerising

This most common and versatile emerising machine consists of four to eight independently driven emery paper-covered rollers of various grades (Fig. 2.4). The rollers in multi-roller machines may be cylindrical or of a slatted design; the later type exerts more extreme mechanical action on the weft yarn. Silk, jacquard and other delicate fabrics are treated only with cylindrical rollers. The rollers turn with or against the direction of the fabric run. Generally, the first and third rollers run in the opposite direction of the fabric passage to

intensify weft yarns on the surface, while others run in the same direction to produce the nap. Care should be taken to ensure that a loss in fabric strength is within tolerance range. Precise tension control is required to transport the fabric through the machine at an open width without folds or fluttering.

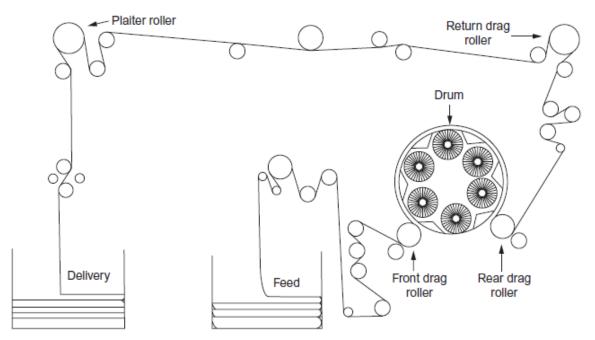


Fig. 2.4 Fabric path in an emerising machine.

The emerised effect is dependent on the mechanical action on the fabric which in turn depends on:

- (1) The number of rollers
- (2) The direction of the rollers' rotation
- (3) The fabric tension
- (4) The fabric speed
- (5) The grade of the emery paper

Typical operating speeds as reported (Heywood, 2009) are as follows:

(1) Micro-fibre-woven fabric: 12–15 m/min

(2) Spun yarn-woven fabric: 15–25 m/min

(3) Knitted fabric: 10-20 m/min

The drawback of the multi-roller machine is that some yarn knots may miss without emerising. The machine is much more versatile as different rollers may carry different emery paper and run in different directions.

# 2.4 Raising or Napping

Raising on cylinder machines is considered to be a plucking action by the card wire of the raising rollers on the fibres of the weft yarns in the cloth. The rate of raising, or the amount of nap produced under given conditions, depends on the ease with which the ends of the fibres can be raised from the weft yams. Many factors affect the raisability of a cloth, in particular, the twist factor of the weft and the frictional properties of its fibres. Increasing the twist factor of the weft reduces the rate of raising. However, much more important is the effect of the twist factor on the uniformity of the nap, which deteriorates steadily as the twist factor is increased. For cotton wefts in flannelette and winceyette types of cloth, the twist factor should not be much above 3.0. The ease with which fibre ends are plucked from the weft yarn depends on the presence of a lubricant, either natural (e.g. cotton wax) or one that is added during preparation. The characteristics of the weft fibre, particularly the fibre's length and weight, have a pronounced effect on the ease of raising and on the quality of the nap produced. Close cloths are more difficult to raise than open ones, and a  $2 \times 2$  twill structure is easier to raise than a plain one. The raising inevitably causes a loss in strength of the cloth weft-way. Consequently, cloths for raising are usually constructed to be much stronger weft-way than warp-way before raising, so that they can be approximately square in strength after raising. Flannelettes, for example, are approximately twice as strong weft-way before raising and lose up to half this strength during raising. Napping is the finishing process that raises the fibres on a fabric to produce a mat of fibre ends, or nap. It may be used on knit or woven textiles made of staple fibres, such as wool and cotton, or with fibres cut to staple length and spun into yarns such as silk, rayon and polyester. Napped fabrics are usually made with loosely spun yarns in the filling direction (weft) so that the fibres can easily be pulled out to form the nap. The hand raising of woollen goods with teasels is being done at least since Roman times. The first real advance came with the invention of the double-action card wire raising machine by Grosselin prior to 1870. No fundamental change in the process has since taken place, and all modifications have been in the nature of refinements.

Historically, napping was done with teasels, which are the flower heads of thistle- like plants that have many sharp, hook-like ends. Dipsacus, native to Europe, Asia and northern Africa, is a genus of flowering plant in the family Caprifoliaceae. The members of this genus are known

as teasel, teazel or teazle. Teasels are easily identified by their prickly stem and leaves, in addition to the inflorescence of purple, dark pink or lavender flowers that form a head at the end of the stem(s). Except in the case of some fine wools that are still napped by hand, napping in the early 2000s was executed with machines that mimicked the early process. These napping machines involve feeding the fabric through rollers that are covered with heavy fabric that is embedded with small, brush-like wires.

Napping can be done on one or both sides of the fabric. The technique can improve durability, hide defects or obscure the weave of the cloth. On the other hand, napped fabrics can also have increased pilling, abrade more easily even with care or flatten with wear. Common fabrics that are napped are wool and cotton flannel, flannel-back satin, polyester fleece, flannelette and outing flannel. Sueded fabrics are also napped through a process that includes an additional step to shear the nap close to the surface of the fabric to produce a smooth, soft finish. Since the 15th century, the term nap generally refers to a special pile given to cloth. The term pile refers to raised fibres that are there on purpose, rather than as a byproduct of producing the cloth. In this case, the nap is woven into the cloth, often by weaving loops into the fabric, which can then be cut or left intact. Carpets, rugs, velvet, velour and velveteen are all made by interlacing a secondary yarn through woven cloth, thus creating a nap or pile.

During a raising or napping finishing process, the fabric surface is raised by passing over rapidly revolving cylinders covered with metal points or teasel burrs, thereby a hairy surface can be given to both the face and the back of the cloth, thereby imparting hairiness, softness and warmth, as in flannelette. Outing, flannel and wool broadcloth derive their feathery appearance by raising. Napping is also used for certain knit goods, blankets and other fabrics with a raised surface. The raising process is a very old technique; some paintings found in Pompeii show that the raising of textiles was known to Romans. Raising is particularly suitable for wool and cotton fabrics; it gives a fuzzy surface by abrading the cloth and pulling the fibre ends to the surface. During the last few years, this process has also been applied to polyester/viscose blends and acrylic fabrics. Raising is done after shearing and cropping, dyeing and drying. Raising enhances the resistance of the textile material to atmospheric agents by improving thermal insulation and warmth provided by the insulating air cells in the nap. The fuzzy surface is created by pulling the fibre end out of the yarns by means of metal needles provided with hooks shelled into the rollers that scrape the fabric surface. The ends of the needles protruding from the rollers are 45 degrees hooks; their thickness and length can vary

and they are fitted in a special rubber belt that is spiral wound on the raising rollers. A both side raising machine is shown in Fig. 2.5.

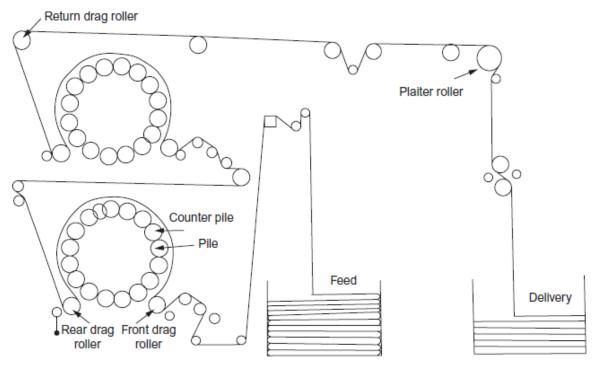


Fig. 2.5 Fabric path in a bothside raising machine.

Most modern raising machines are made of two opposite types of rollers, a roller with hooks directed towards the fabric feed direction (pile roller) and a roller with the hooks fitted in the opposite direction (counterpile roller). A typical raising machine may have 24 rollers, 12 of each type mounted alternately. Each set of rollers is independently driven. The machine consists of a drum or cylinder around the surface of which are mounted with wire-covered rollers. The fabric is transported over the wire points, which penetrate the cloth surface to a depth depending on the relative speed of the cloth and rollers. To obtain the desired effect, the fabric may need to be passed through the machine several times or through a line of several machines.

The metal teeth are formed of a moulded metal wire fixed onto a fabric tape of varying rigidity. Each section of the wire is characterized by various parameters, such as the diameter of the wire, the angle at which it is bent and the length of each section, the angle and length of the pointed section, the surface of the wire and the wire density.

The raising conditions for various fabrics may be stored for future use. The machines are attached with a seam detector which interrupts the raising process while a seam passes through the machine. The type of nap is controlled by the ratio of pile and counterpile energies. With

high counterpile energy, a shorter and thicker nap is produced, while high pile energy produces a longer and loftier nap. If the peripheral speed of the roller is equal to the cloth speed, no raising takes place. Cylinder raising is an extremely variable process. It varies considerably from machine to machine as well as on the same machine from day to day or even from hour to hour in some circumstances. It is not sufficient to maintain the same drive settings, cloth speed and tension. The effectiveness of raising depends on the construction of the knitting fabric and type of yarn used. The twist factor (British) should be lower than 3.8 so that the pile can stand erect without a twist. Some of the causes for the faulty raising of knitted fabrics are as follows:

- (1) Variation in temperature and humidity; cotton is usually processed in warm and dry conditions. Fabric with creases should be wetted, have creases removed and redried.
- (2) If pile action is higher, the fabric tends to cling on the pile roller. The fabric becomes tight at the feed side and very slack on the backside, resulting increase formation.
- (3) Damages may be caused by the bad setting of the cleaning brushes.
- (4) Uneven raising of metal needles should be corrected by regrinding or replacement.
- (5) The wire may be damaged at the selvedge. When changing fabric width, a damaged roller like this could produce lines.
- (6) Yarn variations may cause lateral striping.
- (7) Streaky or patchy raising may be due to traces of finishing agents.

#### 2.5 Stenter Finish

Before the invention of the stenter finish, traditionally the fabric after application of finishes were dried mostly on cylinder drier. But for other fabrics, especially synthetic and blends, the method results in huge shrinkage. A good alternative is a stenter frame, which was described in detail in the first chapter. A stenter performs multiple functions. After the application of finishes by a padding mangle, the wet fabric is sent directly to a stenter where it is dried, and the finish is cured while passing through multiple chambers of the stenter. As the fabric is tightly held during the passage and air is blown above and below the fabric, the fabric gets a unique full handle, which is more acceptable than the paper-like handle obtained by calendering, so calendering may be altogether be avoided. Setters originated as simple stationary pin frames, capable of width adjustment. These were developed into travelling hand-

fed pin frames and later to high-speed pin frames. They were still simple automatic clip frames. The object of all these machines was to hold cloth at a predetermined width during drying, largely without regard to the fabric length. For certain fabrics, gains in both length and width could be made by applying lengthwise tension to the fabric.

The demand for stable fabric dimensions led to the development of relaxed or overfeed methods of stentering and thereby to the revival of the pin stenter, since it is not practicable to feed surplus fabric into a clip machine. The present trend is to supply stenters that are universal in their application. Modern stenter chains combine both the clip and pin features so that the cloth requiring dimensional control may be processed on pins. Clips may be used either for the final finishing runs of cloth previously stentered on pins or for the finishing of cloth which does not require dimensional control. Knitted fabrics usually approach the stenter with width and length considerably different from the finished width and length. They are very elastic in construction and frequently have a considerable curl or roll on the selvedges.

The multiroller entering-frame deals with overfeeding. The scroll rollers have the double purpose of removing the edge curl and of increasing the fabric width. The driven rubber rollers, by means of their variable speeds, control the length of fabric when it is finally fed to the stenter.

The quality of the finished fabric must always be the prime consideration, and we should consider how far quality may be combined with high rates of production, which are also desirable. To ensure quality, the following conditions must be fulfilled:

- (a) The application of air and the drying process across the width of the fabric, including the selvedges, must be even.
- (b) The air streams above and below the fabric must be balanced to avoid straining, flapping and blowing off the pins.
- (c) There must be no migration of impregnated finishes or dyes, either from one face to the other or from place to place on the surface. Success in this manner usually follows from the correct application of conditions (a) and (b).
- (d) The temperature of the fabric must not be so high as to cause deterioration. This may be largely a matter of a correct stenter operation and does not necessarily preclude the use of high air temperatures. Quality is also, of course, dependent on correct feeding, pinning and clipping and the correct methods of removal. High drying rates require:

- (a) The highest possible air temperature, which is consistent with condition (d) above, enabling the maximum amount of heat to be transferred to the cloth. In practice, when steam is the heating medium, air temperatures are limited to about 320°F (160°C).
- (b) The lowest reasonable air humidities. Low humidity ensures low cloth temperature during most of the drying period, thus ensuring quality while also ensuring high drying rates by increasing the temperature difference between the hot air and the cloth. On the other hand, low humidities are obtained only by the addition of fresh air, thus increasing the steam consumption; compromise is therefore necessary.
- (c) The highest possible air jet speeds at the surface of the fabric. These high air speeds greatly increase the rate of heat transfer to the fabric by removing more of the stagnant air film. The penetration effect also helps to reduce any possible migration. This matter of high air speeds is a most important feature and one where the stenter designer has the most scope. The high air speeds must be obtained consistently with even application and balance and with the lowest possible fan horsepower, using the simplest and most accessible design of housing. The quantity of air, as well as its velocity, must be sufficient to convey the necessary heat for evaporation without an undue drop in temperature.

### 2.6 Summary

The general appearance, gloss and softness of all types of fabrics can be distinctly improved by calendering. However, different types of fabrics for different end uses demand different types of calenders and Schreiner. For subdued lustre or a matte finish, a felt calender is used. For a 3-D structural effect, an embossing calender is used. When considering the embossing of cotton, blended fabrics and nonwoven webs, one must be careful to utilize the thermoplastic properties of polyester, polyamides and similar fibres. High content wool fabrics, due to their inherent resiliency, do not lend themselves well to embossing. High content cotton or cellulosic fibre must be treated similarly to a paper product in that the fibre must be broken in order to transfer the pattern to the fabric. The use of pressure and/or the physical deformation of the fibre are required to get a permanent calendering result. The temperature to a maximum of 180°C, along with a dwell time or change in speed will enhance the effect, but neither the temperature nor the dwell time will afford any calendering effect without the use of pressure and/or deformation.

On the contrary, synthetic fibre properties and characteristics dictate that the use of heat in the presence of sufficient pressure is required to get a permanent calendered result. The

temperature normally ranges to a maximum of 230°C to enhance the effect, and the dwell time or change in speed will do the same, but neither the temperature nor the dwell time will afford any calendering effect without the use of some pressure-typical maximum to 250 PLI.

Calendering is not an exact practice; in many ways, it is an art rather than a science. It is like many other textile processes in that there are times when a theory presents the possibility, but experience is always the best teacher. Sueding (emerising) and napping (raising) are two very similar surface finishes. In both cases, the fabric is run adjacent to rollers or cylinders with special surface characteristics. In the case of sueding, the rollers are covered with sandpaper, whereas with napping, the cylinders are covered with metal points or teasel burrs. The sueding or emerising process aims to recreate the natural suede (i.e. leather-like) feel, whereas napped fabrics have a softer handle and better heat insulation (e.g. blankets, sleepwear, and winter clothing). Stenter frames are the most accepted machines for normal surface finishing today. They are universally used for finishing of all type of fabrics. Stenter frames have multiple functions like heat setting, drying and curing of chemical finishes. They impart a special type of surface feel on fabric called a 'stenter finish', and they have virtually replaced roller calender machine for surface finishing.

# **Chapter 3 Anti-shrink Finishing**

#### 3.1 Introduction

Cotton fabric is very popular choice for manufacturing apparel for various reasons: it is durable and can withstand a rough laundering treatment, especially under alkaline conditions; it has good perspiration and absorption characteristics; it is comfortable to wear; and it is able to take on a wide range of dyes. But the main problem with cotton fabric is shrinkage during washing or laundering. Shrinkage is an undesirable property of apparel, so in order to manufacture high-quality clothing, shrink-resistant fabric should be used.

However, there are fabrics which are more naturally resistant to shrinking. Synthetic fibres such as polyester or nylon are typically less prone to shrinking than others, although they are not 100% shrink-proof. It helps if they are washed and preshrunk, which helps further boost their resistance to future shrinking. The more synthetic fibres there are in a garment, the less likely it is to shrink. Cellulosic fibres are not as easily stabilized as thermoplastic synthetics, because they cannot be heat set to attain stability. Also, synthetic fibres do not exhibit the swelling/ deswelling scenario that cotton exhibits. However, the comfort and overall appeal of cotton has resulted in a greater demand for dimensional stability by both the consumer and the textile industry. The relaxation of fabrics made with cotton fibres, therefore, requires either mechanical and/or chemical means for stabilization.

Much of a fabric's residual shrinkage is the result of the tension applied to the fabric during wet processing. Some woven fabrics will shrink both in width and length during preparation and dyeing. These fabrics must be pulled out in order to maintain width and yardage yields, and the stress causes residual shrinkage. Knit fabrics are inherently wrinkle resistant; however, some are pulled out to a width wider than the fabric's knitted gauge, which also adds to residual shrinkage. Much of the stress-induced shrinkage can be eliminated by mechanically compacting the fabric. Compacting will result in reduced yardage yields, and cross-linking also reduces fabric shrinkage. A good resin finish will stabilize the fabric and reduce the residual shrinkage to less than 2%. The degree of stabilization required by chemical finishes will depend on the fabric's previous history.

#### 3.2 Shrinkage

The term 'shrinkage' can simply be defined as a change in the dimensions of a fabric or garment. This dimensional change may be in a positive (growth or elongation) or negative (shrinkage) direction for fabric length, width and thickness. Although the thickness of a fabric

also changes with processing and use, it is not usually considered problematic. For a cotton fabric, shrinkage relates to the loss of the length and/or width dimensions. In garment form, the shrinkage characteristics relate not only to a change in fabric dimensions, but they also can relate to other parameters, such as seam puckering, torqueing and overall garment fit. Shrinkage can be further defined as a dimensional change in a fabric or garment caused by an application of a force, energy or a change in environment that either allows the fabric to relax or forces it to move in a given direction. Cotton fabrics are often predisposed to dimensional instability, especially knits, which are very sensitive to applied forces or energies. Therefore whenever cotton knit fabric is manipulated in processing, its dimensions will change. If the fabrics are tested for their dimensions after a given process, the results may be different from the previous process. Shrinkage at any step in processing is residual in nature. By definition, 'residual' is defined as 'something that remains after a part is taken, a remnant, a remainder'. Another definition is 'an internal after-effect of an experience or activity that influences later behaviour'. From these definitions, it is easy to see that the forces in processing that stretch fabric result in more shrinkage, while the forces that contract or compress the fabric result in less shrinkage. Therefore residual shrinkage is the amount of shrinkage a fabric experiences plus or minus whatever the subsequent processing stresses apply to or remove from the fabric. Poor control of these processing forces can lead to high garment shrinkage as the after effect.

Woven fabrics are much more stable than knitted fabrics and do not react to stresses as severely. However, much lower shrinkage specifications are demanded of woven fabrics, thereby making the impact of processing stresses just as important for cotton woven fabrics as they are for knitted goods. Shrinkage and the cause of shrinkage can be further defined or broken down into two different types: construction shrinkage and processing shrinkage. This means that shrinkage is affected by the construction parameters of the fabric, and it is also affected by the forces applied during the dyeing and finishing processes, as well as during apparel manufacturing.

### 3.2.1 Construction Shrinkage

After cotton fabric is constructed on a knitting machine or weaving loom, it has inherent characteristics based solely on the yarn construction variables used. These characteristics or conditions are referred to as the greige delivered state and can be tested for various specifications, including shrinkage. The type of shrinkage measured at this point is defined as construction shrinkage, which is the amount of dimensional change in a fabric based solely on

the construction variables used to create the fabric. Construction shrinkage is measured after fabrication but before subsequent processes.

# 3.2.2 Processing Shrinkage

The dyeing and finishing processes, along with the apparel manufacturing operation, affect the dimensions of a product. Some techniques have more impact than others. These steps create processing shrinkage, which can be defined as the dimensional change that a process adds to or removes from the construction shrinkage of a fabric and thereby changes the residual shrinkage accordingly. The length and width dimensions are both affected, and the fabrics may either be stretched or consolidated. Most often, the length is stretched and the width is reduced during wet processing. Some of this shrinkage is composed of elastic shrinkage and can be easily recovered; however, some of the changes in dimensions may not be recovered because the elastic limits of the fabric as constructed have been exceeded.

## 3.2.3 Elastic Shrinkage

Elastic shrinkage is defined as a change in the dimensions of a fabric as a result of the fabric's ability to freely relax from the tensions experienced during construction and other processing. In the case of cotton greige knit goods, tensions in forming the knitted loop from the takedown and from spreader mechanisms on a knitting machine are examples of stresses that may induce elastic shrinkage, which becomes a part of the construction shrinkage. The stress in transporting fabric in bleaching and dyeing machines as well as finishing operations will also induce elastic shrinkage. Normally, the recovery from elastic stresses (i.e. realization of elastic shrinkage) is fairly spontaneous when these stresses are relieved, especially in a dry medium. It should be noted that due to these stresses during processing, the delivered dimensions that were measured for the greige fabrics are no longer applicable and so the residual shrinkage has changed. In fact, the stresses involved may exceed the elastic limit and will prevent the finished fabrics from relaxing or bulking as much as the greige fabrics. Therefore not only will the residual shrinkage be different, but also the relaxed dimensions of the processed fabrics will be different from the greige fabrics. Relaxed dimensions are defined as the state in which a fabric is fully relaxed and will not shrink further as a result of washing and tumble drying. Relaxed dimensions are also referred to as the reference state.

# 3.2.4 Drying Shrinkage

Drying shrinkage is defined as a dimensional change in a fabric when the 'deswelling' of fibre, yarn and construction occurs in the drying step. The structure shrinks upon itself as a result of

the physics of drying. Continuous processes during dyeing and preparation for drying usually stretch the length and pull down or reduce the width, sometimes beyond their elastic limits, thereby changing the relaxed dimensions. These swelling and deswelling phenomena, along with mechanical actions, are used in the AATCC Test Method 135-03 (AATCC, 2015). The test uses a washing machine to wet out (swell) the fibre/fabric under tensionless conditions, then a tumble dryer applies energy in the form of mechanical tumbling with heat to de-swell and fully relax the fabric/garment. Tumble drying without restrictions (i.e. tension) is a form of mechanical compression and allows for maximum 'drying shrinkage' to take place.

### 3.3 Shrinkage of Woven Fabric

The shrinkage of cotton woven goods on laundering has been one of the major problems and has been a cause of annoyance for users of shirts, pants, undergarments, etc. One of the causes of this shrinkage is the severe stretching of cotton goods during their manufacture; sometimes stretching is deliberately done to produce the maximum possible length and width. The shrinkage, which can occur in fabrics and garments of all kinds usually results from a relaxation of the stresses and strains, which have been introduced during previous processes such as finishing, making up, hot pressing, ironing and other forms of treatment designed to improve their appearance. Most textile materials can be stretched to a large extent, and those that are temporarily set in a stretched state while drying will retain that state as long as they remain dry. However, as soon as they are moistened, the stretched fibres rapidly contract to their true upstretched length, and the textile material may change its shape and size. To counteract the problem domestically, the fabric is wetted before the manufacture of the garment and is dried without tension. In the laundering of cotton goods, full shrinkage is not always produced in the first wash; often three launderings are required before dimensional stability is reached. It has generally been found that a fabric which shrinks 5% in the first washing treatment may shrink 7% before reaching stability. The ratio of the final shrinkage to initial shrinkage is often 1:4.

### 3.4 Shrinkage of Knitted Fabric

Each step in wet processing applies some stress to a knitted fabric. Some processes require that the fabric is pulled continuously through a range or cycle through a vessel in order to get a desired effect. Jet dyeing machines, becks, bleaching ranges and pad and beam processing units all pull on the fabric lengthwise during the process. Equipment that is manufactured today applies less stress on the fabric than the equipment that was manufactured only 10 years ago. At the same time, some soft flow and overflow jet dyeing machines actually do not stretch the fabric and may even relax the goods lengthwise. Unless it is restrained, the width of a knit

fabric will relax (shrink) upon wetting out in all these vessels as a result of the lengthwise tensions on the fabric. Relaxation dryers are available in conveyor belt systems, suction drum units and combinations of both, as well as continuous tumblers. All systems make use of mechanical actions during drying to provide the energy needed to yield a lower shrinkage. The methods used to shrink the fabric must be able to overcome the static friction that exists within the loops of the knitted structure. The softeners are used to aid fabric structure mobility by reducing the static friction at yarn intersections. In stabilizing knit goods, there is some question as to whether the use of the term 'relaxation shrinkage' is a proper one. The geometry of jersey construction does not lend itself to the application of the classical principles of stress relaxation and fibre swelling, considered to be the major causes of woven fabric shrinkage. It seems that in jersey constructions, one must reestablish the normal relaxed formation of the loop stitches of the fabric rather than only relieve stresses in yarns. Knitted fabrics are prone to stretching and mechanical deformations because the yarns are subjected to high stress during production. The recovery of the fabric from extension is poor, so after the removal of applied forces, the fabric tends to remain in a distorted configuration rather than return to its undistorted shape. The instability of the knitted structure is not only due to the looseness of construction, but also to the degree of freedom caused by the slipping of the crossover points in a loop. It is demonstrated that relaxation shrinkage contributes to the easy distortion of the fabric. Distortions caused by load or tension involved in the manufacturing process depend upon the resistance of the individual loops. The degree of instability also depends upon the frictional force exerted on the loops during the application of the load. On the release of load, the structure tries to regain the original configuration or at least to stable form provided the frictional forces against movement are overcome. Knitted fabrics are inherently difficult to stabilize, as the construction allows for a contraction of up to 40%. Thus knitted fabrics often never fully recover from the strains they are subjected to and have to withstand the considerable wear and tear of everyday use and laundering processes. Home and commercial laundering equipment can accelerate shrinkage/stretching to a certain extent.

The shrinkage of cotton and rayon goods is merely a relaxation of the tension and extension that occurred during the manufacture of the fabric. Water acts as a molecular lubricant, causing the micelles, fibres and yarns to contract to the position of minimum strain from which they were originally stretched during spinning and weaving. Such shrinkage is often referred to as linear shrinkage or length shrinkage.

### 3.5 Shrinkage of Woollen Fabric

The shrinkage of animal fibres is essentially different from that of cotton and rayon fibres. In wool, large shrinkage is possible, as considerable distortion may occur due to folding and unfolding of keratin molecules. The presence of scales and crimps is responsible for the considerable diminution of area by way of felting. The felting of wool is often expressed as area shrinkage; this phenomenon will be dealt separately.

#### 3.6 Causes of Shrinkage

During the spinning, weaving, bleaching, dyeing and various other finishing processes, yarns and cloth are under continuous tension. Yarns and/or fabrics are not fixed materials; they consist of separate, stretchable fibres which submit to the tension. In other words, fabrics can stretch in length and width. The tension within the yarn, which is caused by this stretching, can be eliminated when the friction within the fabric is reduced. This reduction in friction occurs during laundering, where both water and soap act as lubricants. The lubricant, along with the mechanical action of the washing, helps the fibres relax and contract to their original length before the elongation takes place; this means that the fabric shrinks and recaptures its original equilibrium.

However, shrinkage is not only due to the release of strains imposed during manufacturing processes; it is also due to swelling caused by wetting bringing about an internal arrangement of the material resulting in external shortening. The diameter of cotton hairs may increase by 20% and the contraction due to swelling may amount to 2% out of the total fabric shrinkage, which is about 10%. Materials made of rayon show much greater swelling and consequent greater shrinkage. Unlike cotton, rayon may exhibit up to 9–10% yarn shrinkage. Fabrics made of synthetic fibres swell only very slightly on wetting, and so such fabrics are almost free from this type of shrinkage.

In general, the warp yarns bend round the weft yarns to a greater extent. When the fabric is wetted, the yarns increase in diameter, and as the fabric is under no constraint, straighter weft threads move closer together so that the warp yarn can remain the same length. The fabric therefore shrinks in the warp direction. Alternatively, where the warp threads are straight, then the weft threads are crimped and the fabric shrinks weft-way. A fabric must contract one way or another on wetting. In most fabrics, the warp yarns are finer than the weft, and therefore bend more easily. Secondly, they are closer together for good cover and are less able to bend the weft threads; hence the stable structure is that in which the warp crimp is high and weft

crimp is low. The considerable tension on the warp during the wet processing of piece goods, particularly while drawing the fabric through various machines, generally, extends the fabric lengthways. The stretched structure has a low warp crimp and is therefore unstable; when washed, the fabric takes up a more stable position and being free from restraint, it shrinks. A relatively large shrinkage of 10% may occur in cotton fabric. Sometimes it has been found that cotton materials are not completely shrunk on the first wash, but continue to shrink in further washes.

The knitted fabrics, by reason of their stitch structure and the ease with which they can be stretched and distorted, are not so amenable to shrinkage control as are woven fabrics. Because of this looseness in construction, the twisted yarn in the loop has a tendency to untwist and release built-in torsional strain so as to acquire the natural configuration of a minimum energy state. Hence the looped yarn uncurls inside the fabric, giving rise to chirality and a dimensional instability.

## 3.7 Measurement of Residual Shrinkage

Residual shrinkage can be measured using the following various test methods: ISO 3759/ISO 6330/ISO 5077/AATCC 150/AATCC 135. AATCC Test Method 135-2004, dimensional changes of fabrics after home laundering, is briefly discussed below:

Dimensional changes are the changes in the length or width of a fabric specimen subject to specified conditions. The change is usually expressed as a percentage of the initial dimension of the specimen.

This test method is intended for the determination of dimensional changes of fabrics when subjected to home laundering procedures with an aqueous detergent solution and normally including rinsing, extracting and drying. Four washing temperatures, three agitation cycles, two rinse temperatures and four drying procedures cover the common home care options available to consumers using current laundering machines.

The suggested steps for testing are as follows:

- (1) Condition each specimen for at least 4 h in an atmosphere of  $21 \pm 1^{\circ}$ C ( $70 \pm 2^{\circ}$ F) and  $65 \pm 2\%$  RH.
- (2) Lay the sample on a flat surface. Do not allow any section of the sample to hang over the edge of the work table. Using a template for the selected test size (e.g.  $15 \times 15$  in.  $24 \times 24$  in.

or a mutually decided size), mark specimens parallel to the selvage or fabric length direction. Specimens should be taken from areas with different lengthwise and widthwise yarns. Identify the length direction of the specimens before cutting them out of the sample. Use a pen with indelible ink to mark each test specimen with three 250 mm (10 in.) or pairs of benchmarks parallel to the test specimen length and three 10 in. (or 18.0 in. in case of larger test specimen) pairs of benchmarks parallel to the test specimen width. Each benchmark must be at least 50 mm (2 in.) from all test specimen edges. Pairs of benchmarks in the same direction must be spaced approximately 120 mm (5 in.) apart.

- (3) Washing load fabrics/ballasts (generally hemmed pieces of bleached cotton sheeting) are used to ensure that a wash load used for garment testing is of a sufficient weight. The products simulate the washing of a garment in domestic washing machines.
- (4) Weigh test specimens and enough ballast to make a  $1.8 \pm 0.1$  kg load or alternately  $3.6 \pm 0.1$  kg may be used. Dimensional change results in both cases will be different and should not be compared.
- (5) Add specified amounts of 1993 AATCC Standard Reference Detergent and water in automatic washing machine.
- (6) Agitate water briefly to dissolve detergent.
- (7) Stop the machine agitation. Add test specimens and ballast to machine. Set the washer for the selected washing cycle and time.
- (8) The water level, agitator speed, washing time, spin speed and final spin time in the washing machine are specified for three categories of fabric: normal, delicate and permanent press. Choose the suitable specification and begin wash cycle.
- (9) The water temperature for the washing cycle may be  $27^{\circ}$ C,  $41^{\circ}$ C,  $49^{\circ}$ C or  $60^{\circ}$ C ( $\pm 3^{\circ}$ C each case).
- (10) After the completion of the washing cycle, rinse the samples at a temperature less than 29°C.
- (11) Dry by any of the alternate drying methods: tumble, line or on a horizontal screen or perforated surface to remove wrinkles without distorting or stretching.

- (12) Repeat the selected washing and drying cycle two more times or to an agreed number of cycles.
- (13) The wash samples are conditioned for at least 4 h in an atmosphere of  $21 \pm 1$  °C ( $70 \pm 2$  °F) and  $65 \pm 2$ % RH.
- (14) The fabric for a form-fitting garment may require restoration by pulling, or some fabric may require light ironing.
- (15) Measure and record the distance between each pair of benchmarks to the nearest millimeter (eighth or tenth of an inch).
- (16) The percentage of dimensional shrinkage is the difference in the dimension of the benchmarks expressed as a percentage of original length or width. For a detailed specification, please see AATCC Technical Manual (AATCC, 2015).

## 3.8 Shrink-Proofing Methods

The fabric shrinking machines, popularly known as zero–zero and Sanforising ranges, impart dimensional stability and an attractive hand to the fabric, which are required by the fabrics for garment and non-apparel end use. This is an ecofriendly compressive shrinking process as it is carried out without the aid of any chemicals. An early idea for producing unshrinkable cotton fabrics was that of giving the fabric a thoroughly good wash in a rotary or other convenient type of washing machine where the cotton fabric was free to shrink, then drying and finishing it with the least possible stretching. However, the method is not reliable and requires a huge washing plant. For long time fabric shrinkage was reduced by relaxation drying; in the primitive 'London shrinking' method, the fabric was wrapped with a wet cloth under tensionless conditions for a few days, and then hung on a frame to dry.

There are two ways in which woven cotton material may be shrunk before it reaches the consumer. The preshrinking may be done by mechanical means as a final finishing operation, or the cotton may be given resin treatment which prevents creasing as well as swelling and contraction. It is known that a preshrinking process like Sanforising enables residual shrinkage in laundering to be reduced to 0.75% or less. However, it is important that after preshrinking, the fabric should not be stretched further. Contact rollers are now used for tensionless batching. Dimensional stability of the fabric compared to that of untreated fabric can also be improved by a resin treatment (chemical method), though the primary aim of such a treatment is to improve the crease recovery property of the fabric. Compacting, stentering, decatising,

calendaring, sanforising, etc., are some of the common mechanical finishes which reduce residual shrinkages on both knits and wovens. However, the process of subjecting tubular knit fabric to mechanical compaction invariable provides a 'one-sidedness' to the fabric, which creates problems when it is cut, sewn and made into finished garments. Moreover, mechanical preshrinking systems are generally fairly expensive because of the capital investment in equipment and reduced production speed. It is noteworthy that the mechanical processes, such as contractive drying and compressive shrinkage, which are successful with cotton but not with rayon, particularly spun rayon. The chemical crease-resisting process with resins, however, gives a substantial degree of resistance to shrinkage with spun rayons, and in many cases the potential shrinkage is reduced from 12% to about 2%. If a higher degree of shrink prevention is required, then it is possible to combine mechanical shrinkage with resin treatments. For instance, during the normal processing of resin-treated goods, they may be dried on a stenter with an overfeed device or some contractive method of imparting shrinkage during stentering. Again, it is possible to combine the resin treatment with controlled compressive shrinkage.

In the overfeed method of compressive drying, the wet fabric is fed on to the stenter faster than the speed of the machine, providing sufficient slack to accommodate the shrinkage produced by the subsequent stretching of the weft. The amount of shrinkage can therefore be controlled. The relative rate of feeding and running, however, is not the only point to be considered, for it is necessary that the amount of slack between the pins should correspond to the amount of shrinkage required. This is effected by a brush roller made in two parts, one of which floats on the slack fabric and impales it on the pins, after which the heavier brush wheel force the fabric into full engagement with the pins. During stentering, the fabric is extended the weft-way as usual and with no warp tension, the crinkles are removed from the warp. In this case, the weft takes care of itself provided there is no undue lateral extension during stentering but merely sufficient tension to produce a simple flat fabric; it is warp shrinkage and not weft shrinkage. Sanforising and Rigmel are two methods of producing anti-shrink cellulosic materials developed by Sanford Cluett (US) and Wrigley and Melville (UK), respectively. Both the compressive shrinkage machines reduced the residual shrinkage in cotton and linen to not more than 1%. Tests have been developed to ensure that the proper application of this process conforms to this standard. The fabric is deliberately shortened in both width and length, resulting in a tighter and closer weave and consequently, a higher thread count. Both the methods depend on the cloth being held in contact with an elastic surface that is changing from an extended state to a contracted form under the action of a compressive force so that the fabric

is also subjected to the same compression. In a sinuous waveform of an elastic material, the convex surface is extended and the concave surface is contracted. If a fabric is placed on the extended crest of the wave formed by the elastic material and moves with it into a contracted portion that is being held firmly in contact with the elastic material during the movement, then the fabric will be contracted by the compression force. Both methods utilize very judiciously a thick blanket for bringing about a closing up of the warp. They both allow the weft closing up to take care of itself. The fabric is not stretched weft wise or set to an upstretched width in the stentering operation as a final stage in fabric finishing. Both processes cause warp shrinkage as most the fabric shrink more in the warp than in the weft and in garment warp shrinkage is more usually more important. At first, a measured piece of fabric is washed under condition to produce maximum possible shrinkage. It is carefully dried and smoothened without stretching. The warp and weft shrinkages are calculated in order to assess the necessary extent of closing up or contraction of the original fabric during the above finishing process so that after washing, it will remain unchanged.

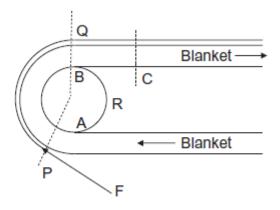


Fig. 3.1 Principle of compressive shrinkage.

### 3.8.1 Principle

The principle of preshrinking cotton and rayon fabrics is shown in Fig. 3.1. A thick endless blanket passes around a rotating roller R of comparatively small diameter. The fabric F is led against the blanket at point P just as the blanket begins to travel around the roller. It moves along with and stays in close contact with the blanket until it reaches point Q where the blanket leaves the roller and travels along a straight path. Because the blanket is thick and the roller has a small radius of curvature, the length of the outer surface of the blanket between P and Q becomes greater than that of the inner surface between A and B. While the blanket continues along the straight path to point C, where the distance AB is equal to BC, then the outer surface of the blanket will contract so that distance PQ decreases to BC or the extent of contraction is

(PQ-AB). The blanket thereafter remains uniform and undistorted until it returns to the underside of roller R. The important point is that the outer surface of the thick blanket is in an extended form while passing around the roller, but it returns to its unextended form when it leaves the roller and moves forward in a straight path.

### 3.9 Sanforising Machine

The word SANFORISED is derived from the first name of the inventor of the compressive shrinkage process, Mr. Sanford L. Cluett. The Sanforized Company, a division of Cluett Peabody & Co., New York, is the sole owner of the registered trademarks Sanforized, Sanfor and Sanforizado. The Sanforized label means dimensionally stable garments made up of Sanforized fabrics. The purpose of the process is to shrink fabrics in such a way that the textiles made up of these fabrics do not shrink during washing. The Sanforized trademark was first registered in the United States in 1930. At present, the trademark is registered in over 100 countries worldwide. The global licensees use the trademark on both 100% cotton and cotton-blend fabrics which meet special test requirements and conform to a precise standard of shrinkage established by the Sanforized company.

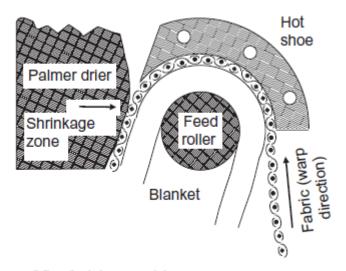


Fig. 3.2 Shrinkage zone of Sanforising machine.

In Sanforising machine (Fig. 3.2), a woollen felt blanket runs over a feed roller. The fabric to be finished runs in contact with the blanket under the pressure of a hot shoe. The hot shoe is placed above the curved portion of the blanket while running over the feed roller so that the fabric is in firm contact with the blanket. The fabric enters the nip of an internally heated cylinder called a palmer drier (60–84 in. diameter) just beyond the point at which the blanket and superimposed fabric leave the roller (shown as the shrinkage zone). Where the blanket

reverses its curvature, the fabric contracts in order to conform to the contracted surface of the blanket as it is firmly held in contact with the blanket by the palmer drying cylinder.

The steps for Sanforising can be summarized as follows:

- (1) The fabric is dampened with steam, which softens the fabric for adjustments to be made in length and width.
- (2) The width is adjusted by a stretching action with the help of a small stenter in which the fabric is gripped along its selvedges.
- (3) The fabric is then held firmly against a heavy woollen blanket, which is under controlled tension.
- (4) As the tension of the blanket is relaxed to the desired measurements, the fabric shrinks uniformly in length.
- (5) The fabric is then carried around a heated cylinder where it is dried and the surface achieves a special type of finish.
- (6) A sample is tested again to assure that residual shrinkage on laundering to be reduced to 0.75% or less.

The thickness of the woollen blanket/felt determines the longitudinal contraction of fabric as shown in Table 3.1. The present version of the Sanforising range uses a rubber belt.

Table 3.1 The relation between blanket thickness and contraction of fabric

Type of blanket	Thickness (in.)	Use	Contraction possible (in./yard)
Thin	0.275	Bleached shirting	Maximum 3.5
Heavy	0.4	Medium weight	3.5–5.0
		fabric	
Extra-heavy	0.45	Denims	3.9–5.6

The schematic diagram of a Sanforising machine is shown in Fig. 3.3. Some components of the machines are as follows:

- 1. Felt endless blanket
- 2. Guide roller
- 3. Palmer drier (steam-heated)

- 4. Teflon compacting apron
- 5. Fabric to be finished
- 6. Water spray to clean the endless blanket after every rotation
- 7. Squeezing rollers to remove water used for cleaning

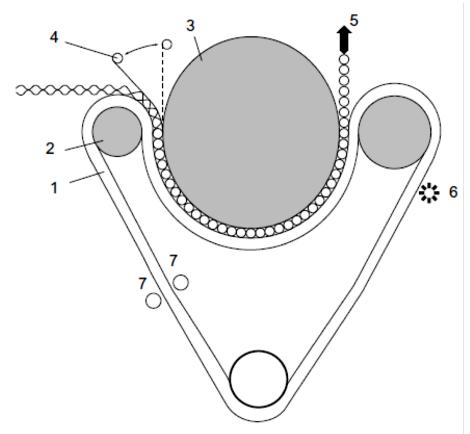


Fig. 3.3 Schematic diagram of Sanforising machine.

Bending the felt blanket around the guide roller stretches the surface of the felt and increases the circumferential speed correspondingly. This change of circumferential speed leads to the compacting of the fabric. The fabric is fed into the Sanforising range through the feed rollers of a mangle, whose function is to hold back the fabric and allow the blanket to contract it only by the required amount and not up to the maximum capacity of the blanket, thus over-shrinkage is prevented. At first, the fabric passes from the mangle through the sprayers, as moist fabric is soft, plastic and more easily compressed by the felt blanket. This moistening may be assisted by a steaming apparatus. The fabric then passes through a short clip expander or stenter about 3 ft. 6 in. in length which controls the width of the fabric as it fed to the blanket. The delivery end of this stenter is matched with the desired width as determined by the standard wash test. The fabric then passes to the felt blanket over the feed roller and placed in firm contact with

the stretched surface of the blanket by electrically heated shoes. The shoes are automatically lifted from the blanket when the machine is stopped, thus eliminating scorching; less shrinkage results with cold shoes. Then the fabric comes in contact with the steam-heated palmer drier when it is already contracted due to a reversal of curvature of the felt blanket. A special device keeps the blanket running in a straight path on the cylinder. There are also devices for measuring and controlling tension on the blanket. As an option, there may be additional palmer unit when a similar finish on both sides of the fabric or additional shrinkage is required. The fabric should be thoroughly dried before leaving the sanforiser; otherwise, it will elongate. After coming out of the felt calendaring unit, the fabric is guided to the plaiter, which plaits the fabric into a trolley.

Cotton and other cellulosic fibre may also be preshrunk by a process called 'Sanforset' which combines ammoniating with the compressive shrinkage process of Sanforising. Shrinkage after laundering is not more than 1% for cotton or 2% for rayon; these fabrics have considerable wrinkle resistance and their seams retain a flat appearance.

### 3.10 Rigmel Finish

A Rigmel machine (Fig. 3.4) gives more lustre than a Sanforising machine because the fabric is held against a thick rubber apron during compressive shrinkage. The endless rubber apron moves over a pair of rollers, but there is no danger of blanket marks on very fine fabric, as is in the case of the Sanforising machine. Due to a higher thickness and greater elasticity, it is easy to pass from the extended to the non-extended state as the endless rubber moves over a rotary drum. The outer surface of the rubber apron will be extended while passing over the peripheral surface of half the circumference of the drum until it reaches straight path on leaving the surface of the drums. If a moistened fabric is held in contact with the rubber apron, compressive shrinkage will take place. A hot pressure plate mounted above the drum keeps the fabric in contact with the surface of the rubber apron. The leading edge of the plate covers a portion of the curved periphery of the rubber apron on one side and a similar portion of the straight path of the apron on the other side.

The drum in the Rigmel machine is about 18–24 in. in diameter. The pressure plate of the Rigmel machine (Fig. 3.4A) is larger than the shoe of the Sanforising machine and the rubber apron is about 2–3 in. thick. In a second type of the Rigmel machine (Fig. 3.4B), an external roller is used instead of a pressure plate which moves in touch with the outer surface of the rubber apron.

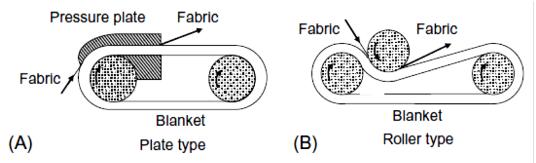


Fig. 3.4 Two types of Rigmel compressive Shrinkage machine.

A woollen blanket is used in the Sanforising machine while a rubber apron is used in the Rigmel machine. In the Sanforising machine, compressive shrinkage of fabric depends on the reversal of a sharp curvature from the small roller on which the shoes fit to the very gradual reverse curvature of the large drum of the palmer machine, whereas the Rigmel machine produces the same result when passing through a relatively small quadrant. A small palmer drying cylinder is connected with the Rigmel machine, but it plays no part in the shrinkage, as it is only employed to dry the fabric completely and set the fabric in a stable state.

# 3.11 Confining Passage Type

Another type of compressive shrinkage machine called the confining passage depends on the principle that if a fabric enters a nip between rollers and leaves at the second nip, which is running more slowly, and the space between the nips is filled in to prevent the fabric from puckering, then it becomes compressed lengthwise; that is, the fabric becomes shortened by a uniform closing up of its weft threads. The machine (Fig. 3.5) comprises three rollers (A, B and C) running together with a smooth polished metal block (D) fitted between the nips. The upper surface of this block has a curvature closely conforming to that of roller B. Rollers A and B rotate together at the same peripheral speed while roller C rotates more slowly. The surface of rollers A and C are covered with relatively soft rubber while roller B is covered with harder rubber. The metal block may be heated.

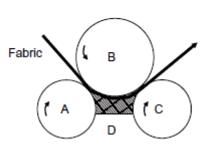


Fig. 3.5 Confining passage type compressive shrinkage machine.

## 3.12 Compactors For Knitted Fabric

A compactor is designed especially for compacting 100% cotton knitted fabric like jersey, pique, interlock, plush, rib and sinker, etc., as well as cotton-blended fabric in rope form, imparting dimensional stability of the fabric and presenting it in plaited form. Two felt compacting units are used to obtain top-quality fabric with minimized residual shrinkage and a soft, fluffy hand. The compacting machine is also called a compactor or felt compactor. This machine is generally equipped with two steam chambers and two felt units. It preshrinks and stabilizes knitted fabric in order to avoid excessive shrinkage after washing. This process with compacting felts gives handle to the fabric. The shrinkage required (between 2% and 10%) is mechanically given by two consecutive endless felts using high temperature and steam pressure. There are two types of felt compacting machines:

- (1) Tubular machines for the shrinkage of tubular knitted fabrics.
- (2) Open width machines for the shrinkage of open width knitted fabrics.

Most of the machines are heated by steam, but some of them can be equipped with a thermo oil heating system in order to finish synthetic fabrics. Some of the popular makers of compactor are: Ferraro Spa, Albrecht, Bianco, Brückner, Fong's, Heliot, Krantz, Monforts, Dezhoutex, etc.

### 3.12.1 Function of Compactor Machine

The compactor is very important knitted fabric finishing machine. The work done by a compactor machine is pointed out below:

- (1) Cloth density of the knitted fabric in terms of grams per square metre (GSM) can be controlled by a compactor. For a higher GSM, the overfeed is increased and fabric width is decreased. For a lower GSM, the overfeed is decreased and the fabric width is increased
- (2) Reduce residual shrinkage
- (3) Control twisting of knitted fabric
- (4) Increase smoothness of fabric
- (5) Simultaneous heat setting of the blended fabric, etc.

### 3.12.2 Checking Parameters of Compacted Fabric

The following parameters are checked after compacting:

- (1) Shade checking: shade of the compacting fabric in the delivery side of the machine is compared by the operator with the buyer's approved swatch.
- (2) Width check: operator measures the width of the fabric with the measuring tape and compares it with the buyer's requirement.
- (3) Weight check: fabric weight of the fabric is determined by GSM. Operator checks the GSM of the fabric by the GSM cutter and the electric balance.
- (4) Edge line checking: two edges of the fabric are checked in on the delivery side. If any fix line is identified, which normally occurs from the expander, it should be connected.
- (5) Design and slanting: operator checks design and slanting of the fabric in the delivery side of the machine.

There are two types of compactor machines:

- 1. Tubular compactor
- 2. Open compactor

## 3.12.3 Tubular Compactor

The treatment of knit fabrics in tubular form on the tubular compactor meets the standards set by customers so that the garment stitched from the fabric finished on this machine will yield the lowest residual shrinkage values. Other features are:

- (1) Width control through a step less, adjustable tubular fabric spreader driven by a variable speed motor for distortion-free fabric guidance.
- (2) Steaming with a condensate-free steam box which is easily operated and completely made from stainless steel.
- (3) Compacting through two Nomex felt belts.
- (4) Calendaring while passing between the felt belt and the heated shrinking rollers.
- (5) Precision plaiting with automatic platform level adjustment controlled by folded fabric height. Alternatively, a fabric rolling system can be provided.
- (6) The fabric is fed through the guiding system and stretcher, which then takes the fabric through the steam box onto the felt of the twin compacting units.

- (7) An optical fabric density-measuring sensor can provide the means to automatically control the compaction of a tubular compactor to achieve the desired course count. An automatic compaction control system based on density measurement and control will:
- (a) Eliminate over- and underweight fabric
- (b) Reduce or eliminate punch-weight measurements
- (c) Provide consistent results from all operators
- (d) Result in uniform fabric, seam-to-seam

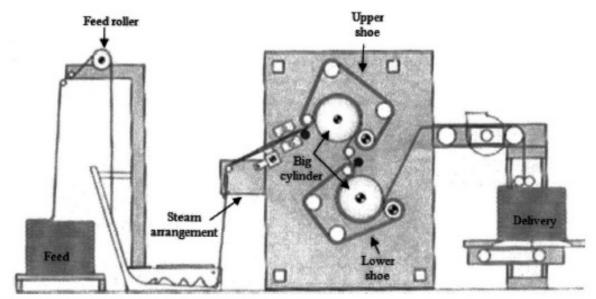


Fig. 3.6 Fabric path in a tube compactor.

Fig. 3.6 shows the fabric path through a tube compactor. The fabric feed from the feed roller under a slack condition is heated and moistened by steam and delivered to double compacting units fitted one above another for compacting both sides of the tubular knitted fabric. Each compacting unit consists of an endless blanket, a hot metal shoe and a big hot metal cylinder, as in the case of Sanforising units. The compacted fabric is delivered on a trolley.

### 3.12.4 Open Width Compactor

An open compactor is used for compacting the open-form fabric. Here, a slitting machine is used to cut tubular fabric to open width form.

The features of open width compactor are:

- (1) Perfect Sanforising finishing of knit fabrics
- (2) Ideally suitable for synchronizing with stenter machines

- (3) Effective shrinkage control
- (4) A.C. inverter drives, programmable logic controller (PLC) with touch screen used
- (5) Equipped with modem connectivity and online troubleshooting system

# (6) Metal detector

An open width compactor is suitable for open width knit fabrics to achieve an exact dimensional stability and a soft feel. The machine generally consists of a feeding frame with a centering device and driven scroll rollers, an equalizing stenter frame with overfeed roller and brush pinning arrangement. The entry section of a pin frame is provided with edge spreaders IR in-feed device, an S.S. fabricated steaming unit for uniform moistening of the fabric. The steaming device has stainless steel sliding shutters that allow steam to flow only per the width of the fabric. The compacting unit consists of two felt compacting units, each of them consisting of a Nomex felt approximately 20 mm thick, a steam-heated chrome-plated centre roller of diameter 400 mm, a rubber covered roller driven by a variable frequency drive, a compacting pressure roller, a felt tensioning roller and a felt cantering roller. Each unit is provided with a special antifriction sheet-type shoe that is controlled by an electrical actuator to control the compressive shrinkage. A fabric cooling roller is provided after a second felt to cool the fabric by means of chilled water circulation. Fabric tension through the machine is controlled with the help of sensitive load cells and a variable frequency drive with PLC and touch screen.

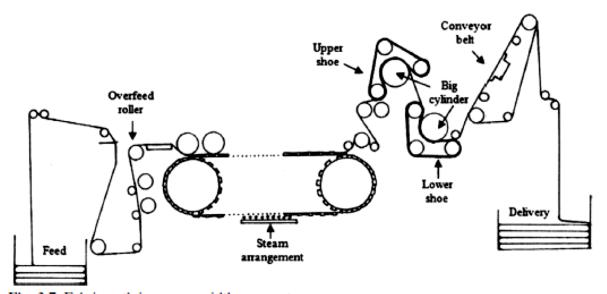


Fig. 3.7 Fabric path in a open width compactor.

Fig. 3.7 shows the fabric path through open width compactor. The tubular fabric is cut into slits and the fabric in open width is passed over a steam-heated conveyor in slack condition. It is passed through a double compacting system as in the case of the tubular compactor. The compacted fabric is delivered into a conveyor and then finally in a trolley.

# 3.12.5 Blanket less Compactor

A new concept for dealing with the shrinkage problem in woven and knit fabrics is currently being commercially exploited. The so-called new compactor apparatus relies on the principle of feeding a fabric at a fixed and uniform speed by means of a feed roll of the proper surface frictional characteristics into a zone sometimes referred to as the compacting zone. In this zone, the fabric speed is reduced by a retarding roll in proportion to the shrinkage to be removed. The compacting zone is established through the careful positioning of the three principal elements of the machine; a feeding roll, a retarding roll and a third element, usually a shoe, a blade or a combination of both. The fabric is fed into the compactor between a roll and a blade or a shoe. In doing so, a forward speed is established. When the fabric is released by the leading edge of the blade or shoe, the second roll, which generally has a surface with a high coefficient of friction, takes over control of the fabric. If this second roll is rotated at a slower speed than the feeding roll, a warp-wise compacting action takes place in the fabric. Ideally speaking, the degree of compaction which takes place is in a direct ratio to the relative speeds of the two compacting rolls. The fabric to be processed is fed into the compactor between the top feed roll and the steam shoe blade combination. The shoe blade assembly is so arranged as to force the fabric lightly against the surface of the feed roll. The leading edge of the blade is displaced somewhat behind the centerline of the two rolls. The amount of this displacement will be determined by the density of the fabric as well as the type of result to be achieved. A typical setting might be, for instance, 0.220 in. The gap or space between the two rolls is adjustable as well, depending on the fabric type and result desired. Here, a spacing of 0.006 in. would be consistent with the first horizontal blade setting of 0.220 in. A method has been described in which the lengthwise stitches of the fabric are mechanically compacted. Since the equipment described by Constantine and Fox (1969), technology has changed a lot. In recent machines, the feed roller and retarder rollers are separated and the spring-type retarder is fitted on the feed roller. In a typical setup (Fig. 3.8), the untreated knitted fabric, supported by the heated plasmacoated main roll (1), is introduced into a converging passage, firmly gripped and conveyed into a treatment cavity, where the compaction takes place. Underneath the tungsten-carbide-coated

retarder (2) are photo etched microgrooves which hold back the fabric and process it with hundreds of individual treatment zones.

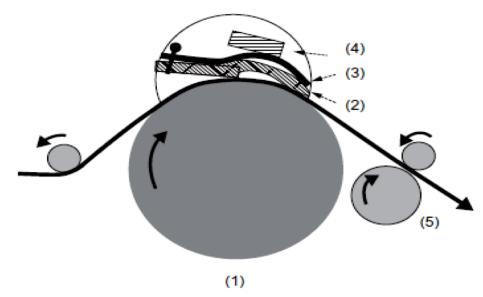


Fig. 3.8 Schematic diagram of a Blanket less compactor for knitted fabric.

The force thus delivered provides high levels of compaction. The Walton curve (3) with its spring-like action combined with the individual treatment zones at the retarder assures a compacting effect, which is characteristic of the technology, resulting in even, uniform compaction. A pressure plate (4) controls the pressure on the Walton curve and retarder. The fabric is finally wound on a pull roller, batch or retarder roller (5). If the main roller (1) and batch roller (5) run at the surface speeds of 30 m/min and 26.4 m/min, respectively, the degree of compaction will be 12%. The compactor is used 'online' after a stenter, which offers the following advantages:

- (1) Convenient size of compactor (length 1.5 m)
- (2) High production speed while compaction remains uniform

### 3.13 Conclusion

Fabric shrinkage during laundering is a common problem for fabric users, resulting in misfit of garments. The fabric may be made shrink-proof by mechanical and chemical methods. Mechanical methods are ecofriendly. The methods are based on moving fabric into intimate contact with an expanded rubber or woollen blanket and the subsequent simultaneous compression of the blanket and the fabric. The Sanforising method of compressive shrinkage was developed as early as 1930, but is still popular till date. The recent trend is the use of blanket less compressive shrinkage machines.

# **Chapter 4 Mercerization**

#### 4.1 Introduction

Mercerization has been used as a pretreatment for other wet processing steps such as durable press finishing and enzyme treatments. Improper mercerization has been shown to adversely affect crease edge abrasion properties. Commercial applications of mercerization are carried out to enhance the value of the finished fabric for the consumer. Therefore it is mostly considered to be a finishing process rather than a preparatory process, which is mainly carried out to facilitate colouration processes. Mercerization refers to the treatment of cotton yarns or fabrics with caustic soda solution. It is not a fibre purification process, but it does induce desirable changes in cotton yarn and fabric properties. The process takes its name from John Mercer, who in 1850 observed that there was a general swelling and shrinking effect on cotton fabric when treated with caustic soda solution. Horace Lowe (1889–90) later demonstrated the high lustre of mercerized cotton by maintaining tension during treatment. Considerable quantities of cotton yarns and fabrics are mercerized to improve lustre and/or dye uptake and more recently without tension to produce stretch materials. The objective of mercerizing is to swell the cotton fibre, increasing its lustre, tensile strength (as well as retain it after an easy-care finish), dimensional stability and dyeability (in terms of uniformity and colour yield).

Mercerization is a well-established finishing process for cotton which affects its fine structure, morphology, mechanical properties and reactivity. In general, crystallinity and crystallite size are decreased, while the orientation of the cellulose chains with respect to the fibre axis is increased. The native cellulose (i.e. cellulose I crystal lattice) is more or less completely converted to the cellulose II form during mercerization depending on the treatment temperature and the concentration of sodium hydroxide, as well as on the tension applied. If tension is applied during the process, the swelling of the cellulose structure is impaired, and the conversion of the crystal lattice to cellulose II is incomplete. In a study, mercerization processes were performed using three different conditions of mercerization, namely slack, constant length and under tension of two types of cotton towels. The effect of these techniques on mechanical behaviour and dyeability with reactive dye were investigated. The barium activity number was used to express the degree of mercerization of the mercerized fabrics. It was observed that mercerization treatments decrease the cellulose crystallinity index where the decrement in the crystallinity index was different in various mercerization conditions. Mercerization treatments may increase dye uptake and wettability. Highest dye uptake and wettability were observed for slack mercerized fabrics followed by constant length then under tension mercerized when

compared with unmercerised fabric sample. Tensile strength of under tension mercerized fabric samples showed higher values than the slack mercerized fabric.

Traditionally, a cold solution of 25–26% by mass of sodium hydroxide is used, although a better penetration and a more even treatment are obtained with the more recent hot mercerizing technique. The addition of a wetting agent to the mercerizing liquor gives a better penetration and a more even treatment, particularly for unscoured materials. The time of impregnation is rarely sufficient for maximum swelling, so that even with the mercerization of absorbent bleached cotton, the addition of a wetting agent is advantageous. The presence of a wetting agent is a safeguard against uneven penetration; however, normal wetting agents are not sufficiently soluble in a moderately concentrated caustic soda solution. The products based on cresylic acid (i.e. a mixture of o-, m- and p-cresols) were popular in the past. Cresylic acid alone is less effective, and the wetting power may be increased by the addition of solvent additives such as alcohols (e.g. cyclohexanol), ethers, ketones, lower fatty acid amides or cellosolve. The presence of such products poses problems because of their toxic and nonbiodegradable nature. The biodegradable wetting agents include certain alkyl-aryl-sulfonates, some organophosphates (e.g. sodium methyl-octyl-phosphonate), alkylated diphenyl-oxide sulphates (e.g. dodecyl-diphenyl-oxide sulphate) and especially sulphated aliphatic alcohols (e.g. 2-ethylhexyl sulphate). The most efficient sulphated alcohols are those with low molecular weight (e.g. chains with four to eight carbons). Moreover, products with branched chains are more efficient than those with linear chains. A cresol-free alternative is sulphated 2ethylhexanol, which is activated with about 10% butanes and un-sulphated 2-ethylhexanol. The primary factors affecting maximum degree of mercerization are the type of cotton and the growth conditions. A higher degree of mercerization is achievable on cotton fibres that have a finer, uniform and circular cross section such as Egyptian and North American varieties. For a short-staple flat-type cotton, the effect is less pronounced. Cotton fabrics are mercerized in a grey or bleached condition or after boiling off in alkali. Mercerization is most efficient after scouring and bleaching; as size applied on warp yarn before weaving is removed by these processes, penetration of caustic soda is not hampered and the effect of mercerization is more pronounced. Moreover, the used-up caustic solution is not contaminated with sizes. For fabrics bleached in rope form, subsequent mercerization aids in removing crease marks. However, for reasons of economy (i.e. to save an additional drying cost) grey mercerization is frequently practised. The problem of wetting raw cotton materials is not easy to solve. A larger quantity of good wetting agent is necessary to assure uniform wetting. When the aim is to improve dyeability without much of an appreciable change in lustre, a less severe process called semimercerization or causticisation is carried out. The treatment may be carried out with about 15% caustic soda with or without tension, as the amount of shrinkage after the treatment is not very high. The cationisation during the mercerization process with an epichlorohydrin (2, 3epoxypropyl trimethyl ammonium chloride or EPTAC) resulted in a novel modified cotton cellulose and presents a new dimension for cotton pretreatment and finishing. The investigation of the chemical and morphological changes of such modified fabrics showed significant difference between the cationisation during and after mercerization.

It is assumed that the cationisation during mercerization is occurring in a more swollen state (i.e. a more open cotton structure), resulting in the bonding of a higher amount of an epihalohydrin. Additionally, the zeta potential and specific amount of surface charge clearly indicated that a charge reversal occurred as a result of covalent bonding of an epihalohydrin to the surface sites forming hydroxyl cellulose ether and the positive charge arising from its quaternary ammonium ion. This is the main reason for the enhanced efficiency and yield of reactions of water, ionic surfactant and direct dyestuff sorption.

## 4.2 Theory of Mercerisation

The hydroxyl groups on long cellulose chains attract the water molecules when cellulose takes up water, and the structure expands transversely as some of the mutual secondary valency linkages are replaced by water hydroxyl linkages. Now in an alkali solution, some of the hydroxyl hydrogen atoms are replaced by sodium atoms and a system of high ionic concentration is established. Owing to osmosis, water tends to enter this system, and more secondary linkages are broken and are replaced by linkages with alkali in water. When the alkali cellulose is washed, the sodium ions and the hydroxyl ions are diffused away. The osmotic pressure falls, and the cellulose gel contracts by virtue of its elasticity.

During this contraction, hydroxyl-hydroxyl linkages are reformed but not in such a great number as before, and the orientation of the micelles is more random. The greater proportions of free hydroxyl groups account for the increase in the reactivity and absorptive capacity. The decrease of this high absorptive capacity on drying, particularly at high temperature, is due to the formation of new secondary linkages on account of a greater amplitude of thermal vibrations of hydroxyl groups, as suggested by Urqhart. The swelling of fibres during mercerization is due to a molecular attraction with associated hydration. Since the alkali cellulose is more hydrated than the native cellulose, a maximum swelling concentration is the

result of attraction of an alkali cellulose in solution on one hand and free alkali on the other hand. The hydration of the cellulose increases with an increased fixation of alkali in a solution of rinsing concentration up to a certain limit, after which the free alkali exerts a dehydrating effect on the alkali cellulose to a greater extent.

The dissociation of alkali ions from the alkali cellulose compound corresponds to an absorption of OH– ions; in this manner, a negative charge results. The cellulose molecules repel each other and absorb water; the greater the absorption, the greater the charge. If, however, the dissociation of alkali cellulose salt is forced back, then there is reduction in the charge. Further, if the concentration of the electrolytes is high in swelling liquor, then the charge of cellulose particles is shielded by free ions and the force of the repulsion is diminished. Swelling is also due to an osmotic phenomenon. The fibre is surrounded by a hardened cuticle which acts as a dialyzing membrane to induce osmotic action, which is better explained with the help of Neale's theory

#### 4.3 Effects of Mercerisation

Mercerization changes the cotton fibre surface by removing part of the cuticle and decreasing the wax layer. Due to a high affinity, caustic soda solution penetrates into the crystalline region, in addition to the amorphous region of fibre. The intermolecular forces are weakened and the strength of the material decreases, but it recovers after deswelling and drying. Due to the distortion of the polymer network and the change in the crystalline structure, the mercerization process is irreversible. The changes in the physical and chemical properties are retained after scouring and bleaching. The changes occurring in textile substrates after mercerization can be summarized as follows:

- 1. Increased swelling and shrinkage
- 2. Change in cellulose structure (cellulose I to cellulose II)
- 3. Decreased crystallinity
- 4. Change in orientation
- 5. Increased tenacity
- 6. Increased lustre
- 7. Increased reactivity

- 8. Increased resistance
- 9. Enhanced dyeability
- 10. Enhanced enzymatic hydrolysis
- 11. Enhanced colour fastness
- 12. Removal of immature fibres

# 5.3.1 Swelling and shrinkage

Mercerization causes the swelling of the fibres. In a mercerization process without tension, the fibres, yarns and fabrics shrink lengthways considerably. The fibre, as well as the yarn diameter, increases with a corresponding decrease in metric count. Under tension, the shrinkage can be controlled. With mercerization, the convolutions or natural twists of the cotton disappear, and the morphological structure changes from a twisted ribbon-like structure to a tubular one. The characteristic hollow channel inside the cotton fibre or lumen almost completely disappears. Microscopic examination shows that up to about a 7% concentration of caustic soda, the convolutions disappear and the cross sections become elliptical, but there is no visible swelling. Between 7% and 11% caustic soda, a definite swelling occurs partly inward. At just over an 11% concentration, the inward swelling is complete with the lumens filled, but the primary wall is not fully extended. At 13.5%, maximum outward swelling takes place when the primary wall is fully extended. Finally, a further increase in caustic soda concentration shows no further change. The caustic treatment neither removes nor damages the primary wall, unless it was previously damaged. The cotton from different sources with different thicknesses of the primary wall will behave slight differently without modifying the general trend.

When a cotton hair is brought into an aqueous solution of sodium hydroxide of 40°Tw (18%), the cellulose begins to swell immediately. In few seconds, the hair is elliptical, with the major axis of the ellipse about 25–30% greater than the hair width of the corresponding collapsed hair. On further swelling, the section rounds off. The wall swells inwards until the lumen is practically eliminated. When the hair is washed with water, shrinkage of the cross section begins; on drying, a further and final shrinkage occurs. The shrinkage proceeds uniformly towards the centre, and the lumen does not recover its original size. Of great importance is the fact that up to a concentration of about 17.5%, the swelling action of the caustic soda follows the uncoiling, whereas at higher concentrations, the reverse is the case. As the concentration of

17.5% is the lowest at which any substantial increase lustre was observed, Marsh (1979) concluded that the production of lustre is connected with the uncoiling occurring after or simultaneous with swelling.

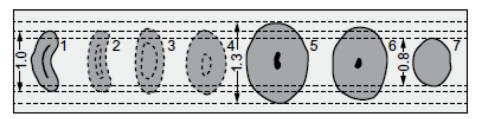


Fig. 4.1 Schematic cross-sectional view of cotton fibre at various stages of mercerisation.

Fig. 4.1 shows a cross section of cotton fibre at different stages: (1) before mercerization; (2– 5) increasing swelling with time after treatment with 18% caustic soda; (6) the washing stage after mercerization; (7) the final stage (i.e. after mercerization and caustic removal). Under optimum conditions, each cotton fibre may contract nearly 9% in length and swell nearly 150%. The disadvantage of this treatment is that the fibre becomes more compact in its swollen condition, which diminishes further penetration of caustic soda into the fibre; that is, the penetration slows down and the mercerization in the fibre's core is lower than that of its surface (M. More recently, swelling evaluations based on the precise measurements of cotton fabric with varying thickness have shown that the swelling would first increase moderately up to a sodium hydroxide concentration of 120 g/L (11% by weight) and then to a much greater extent between 120 and 180 g/L. The swelling value remains constant between 180 g/L (15–16% by weight) and 300 g/L (23-24% by weight). The maximum swelling of cotton fibres occur at about 150 g/L (13% by weight), while the maximum mercerizing effect takes place only at higher concentrations, indicating that the swelling is not the only determining factor. Rather, mercerization involves a largely internal modification in the arrangement of the fibrils. Many investigators found that the maximum contraction of cotton yarn takes place at a concentration of 20% caustic soda solution (45°Tw or 26.5°Bé) at room temperature. The change in the length of yarns is not a reliable measure of the mercerizing effect, as a great deal must depend on the structure of the yarn. The colour yield enhancement, strength increase and shrinkage effects arising from treating cotton-woven fabric with NaOH solutions of varying concentrations were determined. It was seen that the major effects from alkali treatment occur at NaOH concentrations of 15%. Higher concentrations of NaOH in the treatment solutions do not yield significantly different performance characteristics than those obtained at 15% NaOH. Therefore, companies that utilize alkali treatments for performance enhancement of cotton fabrics can consider lowering the concentration of NaOH in the treatment solutions to 15% in order to reduce costs without losing the desired property improvements.

# 4.3.1 Change in Cellulose Structure

The most important effect of mercerization is on the fine structure of cellulose fibres. An X-ray examination of native and mercerized cotton fibres shows that a change in the crystal structure takes place: the original cellulose I is transformed to a distorted crystal cellulose II on mercerization. The comparison of the unit cells shows that the planes carrying the long chains of  $\beta$ -glucose residues have been moved apart due to mercerization. The extent of the conversion depends on the tension applied during the process. Mercerization without tension causes a complete transformation, while with mercerization under tension, a mixture of the two forms is found.

In the early literature, the form of cellulose found in mercerized cotton was called 'hydrate cellulose' in the mistaken belief that water was involved in the lattice of the dried sample. It has now been renamed cellulose II after later work proved that water is not included in the structure. Structural changes associated with mercerization do not occur at one particular, well-defined concentration of alkali, but take place over a range of concentrations, referred to as the 'transition range'. Over this range, the cotton becomes gradually more accessible and hydrogen-bonds get disordered. With increasing alkali concentration, the crystal lattice gradually changes from cellulose I to cellulose II, and the levelling-off degree of polymerization is gradually decreased. Warwicker and colleagues listed several published results using various methods, concluding that at 20°C, the transition begins at about 95 g NaOH/L and is complete at 150 g NaOH/L.

The usually accepted unit cell for cellulose I has the dimensions: In comparison, the monoclinic unit cell of cellulose II has the dimensions: During the process, various intermediate complexes form between cellulose, sodium hydroxide and water. These 'soda-celluloses' occur at well-defined conditions of temperature and sodium hydroxide concentrations. X-ray diffraction studies show that a third form of cellulose, cellulose III, is produced by swelling in liquid ammonia and evaporation of the ammonia. However, this form is converted back to cellulose I in aqueous treatments.

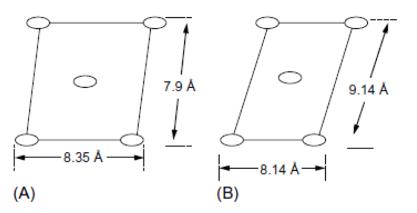


Fig. 4.2 Schematic lattice structures of (A) cellulose I (native cellulose) and (B) cellulose II.

Fig. 4.2 A and B shows the schematic diagram of cellulose I (native cellulose) and cellulose II crystal lattices.

## 4.3.2 Decreased Crystallinity

Swelling with caustic soda reduces the degree of crystallinity from 70% to 50% when cotton is thoroughly mercerized. The reduction is slightly less for the treatment under tension. The size of crystallites also changes.

## 4.3.3 Changed Orientation

The orientation of the crystallites, as measured by birefringence, changes little with mercerization under tension, but significantly under tensionless condition. The orientation of cellulosic chains decreases in the case of slack mercerization and increases when mercerization is carried out under tension. The change may arise from variations in the spiral angle of the fibrillar elements and not necessarily from variations in orientation of the crystallites within the fibrillar structure.

#### 4.3.4 Increased Tenacity

Mercerization tends to increase the tenacity of cotton fibres. The two factors that make this true are the changes in both the spiral angle and orientation, but the explanation is controversial. The increase is also attributed to a strengthening of weak points along the fibre. The weak points include internal strains (i.e. spiral reversals), the places of low cross-sectional areas and the places of high distortions. The other important factors may be the crystallite length and the degree of crystallinity, as well as the removal of cellulose of a very low degree of polymerization. The increase in tensile strength is significantly higher on fibre bundles than in the case of yarns and fabrics. The elongation at break decreases, and the Young's modulus increases significantly on mercerization under tension. When mercerization is carried out

without tension, the value of elongation at the break is three times the value of the unmercerised material, thus conferring elastic properties to cotton fibres. It is the basis of the preparation of stretch materials.

$$a = 8.35$$
,  $b = 10.3$ (fibre axis),  $c = 7.90$ (Å) and  $b = 84$  degrees

$$a = 8.14$$
,  $b = 10.3$ (fibre axis),  $c = 9.14$ (Å) and  $b = 62$  degrees

Mercerization is done in yarn and fabric form. It is never done in fibre form, as in that case no tension control is possible. Moreover, the cylindrical mercerized fibres are difficult to spin. Due to fibre swelling, the fibres in the yarn come closer to each other and become denser, thereby increasing fibre-to-fibre cohesion with a consequent increase in yarn strength. In other words, the increase in yarn strength is largely due to the consolidation of the structure rather than an increase in the strength of the individual fibres. The twist also plays an important role: the increase in strength is high for low twist yarn and low for high twist yarn.

#### 4.3.5 Increased Lustre

If the shrinkage is controlled by applying tension during or immediately after the caustic treatment, the fibres acquire a silky lustre, a valuable property of mercerized cotton. Due to the change from an elliptical to a uniform cylindrical cross section and untwisting of the fibres, mercerized cotton is close in lustre to cylindrical man-made fibres. The lustre depends on the quality of raw material; long staple fibre acquires a greater lustre than short-staple fibre. In a similar way, twisted double yarn is more lustrous than non-twisted loose yarn.

When mercerization is carried out without tension, no increase in lustre is observed. The lustre obtained by impregnating and washing under tension is the same as that obtained by loose impregnation followed by washing under tension, but a higher force is required in the latter case. Tensionless impregnation and washing followed by stretching shows inferior results. In most mercerizing processes the material is allowed to shrink in the alkali in order to assist thorough penetration, and the tension is applied later, either before or during the removal of the caustic soda. To retain an optimum level of lustre, it is necessary not to release tension before the caustic soda concentration is lowered to about 60 g/L. For this purpose, the first rinsing must be carried out under tension. The presence of short fibres may hamper lustre and should be removed by singeing before mercerization.

Heap attributed the lustre of mercerized cotton to the fibre's circular cross section as compared to the unmercerised fibre's flat kidney shape without explaining why this change should cause

lustre. Heap also stated that because of the thicker fibre wall after mercerization, the path length of the light through the fibres is increased, and therefore the unmercerised fibre appears paler. However, Goldfinger in a communication discussed some experiment data which gave a very strong indication that the major effect of the colour change on mercerization is due to the change in the efficiency of internal scattering.

### 4.3.6 Increased Reactivity

Since the degree of crystallinity decreases with mercerization, the resulting fibres have a greater absorptive capacity and are more reactive to chemical agents, resulting in the acceleration of hydrolysis under the action of acids and damage by oxidizing agents.

#### 4.3.7 Increased Resistance

Mercerized fibre is more resistant to light and weather effects than non-mercerized fibres. Ordinary commercially mercerized bleached cotton cloth was found to resist deterioration upon exposure under glass to sunlight or to the full effects of weathering, better than unmercerised cloth. On the other hand, in fluidity tests by both the cupra-ammonium hydroxide and cupriethylene diamine hydroxide methods on the same exposed materials, higher fluidities for the mercerized exposed samples indicated greater degradation (cellulose chain breakdown) for the mercerized cotton. This behaviour is consistent with reports in the literature from which one can conclude that mercerized cotton will withstand oxidation and hydrolysis with less of a loss of strength than unmercerised cotton, but here also it showed higher fluidities (greater degradation) after such treatments than unmercerised cotton. Mercerized cotton is sufficiently superior in strength retention to suggest that advantage could be taken of it to obtain cotton end products of longer service life where deterioration is due mainly to the effects of sunlight; its greater retention of strength under a chemical attack supports this view

### 4.3.8 Enhanced Dyeability

Mercerized material requires less dye to obtain the same colour strength than that of unmercerised material. The dye consumption is reduced by about 10–15% in light shades and about 25–30% in deep shades. The uniformity, purity and brilliancy of the shades are also higher due to the change of optical properties of the fibre by mercerization. The increased depth of shade of mercerized cotton has been attributed to the following.

- 1. Optical effects arising from the modifications of fibre size and shape
- 2. Changes in internal light scattering

### 3. Actual increases in dye content due to the increased amorphous part of the fibre

About half the total dye savings is attributed to optical effects. The change in pore volume and reduction in lumen diameter are primary causes of the consequent improvement in colour yield and reduced light scattering within the fibre. Mercerization thus lowers the average dye cost savings at are about 40% and 60% at 2% and 6% depth respectively with same dyes.

### 4.3.9 Enhanced Enzymatic Hydrolysis

Recently, there has been interest in using enzymes in finishing treatments for cotton textiles. The effect of enzymatic hydrolysis and acid hydrolysis on slack and tension mercerized cotton yarn has been investigated and compared to the effects on the non-mercerized starting sample. Enzymatic hydrolysis was dramatically enhanced when the yarn was slack mercerized. Disintegration into short fibres occurred after 16 hours incubation time while the tension mercerized sample did not disintegrate within 48 hours, although its tensile strength decreased distinctly faster than the non-mercerized sample. It appears that the higher accessibility and decreased crystallinity of the cellulose structure after slack mercerization are dominating factors for the acceleration of enzymatic hydrolysis. In the case of acid hydrolysis, the retention of tensile strength proved to be superior for the tension mercerized yarn, followed by the slack mercerized sample and then the non-mercerized counterpart.

#### 4.3.10 Enhanced Colour Fastness

The colour-fastness of dyed mercerized materials is higher than that of dyed unmercerised materials. This is probably due to the swelling and opening of structure with a consequently higher penetration of the dyes.

#### 4.3.11 Removal of Immature Fibres

In addition to the enhancement of lustre and dyeability, mercerization removes crease marks and neps of immature fibres which otherwise appears as white specks after dyeing. The dead fibres are underdeveloped and appear as flat or slightly twisted tapes. They are non-crystalline, their convolutions are sometimes absent, their cell walls are extremely thin and the lumen is collapsed; hence, they do not contain dye to the same extent as matured fibres. Cotton print cloth containing both immature fibre neps and process neps composed of torn and ruptured mature fibres was treated with sodium hydroxide of mercerizing- and sub-mercerizing strength and liquid ammonia. This study illustrates that neppy cotton fabric can contain two types of neps: those composed of immature fibres and those composed of thick-walled, thin-walled mature fibres that were tangled and occasionally damaged during processing. Mercerization

swells both immature fibre neps and process neps and promotes almost complete coverage of all the visible neps when the fabric is dyed with a variety of direct dyes. Sub-mercerization strength (4%) sodium hydroxide is almost as effective as full mercerization strength sodium hydroxide in reducing the number of visible neps in direct-dyed fabrics. An SEM examination of neps in sub-mercerized fabrics showed that this treatment appears to affect primarily the superficial fibres in the neps while leaving the bulk of the compact interior mass almost undisturbed. Although the surface immature fibres were not as completely swollen by the lower strength caustic solution as they were by the mercerizing strength solution, the degree of swelling appeared to be sufficient to promote visual nep coverage after dyeing.

### 4.4 Classification Of Mercerisation Processes

A unique classification of mercerization processes is as follows. Classification according to the form of the product

### (1) Yarn mercerization

- (a) Batch: hank mercerization, cheese mercerization
- (b) Continuous: single-end mercerization
- (c) Tow mercerization
- (d) Warp mercerization

#### (2) Knit mercerization

- (a) Open mercerization
- (b) Closed mercerization (round mercerization and tubular knit mercerization)

### (3) Cloth mercerization

- (a) Chain mercerization (stenter mercerization)
- (b) Chainless mercerization (Roller mercerization)
- (c) Batch up mercerization

### Classification according to mercerizing conditions

- (1) Water content
- (a) Dry mercerization

(b) Wet mercerization
(2) Tension
(a) Fixed-length merce

- rcerization
- (b) Tension mercerization
- (c) Tensionless mercerization
- (3) Alkali concentration
- (a) Low-alkali mercerization
- (b) High-alkali mercerization
- (c) Two-step mercerization
- (4) Temperature
- (a) Ambient-temperature mercerization
- (b) High-temperature mercerization
- (c) Low-temperature mercerization

# Classification according to stage of treatment

- (a) Grey mercerization
- (b) Pre-dyeing mercerization
- (c) Post-dyeing mercerization

# Classification according to the number of treatments

- (a) Single mercerization
- (b) Double mercerization

# Classification according to the type of alkali used

- (a) Caustic soda mercerization
- (b) Ammonia mercerization
- 5.5 Yarn mercerization

Mercerized yarns are used for:

- (1) Sewing and embroidery thread, where lustre, strength and smoothness are required
- (2) Top-quality knitted fabrics
- (3) Weaving, especially high-quality, expensive shirtings.

Before mercerization, the yarn should be singed, otherwise the projected fibres will hamper yarn lustre. The mercerization may be carried out on dry or wet yarn, the latter coming from a preliminary wetting or scouring process. However, the excess water must be removed uniformly from the material to prevent any dilution of the caustic solution. Irregular distribution of residual water will cause uneven swelling and consequently uneven dyeing.

There are three main types of yarn mercerizing methods:

- (1) Hank mercerizing
- (2) Warp mercerizing
- (3) Cone-to-cone mercerizing.

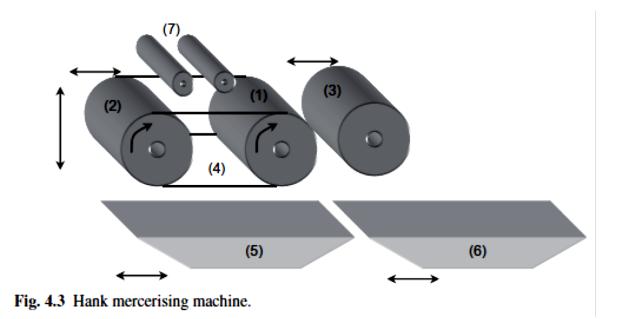
Most European processors employ the batch wise-hank mercerizing process, while warp mercerizing used to be known as the 'American process' not only because of its widespread use in the United States, but also because it is a continuous, high production technique. The main disadvantage of hank mercerizing is the necessity for reeling the yarn into hanks for treatment, then rewinding afterwards for subsequent use. With the advent of automatic controls and robotic systems for loading and unloading, hank mercerizing has become a high production operation, rivalling the output of warp mercerizing. Both methods can give between 100 and 200 kg per hour per machine.

### 4.4.1 Warp Mercerization

Warp mercerization is done in developed countries not only for warp yarn but also for knitting yarn. The process ensure uniform mercerization followed by uniform dyeing, though the lustre may be less than that obtained in hank mercerization. The warp mercerization machine resembles an ordinary warp dyeing machine, which is a series of as many as 18 boxes or compartments, each of which is fitted with guide rollers and squeeze rollers. The latter hold the yarn at a proper tension as it passes from one bath to the next. The first two or three boxes contain boiling water that is with or without dilute caustic soda solution. The warp after passing

through these boxes is then squeezed and passed through three boxes containing caustic soda solution of mercerizing strength allowing a contact time of 2–5 minutes. After the first impregnation, slightly more tension may be applied with the help of rubber-coated rollers, the speed of which determines the extra tension. These rollers also assist penetration. The washing is done in three or four subsequent compartments working countercurrent to the liquor flow. The washing starts with a hot (50–60°C) dilute (4.5%) caustic soda solution and finishes with cold water. After a thorough squeezing, the material is allowed to relax from the previously applied tension and is sent to one or more souring chambers, where it is treated with dilute sulphuric or hydrochloric acid for a period of at least 1 minute. The last five or six compartments are designed for the final washing with water of increasing temperature up to 50°C; the first chambers may contain sodium carbonate or ammonia to neutralize the last traces of acid. Finally, the yarns are dried preferably by hot air instead of cylinder, as the latter are apt to produce a harsh feel.

The warps, good quality combed and singed single or double cotton yarn, are about 6000–10,000 yards long with 300–400 parallel threads wound into cylindrical balls, all are wound approximately under same tension. The yarn comes evenly in contact with alkali, whose concentration must be uniform and high enough to ensure required degree of swelling. Warp mercerizing is essentially a large-scale process. In the United States, fine warps of 2/60s count are mercerized in these machines in large quantities. The finer yarns of 2/100s and 2/120s count may also be processed successfully.



### 4.4.2 Hank Mercerization

The yarns used as sewing threads, embroidery yarns, etc., may be mercerized in the form of hank. The hanks (3–5 kg) are cross-wound at a uniform tension and spread on to two parallel rollers of the hank mercerizing machines (Fig. 4.3). One of the rollers is fixed (1) while the other (2) can be moved horizontally to stretch the yarn. Hank yarns (4) are placed on the roller pair (1 and 2). An additional squeezing roller (3) is fitted near the fixed roller and is pressed against it while squeezing. The rotation of the roller cause uniform impregnation with the liquor. The machine is provided with two trays carrying caustic liquor (5) and souring acid solution (6). The trays can be placed below the rotating rollers or away from them as required. Water can be sprayed on the yarn during washing from the water sprayer (7) fitted above. The yarn is allowed to shrink on immersion in order to facilitate liquor imbibition, the tension being applied later on the thoroughly impregnated hanks. A typical mercerizing cycle in yarn mercerizing has the following steps:

- (1) Loading of material on the rollers in rest position
- (2) Lowering of rollers
- (3) Pre-tensioning of yarn by the movable roller
- (4) Movement of caustic tray below rollers
- (5) Yarn impregnation in caustic lye for 1–1.5 minutes
- (6) Free shrinkage
- (7) Tensioning of yarn while immersed in lye
- (8) Squeezing of yarn by pressing squeezing roller onto fixed roller
- (9) First wash and tensioning
- (10) Movement of souring tray below rollers and immersion of yarn
- (11) Second wash
- (12) Third wash
- (13) Final wash
- (14) Squeezing
- (15) Lifting of rollers

# (16) Unloading the hanks

In other types of machines, caustic liquor is sprayed from the top while the hanks are rotating and the liquor is collected on a tray placed below the rotating rollers from which it is pumped back to the sprayer.

### 4.4.3 Yarn Strength after Mercerization

Yarn mercerization aims at increasing dye uptake and light reflection properties. But if all technical aspects are not considered, there may be a reduction in yarn strength and an increase/decrease in yarn elongation. McDonald et al.'s investigations on six varieties of cotton fibres showed that mercerization in a slack and stretched condition results in an increase in the evenness of all yarn properties and a reduction in their standard deviation. However, moisture absorption, fibre and yarn extension and swelling in slack mercerization are higher than when the yarns are treated under tension. Yarn and fibre strength in tension mercerization are 60% and 53% higher than those in slack treatment, respectively.

A study of the mercerization of open-end yarns showed that the strength of the yarns with a minimum twist (twist factor 4.25) may be improved by 52.8% in tension mercerization and 46% in slack mercerization. It was also found that although the degree of mercerization and improvement in the strength of low-twist yarns are higher than those of high-twist yarns, yarns with an optimum twist always possess better strength. In other words, the effect of mercerization on improving yarn strength is not comparable to that of the twist. It was also observed that twofold (and multi) yarns may be mercerized better than single ones, provided that the twist direction of plying is opposite to the single yarn spin. This is because the movement of fibres within the folded yarns happens more easily, hence they may be better arranged in the yarn structure.

A study on mercerization of cotton yarn showed that:

- 1. Cylinder pressure has a considerable effect on the strength of all kinds of mercerized yarns in the range studied. It is optimized at 0.595 MPa and is independent of yarn characteristics.
- 2. The warm rinsing temperature and caustic soda bath concentration, especially for thicker yarns with a higher number of plies, have a noticeable effect on the mercerized yarn strength.
- 3. The duration of their immersion in the caustic soda bath has a diverse effect on carded and combed yarns. By increasing the immersion time, the strength of combed/carded yarns increases or decreases, respectively.

- 4. The effect of the wetting agent concentration is elevated by decreasing the yarn's linear density and/or increasing the number of plies.
- 5. The effect of the cold rinsing temperature is more obvious in higher quality yarns and fibres. The effect of the cold rinse temperature on Egyptian and Uzbek cotton combed yarns is more obvious than for carded yarns spun from Iranian cotton fibres.
- 6. Statistical analysis shows that fibre type, yarn structure (carded/combed) and the number of plies have a significant effect on the optimum settings of the mercerizing machine in achieving the maximum strength of mercerized yarn, while the linear density of single yarns and the mercerizing machine type have no significant effect. The orientation, lustre, tensile strength, elongation, work of rupture and decreased initial modulus and abrasion resistance of yarn increase on mercerization. A comparative study with yarns spun in different ways showed greatest increase in tensile strength (49%), elongation (43%) and work of rupture (25%) on mercerized ring spun combed yarn. It also had the greatest decrease of initial modulus (48%) as compared to those of mercerized ring-spun carded yarn and open-end spun yarns. Mercerized open-end spun yarns gave the lowest increase of the degree of orientation (4%), tensile strength (25%), elongation (25%), work of rupture (9%), and the greatest decrease of abrasion resistance. The changes in the case of mercerized ring-spun carded yarns were in between.

### 4.4.4 Fabric Mercerisation

The fabric after scouring is efficiently squeezed and with water content of 60%, it enters into a large caustic bath containing 300 g/L caustic soda in the usual process or 530 g/L in addition to mercerization. The exchange of caustic soda and water takes place by diffusion, which is accelerated by an increase in temperature due to a decrease in viscosity. Due to the rapid and extensive swelling of cotton fibres during conventional mercerization at cold temperatures, the structure is compacted at the surface of mercerized yarns or fabrics, and further penetration of caustic soda is almost impossible. This results in the non- uniformity of mercerization effects. Because of the lower level of swelling, mercerization at elevated temperatures may allow a more even penetration of caustic soda solution to come into the inner parts of yarns and consequently better uniformity of the alkali treatment. However, the conversion of the crystalline structure from cellulose I to cellulose II is retarded at high temperatures, and the yarn assumes a sheath-core appearance. A reasonable two-step approach is to use a hot caustic soda solution for better penetration into the textile structure and subsequently to allow the

impregnated material to cool to room temperature in order to have a good swelling value. Hot mercerized cotton shows more uniform colouration than cold mercerized cotton. The total dye uptake is better, but the colour yield is less pronounced due to less peripheral dye absorption. A better wet crease recovery is achieved by hot mercerization, which makes subsequent scouring and bleaching easier and faster. It is claimed that the scouring step may be eliminated or simplified by inserting a steaming step (10 minutes under atmospheric pressure or 5 seconds at 130–140°C under pressure) between hot alkali saturation and stabilization. The de-sizing process could also be eliminated; however, it should be remembered that the chances of alkali degradation of cotton by concentrated sodium hydroxide solution are higher in the presence of air at high temperature. Technically, hot mercerization is carried out at 70–100°C for 4–60 seconds, which leads to a uniform high swelling of cotton fibres with a softer feel. One process of hot mercerization is to saturate cotton piece goods in caustic lye (50–58°Tw) for 5 seconds at 97°C under relaxed fabric conditions. The goods are subjected to a controlled hot stretching after caustic saturation. The hot stretched fabric is cooled under proper controls below 25°C followed by successive tension-controlled washing called stabilization (up to caustic soda concentration of 60 g/L) and washing under normal conditions.

To achieve a high degree of swelling, a caustic concentration of more than 270 g/L and preferably 350 g/L is necessary. In the conventional process, a concentration of 300 g/L is taken in a longer bath, with diffusion aided by intermediate nips, when the exchange takes place. In the addition process, caustic soda at a concentration of 530 g/L is added to wet and squeezed fabric. Due to higher concentration and higher temperature, the penetration inside the fibre is quicker and a high degree of mercerization is achieved. The optimum time necessary is 35-50 seconds for cold mercerization and 25-35 seconds for hot mercerization at 70°C, depending on the type of cotton, weave and fabric density. Due to the high degree of swelling of the cotton fibres, the fabric gets fixed in its dimension, leading to a low degree of residual shrinkage. When the fabric is not allowed to shrink, the swelling of fibres leads to an internal tension. When the impregnated fabric is stretched, the fabric shows a rubber-like elasticity and at levels of 5–10% stretch, there is a linear relation between the stretch force and the resulting elongation. However, the higher the stretch, the poorer the dimensional stability. For the continuous mercerization of fabric, mercerizing ranges are employed. These consist of a sequence of machines for impregnation, washing under tension, removal of alkali, neutralizing, washing, squeezing and plaiting of fabric. Such ranges can be classified into three types as follows:

- (1) Stenter-type or chain mercerizing machine
- (2) Chainless mercerizing machine
- (3) Chainless pad-less mercerizing machine

The chain mercerizing machine runs at about 40 m/minute, whereas the chainless pad-less machine runs at 18–20 m/minute. In the latter, however, two to four ends of fabric can be run at a time, thus giving an equivalent or higher productivity compared to the former. Direct labour employment is three to four workers per shift. The washing liquor collected from the mercerizing machine is usually of 9–10°Tw strength, and this is sent for caustic recovery. The net consumption of caustic soda should not be more than 10% on the weight of material. Mercerization can be carried out either in the beginning (grey) or in between (scoured) or at the end (bleached) of a bleaching sequence. For carrying out mercerization in the last two stages, the cloth is opened on a scutcher and usually dried. Today, mercerizing units are usually equipped with a liquor concentration regulator, measuring unit, cooling unit, lye recovery unit, purification unit, a reciprocating metering pump and another unit to dissolve caustic soda. Generally, a mercerizing machine consists of few sections which will have different tasks according to the process technique:

- 1. Mercerizing section: impregnation with caustic soda
- 2. Intermediary squeezing aggregate: dividing off the mercerizing section against the stabilizing section
- 3. Stabilizing section: water treatment and thinning down concentration of the lye
- 4. Intermediary squeezing aggregate: dividing off the stabilizing section against the washing section
- 5. Washing section: washing off the alkali and neutralization (if necessary)

A comparison between chainless and chain-type mercerizes is given in Table 4.1

# 4.5 Chain Mercerizing Range

On a chain mercerizing machine, the fabric is passed through the nip of a pair of rollers into the caustic soda solution. On emerging, the fabric is threaded around a dozen of idling rollers to allow time for the caustic soda to penetrate into the fibres. After plunging through a second trough of alkali and through the nip of a pair of squeezing rollers, the fabric is fed into the clips on the two chains of a stenter frame in which it is firmly held. Warp tension is applied by running the second nip at a faster speed. Tension in the weft direction is applied by the chains of the stenter frame, which slowly move away from each other as the fabric proceeds through the stenter. As the fabric approaches its fully stretched state, it is showered with hot water to remove caustic soda, which should be brought down below 5% before the tension is released. During washing, it is desirable that the original woven width is maintained so that maximum dimensional stability is achieved. In this machine, the mercerizing lye causes fabric shrinkage. It is therefore necessary to arrange the machine compartments in such a manner that the finished fabric satisfies the dimensional requirements in all respects.

Table 4.1 Comparison of chainless and chain-type mercerisers

Chainless type		Chain type
1	Possible to process light- to heavyweight fabrics simultaneously	Not possible
2	Lower processing cost	Higher processing cost
3	Low investment cost	Relatively higher
4	Low maintenance cost	Relatively higher
5	Low fabric speeds	For speeds from 40 to 120 m/minute
6	Compacting of selvedge	No compacting
7	Restriction in shrinkage	Free shrinkage in mercering and
		impregnation section
8	Control over fabric is less	Better

### 4.5.1. Fabric Entrance Section

The fabric is taken off from its plighted condition through a set of automatic cloth guiders, guide rollers and tension bars.

### 4.5.2 Impregnation Section

In the case of chain-type mercerizing machines, impregnation with the mercerizing lye is carried out in suitably dimensioned padding mangles. The mercerizing lye, with an optimum concentration of caustic soda, is continuously fed to the trough of the padding mangle. The single web of fabric gets a dip in the lye and is then passed through the padding mangle, where the excess lye is squeezed out from the impregnated cloth. Small machines have one padding mangle whenever more mercerizing production is required, but the passage through one padding mangle is not sufficient to make up the lye action time. A second padding mangle is therefore introduced in the system.

Usually, two lye pads give the required production but for still higher production speeds, a third impregnation padding mangle is necessary in order to decide on the expected production before the installation of the mercerizing range.

# 4.5.3 Width Stretching and Stabilizing Section

In the last step, the fabric that shrunk due to a caustic treatment is to be recovered by stretching the impregnated cloth at the stenter. The required length of the stenter depends on the production needed and is to be decided upon beforehand. A weak lye is sprayed on the fabric as it is being stretched in the stenter frame. Later, still weaker lye may be sprayed on the cloth.

### 4.5.4 Steam Recuperation Zone

After squeezing the fabric at the end of the stenter through the squeezing mangle, the fabric is introduced into the lye recuperation section, where nearly boiling water recovers most of the lye in the recuperation washing compartment with top and bottom rollers.

### 4.5.5 The Washing Section

The remaining portion of the lye is washed out of the cloth in the washing section having the requisite number of washing compartments. The chain mercerizing machine has one inherent disadvantage. As the force for keeping the material under tension acts mainly on the outer edges and the line of force diminishes towards the middle, a greater elongation takes place at the edges as opposed to the middle of the fabric. Prior to gripping on the chains the warp density, threads per centimetre over the whole width of the fabric is constant, after stentering it is less at the edges than in the middle. Different measures were attempted in order to avoid this considerable disadvantage, but none were really successful. Therefore development took place from chain mercerizing machines to chainless mercerizing or roller mercerizing machines.

## 4.6 Chainless Mercerizing Range

In a chainless mercerizing machine, the fabric after padding is passed over a set of expander rollers, which are bowed rollers and stretch the width of the fabric, thereby maintaining maximum width. For many types of fabric, the machines provide satisfactory mercerization more economically than chain machines. With expander rollers, the fabric width is determined by the tension on the warp and cannot be readily adjusted. The lustre of the fabric is less than that attained by a chain merceriser. The zone of the chain mercerizing machine in which the chain is applied provides the conditions for good maintenance in width. However, the effectiveness of lye removal is questionable; though a good fabric width results from the chain guiding, the anticipated stabilization may be largely lost due to the excessive lye content upon

leaving the chain. Improvements can be realized on chainless machines by using more effective expanders, preferably in the stabilizing section, together with more complete lye removal in the guided fabric width.

# 4.7 Chainless Padless Mercerizing Range

While in other fabric mercerizing machines, the fabric is allowed to shrink after a caustic treatment followed by stretching. However, in a chainless pad-less mercerizing machine, the impregnation and washing are done under tension, and the fabric is not allowed to shrink. Instead of forcing caustic liquor into the fabric by a heavy mangle as in other fabric mercerizing machines, the liquor is absorbed from a trough by the fabric, which is held under tension by a series of rollers that are touching each other. The fabric must be in a very absorbent state, and a highly efficient wetting agent is to be used in the trough. For fabrics with long staple fibres, a longer time of impregnation is allowed as they have a greater tendency to shrink, resulting in a slower rate of penetration.

In the impregnation section, there are two rows of strong rollers, one above the other, and the fabric runs alternately around a top and a bottom roller. The top rollers are covered with rubber and are carried by the bottom row of rollers, which runs on ball bearings. The fabric runs under tension over the rollers, and the warp tension can be controlled by the drive. The first two lower rollers carry the fabric through the caustic solution, which comes through a two-supply pipe immediately above and in front of the first two top rollers and flows continuously over the fabric and into the trough. The fabric runs about 3–4 metres during impregnation, and a further passage of about 5–6 metres around the remainder of the rollers for the alkali to penetrate the fabric. The fabric then passes through a mangle at a pressure of about 5–8 tons depending on the type of the fabric. A further run of about 8 metres takes about 40–60 seconds between the start of the immersion and washing.

The fabric then passes through the washing compartment of a similar assembly of rollers and washed under tension with hot water. A further mangling is done after passing through a series of sprays and troughs, then the fabric passes to a recuperator with five compartments, where steaming reduces the amount of alkali in the fabric to very small proportions. In the final compartment, the fabric is sprayed with fresh water and then heavily mangled to make it ready for plaiting on to wagons. The Benninger pad-less chainless mercerizing machine runs at a speed of about 9–26 yards per minute depending on the type of the fabric. Superimposing

fabrics or running them side by side can increase production. For heavy fabrics, the machine should be run at a slower speed.

## 4.8 BEN-DIMENSA Mercerization Machine (Benninger)

The dimensional stability is the most important objective of present-day mercerization, and it can be achieved with the DIMENSA® by the combination of a chainless positive guide system and a stenter frame (Benninger, 2015). The intensive impregnating zone is followed by the reaction zone. In this area, fabric guidance is based on the chainless principle. At the start of the stabilizing zone that is immediately after the mercerizing section, there is a stenter section using the chain principle. Ultimate stabilization is in a conventional, highly effective stabilizing compartment with chainless guidance. The final section comprises a high-efficiency washing and neutralizing zone consisting of EXTRACTA® or TRIKOFLEX® compartments. Here is the sequence of operations:

Chainless impregnation and reaction zone  $\rightarrow$  initial stabilizing zone with stenter  $\rightarrow$  final stabilization in chainless compartment  $\rightarrow$  efficient washing and neutralizing zone.

Despite logistical difficulties, only the wet-on-wet process is practiced because it keeps costs low. However, this imposes new demands on process engineering and machine design alike.

## 4.8.1 Impregnation/Reaction Zone

With lightweight fabrics, a high liquor exchange is attained so that both limits are maintained (grams NaOH per kg fabric and °Bé) with a short impregnation zone. The same concentration is used with heavy fabrics, though excess caustic soda solution is made available to the cloth during the reaction phase. The fabric is repeatedly impregnated with caustic soda by additional spray pipes in the roll passages. Squeezing is achieved by the weight of the rolls and the cloth tension; this ensures that both the lye content and the concentration in the liquid film satisfy the requirements (200–240 g caustic soda per kg fabric and 28°Bé, respectively).

### 4.8.2 Stabilization Zone

With the classic DIMENSA concept, the lye extraction process is initiated by intensive spraying with a weak lye after the transfer compartment in the middle of the stenter frame. Heavy cloths are sprayed immediately following the stabilization compartment after the transfer compartment (i.e. preliminary lye) extraction. In this way, the same lye extraction levels are reached at the end of the stenter for light- and heavyweight fabrics. With an unchanged process, the lye concentration, the combined impregnation and the reaction

compartment can be flooded or drained very quickly. This way, optimal impregnation conditions can be provided by a single impregnation for lightweight fabrics and a double impregnation for heavy cloths, allowing mercerizing at the same speed. Our experience indicates a weight range around 250–300 g/m2 for changing the impregnation conditions.

### 4.9 Slack Mercerisation

In recent years, commercial cotton yarn or fabric is mercerized without tension to impart stretch properties, such as high elongation and high elastic recovery. These materials are useful for casual wears, skirts, boat covers, etc. Slack mercerization is simple. The cotton fabric being allowed to shrink without tension in a solution of about 10–15% caustic soda solution at 30°C. However, the actual processing procedures can be varied, and those will have considerable influence on the stretch qualities in the resulting fabrics. For some purposes, the weft only is allowed to shrink. The warp will either be held taut or allowed to shrink, followed by stretching in the warp direction.

# 4.10 Mercerisation of knitwear

While woven fabric mercerizing is a process that has been around for 140 years, knit goods mercerizing only started about 20–30 years ago. It has been established that the quality improvements due to mercerizing can be even greater on knit goods than on woven fabrics; lustre effects and dimensional stability obtained are magnificent. Perfectly mercerized knit goods are characterized by:

- Pill-free surfaces
- Increased dimensional stability in both length and width
- Considerably lower residual shrinkage values than with unmercerised cotton
- Permanent lustre
- Excellent brilliance and depth of colour
- Silken lustre
- Lower tensile strength loss during high-performance finishing
- Improved wearing properties

The knitted fabric must be fed into the impregnating part of the machine completely uncurled, under guiding that is controlled both lengthwise and widthwise. This requirement must be met

without compromise, but it can easily be satisfied with a combination of pneumatic and mechanical expanding devices and precisely controlled draw nips. The guiding elements should be arranged so that the last fabric expanding and guiding is applied as close as possible to the liquor immersion point. As soon as the fabric is wetted with liquor, the problem of edge curling disappears. The circular knitted fabrics can be mercerized in the existing knit mercerizing machines after slit opening. However, some major problems of such fabrics cannot be solved with existing machines, namely, selvage mark, selvage curling and selvage compacting. Mercerization may give rise to different warp thread densities between the middle of the fabric and the edge zones. In chainless machines, these differences are due mainly to the loss of fabric width during mercerizing, with the edge zones taking the whole of the shrinkage and entailing an inevitable increase in the number of warp threads at the edges. The more the fabric is stretched across the width, the higher the difference in the warp density, though in practice a certain difference can be tolerated. The most attention is to be given to the patterned goods, especially with circle effects.

Controlled tension is an important factor for optimum tube mercerizing, lustre and uniformity. During the critical phase of stabilizing, when the caustic is washed out, the tubes must be stretched to their natural tubular condition. An even stretching is achieved by a step-less adjustable circular expander, which is placed inside the fabric tube vertically in the centre of the washing tower. Spray rings are arranged around the expander and equipped with flat spray nozzles through which hot water is sprayed at about 80°C. The circular expander is held vertical by four concave rollers. The base is designed as a floating body, and the expander stands in the stabilization bath, its weight nearly compensated by its buoyancy.

### 4.11 Addition Mercerisation

The most feasible processing sequence incorporating the mercerizing process could be obtained if mercerization were to be achieved between scouring and bleaching or in subsequent single-stage processes. This gives the advantage of a clean and absorbent fabric and reduces any lye contamination problems when concentrating lye by evaporation.

The new mercerizing machine exhibited by Kusters at ITMA 1995 aimed to achieve a high degree of mercerization to enhance the dyeability, dimensional stability and tensile strength, as well as lustre and appearance. This is achieved by optimization and reproducible control of the process parameters including temperature and concentration of alkali, time of action and tension. Mercerization is carried out mostly between the stages of scouring and bleaching or

after a single combined step of scouring and bleaching. The primary factors are the type of cotton and the growth conditions. In the case of cotton products, perfectly pre-scoured and squeezed fabric with a residual moisture content of 50–70% is fed into the mercerizing machine in order to save energy for an intermediate drying process. In the mercerizing section of the line, the fabric is brought into contact for a dwell period as constantly as possible with a mercerizing liquor concentration (300 g of NaOH/L or 30°Bé). The number of squeeze points on roller mercerizing machines, the liquor temperature, the quality and quantity of wetting agents and the fabric absorbency all have a decisive effect on the dwell time required for obtaining approximately identical caustic soda and surrounding liquor concentrations on the fabric. Substance interchange and diffusion processes are dependent on concentration gradient and liquor viscosity and temperature. In this way, adequate cotton swelling for the mercerizing process can be achieved after only 20 seconds dwell time.

Comprehensive trials with various machine variants and a relevant evaluation of the particularly interesting textile technical parameters during the mercerizing process led to the addition mercerizing process. After carrying out a pre-scouring process with one or more scouring sections depending on the specific conditions, the fabric passes through a high-performance squeezing unit. Relative to the squeezing effect, caustic soda in the required concentration and quantity is applied uniformly to both sides of the fabric with the Flexnip (Küsters) addition unit, setting up the required mercerizing lye concentration in the fabric after a short diffusion passage. According to the large-scale trials available to date, the high degree of concentration of the added lye has the following main advantages over the conventional mercerizing process:

- 1. A high concentration gradient between the water carried along with the fabric after the highperformance squeeze unit and the strong lye added in the Flexnip result in significantly accelerated diffusion, with the necessary dwell and mercerizing times being reduced to about 10 seconds.
- 2. The entire lye circulation system in the earlier mercerizing section with lye vats, containers, pumps, spray tubes, etc., is eliminated. This section can be significantly shortened on account of the short dwell times required.
- 3. The addition of lye in a considerably higher concentration than before gives rise to immediate, practically spontaneous fibre swelling, resulting in the same mercerizing effect with up to 25% less caustic soda usage/kg of fabric than with the processing method hitherto

employed, resulting in a significant savings in water, steam and chemicals, plus a considerable reduction in the ecological problems posed by the mercerizing process.

### 4.12 Control of Caustic Concentration

During mercerization, the fabric is first immersed in a caustic soda solution of about 23% strength and a relatively cool temperature of 60–90°F (16–32°C). It is then fed around a series of rollers (timing cans) which keep it flat and smooth while controlling the time of caustic exposure. The fabric is sprayed with rinse water and then washed with a neutralizing chemical before final drying. Effective mercerization requires attention to variables such as caustic strength, dwell time (feed rate), temperature and neutralization. The feed rate of the fabric may also be limited by its strength and weight and is usually run at 80–120 yards (73–110 m) per minute. Optimum dwell time of 45 seconds to 5 minutes, depending on the fabric, allows the yarn to swell and the fibres to untwist in the fabric while tension is applied.

Control of the caustic concentration in the bath is important for the uniform and consistent quality of the mercerized textile. Proper caustic control ensures the fabric will have an even and predetermined caustic exposure based on weight and feed rate. It also helps in controlling the neutralization steps by avoiding overexposure to the caustic bath concentration that can prolong the washing and neutralization part of the process.

Electrical conductivity is an economical and convenient method for controlling the caustic bath concentration. Toroidal conductivity sensors are recommended to resist chemical attack by the strong caustic solution and fouling by accumulated solids. The relationship between the caustic concentration, temperature and conductivity is somewhat complicated. However, over the range of 20–28% caustic, the conductivity decreases in a relatively linear manner. The conductivity is a nonspecific measurement and a build-up of salts in the process may produce some background conductivity that may interfere with the concentration measurement.

## 4.13 Liquid Ammonia Mercerisation

Conventionally, for a period of more than a century cotton textiles were mercerized with aqueous solutions of sodium hydroxide in order to improve fabric tensile properties and to enhance lustre and reactivity. Perhaps the main problem associated with conventional mercerization is the complete removal of sodium hydroxide. If cotton could be mercerized with a completely volatile reagent, its removal after treatment would be greatly simplified. Liquid ammonia and certain primary amines are quite volatile and have been reported to cause swelling of cellulose fibres. The use of such agents should permit the development of a process

in which the mercerizing agent is completely recycled. Because of the cyclic nature of this process, there would be no pollution of adjacent streams and rivers, as in the case of conventional mercerization. Although research reports concerning ammonia on cellulose may be found since 1861, truly scientific results were only achieved in this field between 1926 and 1937.

Researches with liquid ammonia began with Bernardy, who found that the action of ammonia on cellulose was much weaker than that of the concentrated solution of caustic soda, and so the fibre characteristics were completely different in the two cases. He also observed an increase in the volume of cellulose, but there was no direct attack by liquid ammonia, as in the case of caustic soda. Some decades had to pass until these results found an industrial application in 1967. When native cotton (cellulose I) is treated with anhydrous liquid ammonia, an unstable adduct is formed and extensive swelling occurs, resulting in the disruption of hydrogen bonds. Depending upon the manner in which this adduct is decomposed, either cellulose I or cellulose III may result. If the adduct is broken through evaporation, or through quenching treatments in tetra-hydro-furan or acetone, cellulose III is formed. If the complex is broken by quenching with water or alcohols, cellulose I is regenerated. Either simple evaporation or heat treatments of the complex causes a substantial decrease in the apparent order of the cellulose and consequently a substantial increase in moisture regain. The treatment of native cellulose with a solution of 25% ethylamine in liquid ammonia followed by washing with tetra-hydro-furan constitutes perhaps the most convenient method for the preparation of cellulose III yet reported.

Cellulose and ammonia can form a complex, ammonia-cellulose II at a temperature below  $-30^{\circ}$ C and ammonia-cellulose I around and above  $-30^{\circ}$ C. The small ammonia molecules along with its weak surface tension favour penetration into the fibre core between the molecular chains of cellulose, without destroying micro-fibrils as in case of sodium hydroxide. The ammonia molecules being smaller than that of sodium hydroxide, swelling is also less. Since the internal structure of the crystallites has been affected, hydrogen bonds are broken. In the swollen state, the fibre is deformable and capable of being set in a new conformation by the newer hydrogen bonds, provided the tension is maintained until the ammonia has been removed. The ammonia, which has penetrated into the fibre, is removed very quickly and totally because of its high volatility and extremely high solubility. Like mercerization with caustic soda, ammonia produces changes in the structure of cellulose (i.e. reorganization of the crystalline network with cellulosic chains rotating and translating around their axle), giving a better accessible network to reagents. The differences between the treatments are:

- (1) Penetration of ammonia into the fibre and its elimination are nearly instantaneous (between 15 and 25 seconds). Therefore, the treatment is very fast.
- (2) Ammonia is recoverable and reusable after a purifying distillation. The caustic solution after mercerization, especially in grey form, is soiled and will create a pollution problem.
- (3) Water consumption is reduced to less than half in ammonia mercerization.
- (4) Ammonia is a natural substance and the process may be considered as ideally ecofriendly.
- (5) It gives reagent-free textile goods.

The differences in fibre properties are as follows:

- (1) The properties provided by traditional mercerizing (i.e. increased dye affinity and the change in appearance) are more or less preserved with ammonia. Improvement in lustre and dye uptake is, however, less than that of caustic mercerization.
- (2) Ammonia, a weak alkali, does not damage the fibre, unlike the very aggressive caustic soda. Mechanical properties like abrasion resistance, tensile and tearing strength are improved. Ammonia treatment increases tensile strength by 40% and the elongation at break by 2–3% that of untreated cotton.
- (3) Less swelling by ammonia causes the fibre to become more plastic and malleable. The life of the article is increased, and the new appearance is retained for a longer time. The difference in swelling also explains the more brilliant aspect of the fibre after the caustic soda treatment and the satin gloss after the ammonia treatment.
- J. & P. Coats Ltd. debuted the Prograde process in 1969, in which the yarn is passed through a liquor ammonia bath at −33°C (contact time 0.7 seconds) followed by a hot water bath (0.1 seconds at 93°C) where it is stretched 10–30% as measured against an ammonia-swollen length. The yarn is wound on a spool and dried continuously with hot air. The method, developed primarily for sewing thread, is claimed to be one thousand times faster than traditional yarn mercerizing processes. Prior moisture treatment (up to 30% of dry weight) improves the effects, especially uniform dye pickup. The ammonia treatment on fabric is being made continuously with a lengthways tension. It consists in impregnation of goods with liquid ammonia at atmospheric pressure (i.e. at the boiling temperature of −33°C) followed by the elimination of the reagent by evaporation (Sanforset process) or by rinsing with water

(Veramtex process). The cellulose is treated under tension in the presence of an alkali meaning mercerization by definition (Cheek et al., 1987). Ammonia mercerization is generally done on de-sized, kier-boiled and bleached (without optical brightener) fabric. No strong alkali treatment should be carried out after ammonia treatment. Ammonia can also be used as a remarkable solvent for all classical dyes, which dissolve mono-molecularly. Continuous dyeing with liquid ammonia may be carried out by mangling with the dyestuff dissolved in ammonia at -33°C. The goods are then steamed in order to fix the dyestuff and to remove the ammonia. Finally, the material is washed to remove unfixed dye. Denim has several deficiencies, with excessive shrinkage after home laundering and tumble drying perhaps being the most serious. Before the 1970s, dimensional stability after laundering was never fully achieved, even with compressive shrinkage. Furthermore, the only way to avoid excessive wrinkling during use was by treatment with resins, which led to a concurrent loss in strength. In 1967, a new mercerizing process (BP, 1967) was announced, claiming that liquid ammonia was superior to conventional mercerizing solutions for improving the smoothness of cellulose-containing fabrics. Denim samples were successfully stabilized against shrinkage caused by laundering and tumble drying. A low-level resin pretreatment (2.3–5.0% DMDHEU and 0.3–0.5% zinc nitrate) of greige denim, followed by slack mercerization with liquid ammonia produced fabric with excellent tensile strength properties. The low-level pretreatment does not improve the durable press rating, but it does improve shrinkage control; however, the high-level pretreatment also accomplishes this, if desired. The initial stiffness almost disappears after one laundry cycle. The resin pretreatment-ammonia mercerization improves DP ratings and usually improves shrinkage control in comparison to the samples that were not pretreated with resin.

### 4.14 Barium Activity Number

The ability of mercerized cotton fibre to absorb more iodine, dyes, moisture and alkalis and to undergo oxidation or hydrolysis at increased rates compared to unmercerised cotton has been used to develop various methods in determining the degree of mercerization. The most successful method consists of measuring preferential absorption of barium hydroxide from its dilute solution by mercerized cotton. The extent of this absorption is more than that by unmercerised cotton under the same conditions. This ratio, multiplied by 100, is known as barium activity number, barium number or baryta number. The mercerized cotton yarn is cut into small lengths, dried over phosphorous pentoxide for 5 hours and then conditioned at 65% relative humidity at 31°C before testing. The samples of both mercerized and unmercerised yarn each weighting 1 g are treated with 30 mL of 0.25 N barium hydroxide solution in 100-

mL conical flasks. After 2 hours, 10 mL of the solution is titrated against 0.1 N hydrochloric acid.

$$N = \frac{b-s}{b-u} \times 100 \tag{4.1}$$

A blank is run without any fibre. If b, s and u are the titration readings for the blank, mercerized and unmercerised samples respectively, then the barium activity number N is given by Eq. (4.1). An appropriately large number of conditioned samples are taken, taking into consideration the moisture regain of the samples. Thus if the moisture regain is 8%, 1.08 g of the conditioned cotton is weighted to get 1 g of the bone dry cotton. The barium activity number of good mercerized cotton may be around 150–160. The fabric's barium activity number has an interesting correlation with many factors, such as the degree of crystallinity, the strength and the dyeability of the fabric, and it can be determined from its crystallinity index (measured from X-ray diffraction pattern in the fibre), strength and colour strength. When the fabric's barium activity number was under 150 or the mercerizing reaction was incomplete, the fabric's crystallinity index was around 0.6–0.8. Once the reaction between the mercerizing agent and the fibre occurred completely as the fabric's barium activity number was over 150, its crystallinity index decreased to 0.5 and lower. This could mean that a complete mercerizing reaction first occurred when the crystalline region located in the fibre was decreased to 50%.

The degree of mercerization of cotton fabric was measured successfully using an online near-IR spectroscopy method. The measurements were performed on a commercial Benninger Dimensa Mercerization Range, where near-IR Ml values correlated strongly with BAN values of the samples that were subsequently analyzed in the laboratory. The effect of the fabric temperature on Ml values was significant; however, either by software modification or using a direct light spectrophotometer technology, the sample temperature effect can be compensated.

# 4.14 Parchmentising or Organdie Finish

Organdie or organdy finishing is done mainly on cotton fabric. In this process, cotton fabric is treated with sulphuric acid at room temperature at 110°Tw concentration of sulphuric acid in 4–5 seconds. With this finish, cotton fabric achieves more lustre, transparency, stiffness like woollen plain fabric. Generally, this finish is given on fine cotton, muslin and voile fabrics, as well as rayon. If thick fabric is used, the fabric looks like linen. The finish is for only higher count (80 Ne) fabric.

Generally, a mercerizing machine is used for organdie finishing, but a stenter machine may also be used. Regardless of which machine is used, it is important that the components of the machines do not react with sulphuric acid, so this process should be done very carefully. At room temperature, the fabric is passed into a concentrated sulfuric acid (100°Tw) for 4-5 seconds. After that, the fabric is washed by cold water to remove acid from the fabric and neutralized by alkali. Sometimes pyridine or glycerine can be used with sulphuric acid to reduce its rapid action. By a controlled process, formaldehyde is also used with sulfuric acid, and nitric acid and phosphoric acid may also be used for this finishing process. Rayon fabric may be mercerized with concentrated phosphoric acid (106°Tw). An organdie finish is mainly used to making screens for hand screen printing. This fabric is also used to create dresses like neckwear, curtains and other items. Sometimes it is also called by parchmentising. The real organdie finish is obtained by treating cotton material with sulphuric acid. The action of sulphuric acid of various concentrations is characterized by three stages of reaction; the first effect occurs with sulphuric acid of 110°Tw (64%), when a soft result is obtained resembling wool, the second effect obtained with 114°Tw acid, when considerable contraction takes place and the cloth becomes stiff, whereas the third effect is produced with 116–125°Tw acid when the fabric becomes stiff and transparent. When cotton is treated with sulphuric acid of 120°Tw (69%) for a few seconds, the fabric becomes thinner, finer and more transparent. The action is more intensive, if the cotton is previously mercerized, and the parchmentising effect is obtained when concentration exceeds 108°Tw. All acid treatments are carried out at room temperature and at the end of allotted time, the fabric is to be washed thoroughly to free it from all traces of acid. With weaker or crêping acid, the most attractive effects are achieved on lightweight fabrics containing fine cotton yarns such as muslins, voiles, lawns and similar fabrics. Such fabrics shrink during the treatment and become soft and elastic in nature. Stronger acids give a parchmentising effect which produces an organdie effect on the above type of cloth.

The use of a heavy calender or sometimes a heated calender is capable of giving translucent effects when applied to the acid-treated fabric directly after washing or drying. When mercerized cotton is treated with sulphuric acid of 103–110°Tw, the lustre disappears and fabric acquires a fine crêpe nature, appearing thicker, fuller and more wool-like. The beautiful transparent effects are obtained on lightweight fabric. The permanent transparent effect, however, is not obtained on coarser fabric, and it gives a linen-like effect. A linen-like effect may also be produced on cotton fabric containing yarns of less than 60s British count, by subjecting them to the action of 103–106°Tw sulphuric acid at 0–5°C for at least 4 minutes,

followed by mercerization under tension with 23°Tw caustic soda at room temperature. The reactivity ratio (treated: untreated) of parchmentised fabric is reported to be 1.6, which is greater than the average from commercial mercerizing, whereas a transparent organdie gave 2.07 and a linen finish from sulphuric acid gave 1.23.

# 4.15 Conclusions

Caustic soda has been used as an important finishing agent since one and a half century. Concentrated sulphuric acid has also long been used as a finishing agent. These finishing agents are still popular even after the invention of a large number of synthetic finishing agents, and the finishing processes have not changed much from their original versions.

# **Chapter 5 Finishing of Wool and Silk**

### 5.1 Introduction

For thousands of year, wool fibre is used to prepare apparels which are known for their warmth and comfort. However, these fibres are very delicate as they readily felt and shrink (even more than 50%) severely on washing. The handle and texture of woollen fabric also changes drastically on felting, though this has not restricted its use. The manufacturers and users are well aware of this problem and the garments and other woollen materials have been prepared and used with proper care to avoid felting. The manufacturers have also turned this problem into an advantage for certain materials like blankets to provide additional warmth. Silk, the aforementioned queen of textile fibres, possesses excellent properties and requires little finish. In early days limited finishes were being applied on silk to preserve its inherent softness and lustre. However, with increase in fields of application of silk, along with stiff competition from other fibres, silk processors have been forced to look into newer finishes. Clothes made from silk are distinctly luxurious and have many excellent qualities, including the material's lustre, wearing comfort, fine and smooth texture, soft handle and excellent draping quality; hence it needs finishing agents to a minimum extent. Some finishing chemicals may lower the quality of the fabric instead of improving it. Silk fabrics, however, have their tendency to crease easily when wet during home laundering. This disadvantage causes considerable inconvenience in the use of silk textile materials, so the world consumption of silk is directly influenced. Among various chemical modifications, the epoxy treatment for silk looks promising, with experimental results showing that the physical properties are improved.

### **5.2 Felting of Wool**

Wool has a scaly surface. It is composed of an outer layer of overlapping scales (the cuticle) surrounding an internal core (the cortex) composed of numerous long, thin, spindle-shaped cells running axially within the fibre and firmly cemented together. These structures are composed largely of keratin, a protein complex with a high sulphur content and which contains a number of disulphide cross-link residues. The surface of a wool fibre is therefore serrated in appearance when examined under the microscope, as are most animal fibres, though the scale pattern can differ depending on the animal from which it is derived. However, scale patterns do not always differ significantly. For example, fine wool and cashmere are very similar in appearance, and differentiating between them by visual examination microscopically is a skilled task. Wool fibres are also not usually straight; they are more or less wrinkled or crimped in appearance. This crimp is dependent on the breed of sheep from which the wool is taken.

Wool fabrics can change in dimension during washing or laundering for two quite separate reasons:

- ✓ felting
- ✓ relaxation

### 5.2.1 Felting

In order to understand the procedures necessary to produce dimensionally stable products, it is important to understand the different mechanisms involved. The major cause of shrinkage in wool is felting, in which the fibres become compacted or entangled with one another, which results in an overall contraction of the wool textile structure. The underlying cause of the propensity to felt is the differential friction effect caused by the scales on the fibre surface. The scales project slightly from the stem of the fibre and point towards the tip of the fibre. Wool fibres, therefore, possess a property called directional frictional effect (DFE), the result of which is that the coefficient of friction of one wool fibre against another is greater when the tips are pointing in opposite directions than when their longitudinal orientation is similar (Fig. 5.1). Though felting is primarily believed to be caused by projecting tips of the scales, some other keratinous substances devoid of scales also show DFE. Therefore the phenomenon is considered to be an inherent property of keratin itself. The ease of deformation and high power of recovery is responsible for the mass contracting in bulk by a series of self-contracting mechanisms. Felting, milling or fulling is the thickening or bulking of woollen fabric, accompanied by a loss of area or decrease in size when they are subjected to vigorous agitation in aqueous medium. Felting is caused by a degree of irreversible movements of fibres in relation to each other brought about by the DFE. The elasticity of wool plays an important role by causing the unidirectional movement responsible for felting. The tendency of felting reduces with the reduction of elasticity. When two adjacent fibres are compressed and relaxed many times, then the compressive force is withdrawn, the DFE will prevent them from returning to exactly the same positions as before. Consequently, the fibres are entangled and a compact mass is formed. The study shows that the root ends of the fibres tend to thread themselves through the fibres arranged in parallel format and when put under compression, they buckle up. Since he root of the fibre plays a significant role, either a hardening of this fibre zone or conversely a softening of the tip promotes shrinkage and thereby felting. The experimental evidence confirms the phenomenon.

Less friction More friction

### Fig. 5.1 Directional frictional effect of wool.

During the processing of wool, all precautions are to be taken to prevent felting. The felting properties of wool are due to three factors:

- (a) Scaly surface structure
- (b) Ease of deformation
- (c) Recovery from deformation

The shrinkage of wool is at its lowest between pH 4 and 8, and it increases rapidly on either side. The curves showing shrinkage and elasticity (i.e., the amount of work required to stretch the fibre by a specific amount) against pH have very similar shapes. Hence the shrinkage is considered to be closely related with the elasticity of wool fibres. Between pH 4 and 8, adjacent polypeptide chains may be bound together by salt linkages, making the structure comparatively rigid. Below pH 4 and above pH 8, the salt linkages are broken, and the fibres can expand or contract under the influence of alternate compression and relaxation.

Three main factors are involved in felting to take place are

- ✓ moisture
- ✓ mechanical agitation
- ✓ heat

Moisture causes wool fibres to swell, as wool is capable of absorbing up to 30%–38% of its own weight in water, which renders the wool fibre more flexible. The exact role of water in felting is the subject of a number of hypotheses, but the most probable explanation is that water increases the tendency of the scales to interact with rough areas on adjacent surfaces or scales on other wool fibres, causing an increase in the frictional differential between the with-scale and against-scale directions. This is probably coupled with the tendency of the wool fibres to collapse against one another when wet, thus increasing the interaction and therefore the tendency to felt. Mechanical action is very clearly needed to induce felting. In general the more vigorous the action, the more quickly felting can be induced. This has presented difficulties over the years as laundering habits have changed, and machinery with more severe mechanical action has been introduced onto the domestic market. Temperature also has some effect on felting, but the relationship is not linear. It would appear that felting reaches a maximum at

temperatures in the range of 50–60°C; above this temperature, there is no clear evidence that felting increases further. The choice of a laundering temperature of 40°C for washing wool in most washing machines is therefore a compromise between the need to use heat to promote cleansing and lower temperatures to reduce felting.

### 5.2.2 Relaxation Shrinkage

When any garment is immersed in water, the stresses and strains introduced into the structure by the various manufacturing and handling processes involved in its production are released. The garment dimensions therefore change. This change usually, but not necessarily, results in what is known as relaxation shrinkage. It may be reversed by pressing out the garment and is therefore not destructive. Mild agitation is sometimes necessary to fully release the potential dimensional change. In some cases, these stresses and strains are the result of deliberate 'setting out' of the garment or fabric in a drying or pressing process, where manufacturing or processing results in undersized items. However, such stresses and strains are more often introduced during production, and they are a particular problem whenever the garment is not subsequently given a wet process or washing step during which they can be released. The problem for the garment producer is that when such garments are laundered for the first time, the dimensions change and therefore so does the fit. Relaxation and felting shrinkage are equally important, and indeed are indistinguishable, as far as the consumer is concerned. Both are therefore taken into account by retail organisations; however, the technologist needs to be able to discriminate between the two in order to diagnose problems.

# 5.3 Prevention and Control of Shrinkage

In any study of wool shrink-resist treatments, it is important to consider the factors that affect felting control and their interrelation. All shrinkage control processes aim to prevent selective fibre movement during washing, resulting from the difference in friction between the with-scale and against-scale directions. This can be achieved either by reducing the frictional differential or by preventing the tendency of the fibres to move.

The most widely used method to control felting is to control the difference in frictional coefficients in two directions. The frictional difference can be eliminated either by raising the coefficient of the friction in the with-scale direction, reducing the friction in the against-scale direction, or modifying the fibre geometry. If the coefficient of the friction of the scale surfaces is increased sufficiently such that the with-scale coefficient of friction nears that of the against-scale direction, then the remaining difference becomes insufficient to create felting, except

under a more severe mechanical action than would be expected in home laundering. However, the end products may have a harsher handle, and therefore may be commercially unacceptable. If attempts are made to moderate handle with softening agents, these have the side effect of lowering the surface friction again. Thus, it re-creates the frictional difference and therefore the tendency to felt.

The coefficient of friction in the against-scale direction can be reduced in two ways

- ✓ modification of the behaviour of the scales
- ✓ the use of friction-reducing agents

The commercially useful shrink-resistant treatments are all based on making the scales more flexible when wet by using aggressive chemicals to attack the protein structure. However, sufficient precautions are needed to minimise the damage of the cortical structure in order to maintain fibre strength. This type of treatment is accompanied by a certain amount of modification of the scale tip geometry, which will assist in reducing the against-scale frictional effect, but is not sufficient for removal of scales. The friction-reducing agents with a sufficiently low frictional coefficient when applied to the wool fibre will reduce these effects in both directions. For this to be effective, the frictional effects must be reduced to such an extent that the difference between them becomes small enough to prevent the felting process from occurring easily. The only substances known to have such a significant effect on friction are poly-siloxanes (silicones) and fluorocarbons. In practice, neither type of material nor any of their variants have been found to be effective enough to be of practical value, other than in specific cases or as a component in a process.

# 5.3.1 Removal of Scale Tips

The idea of modifying fibre geometry by removing the serrated scale tips and therefore eliminating felting has existed for a long time. Early studies on oxidative treatments claimed that they work by this mechanism, but later studies on more controlled processes cast doubt on this. Scale removal has since been achieved in some commercial processes, and it does indeed result in a fibre that strongly resists felting. The severity of the treatment needed to achieve this, along with the subsequent weight loss and alteration of fibre properties involved, have greatly restricted its utility. A modern application of this mechanism results in the production of so-called 'lustre wool', where the drastic changes in physical properties of the fibre are exploited as a commercial differentiation.

### 5.3.2 Scale Masking

This concept later re-emerged in the 'scale masking' hypothesis, which suggested that a sufficiently thick layer of polymer evenly applied to the fibre surface would even out the surface roughness associated with the scales and therefore reduce their effect. A modified version has been advanced as the explanation for the mode of action of Hercosett polymer. This is applied at a rate that is insufficient to meet the needs of the hypothetical model, but will swell in water to a thickness considered sufficient to satisfy the requirements. It is now generally accepted that this mechanism plays some part in shrinkage resistance as imparted by the chlorine-Hercosett and similar processes.

### 5.3.3 Spot Welding

Felting can also be overcome by creating linkages between adjacent fibres within the textile material. If sufficient linkages of appropriate strength are formed, then movement of the fibres become restricted and felting is prevented. Cross-linking polymers can be used to form cross-links of sufficient strength to prevent fibre movement. The main problem of this method is the proper deposition of polymer to form the cross-links, instead of uniform deposition which may affect the properties of wool.

The main problem associated with the development of systems of this type is the appropriate deposition of the polymer onto the textile material in such a way as to form linkages between fibres, rather than an even coating of polymer over the surface of the fibre.

Some commercial processes are developed based on this mechanism. Most of these processes are for woven fabric, which is padded with a solution of the polymer. In such fabrics the fibres are generally in very close proximity to one another, facilitating the developments of the linkages or 'spot welds'. One major problem associated with this method is that the fabric may become much stiffer and lose its drape characteristics due to restricted fibre movement.

# **5.3.4 Application Methods**

Two types of chemicals are in use for shrink-proofing of wool, namely:

- ✓ scale modification
- ✓ fibre coating

### **5.3.4.1 Scale Modification**

The majority of shrink-proofing processes use chemicals to modify the nature of the scales of the wool fibre. All such agents act by attacking various links in the protein structure and rupturing them, thereby making the protein more readily swollen on immersion in water and making the scales more flexible. The keratin structure of the cuticle cells may be degraded in several ways, the most important being the breakage of the cysteine disulphide bond between two protein chains, resulting in two cysteic acid residues. However, the processes may result in unacceptable degradation of the scales, as all disulphide bonds should not be severed.

The proper oxidant should be chosen to achieve maximum felting control for minimum collateral damage. A few oxidants can be used to achieve a useful effect in a commercial process. The most common of these is chlorine, usually in aqueous solution, either as dissolved chlorine gas, or from the degradation of a compound that readily yields chlorine under mild conditions. Chlorine will rapidly attack wool proteins to oxidise the cysteine links and has found the widest utility in industry.

Chlorination is known to degrade and partially dissolve the cuticle with the oxidative breakdown of disulphide linkages, leading to the formation of cysteic acid units and ionised groups. This phenomenon leads to cuticular scale smoothing or removal, which is important for achieving shrinkage control. It also confers hydro-philicity to the surface of wool, which is important for solution permeability into the fibre surface or its interior.

The main alternative to chlorine is permono-sulphuric acid or its salts, particularly the potassium salt (K2SO5). This also modifies the keratin structure, but is more selective in its attack and produces a characteristic striated look to the scales when these are examined under the microscope. The full effect of this oxidant is only produced if the fibre is subsequently treated with an aqueous solution of a sulphite, usually sodium sulphite.

## 5.3.4.2 Fibre Coating

Fibre coating represents the second most important shrink-resistant treatment. The mechanisms of 'scale masking' and 'spot welding' cannot function without a surface coating, though not all polymers work through these mechanisms, as several clearly contribute to surface friction modification. The manner in which the polymer is deposited is critical for polymers that are claimed to work by either the 'scale masking' or 'spot welding' mechanisms. For scale masking the polymer must be evenly applied on the fibre surface, while for spot welding, it should be

concentrated at points where fibres touch. Many different chemical systems have been suggested as fibre coatings in wool treatments, but they should fulfil certain conditions:

- ✓ The coating must adhere to the fibre surface even under extreme conditions, such as dyeing at the boil, or reductive or oxidative bleaching.
- ✓ Where a scale masking or other overall effect is the objective, the polymer must be capable of spreading evenly over the fibre surface.
- ✓ The polymer must cross-link after an application to form a residue with sufficient mechanical strength to perform the function for which it is being used; for example, as an adhesive in the 'spot welding' system.
- ✓ The polymer chemistry must lend itself to the preferred mode of application.

The main problem with fibre coating lies with the issue of surface spreading. In its untreated state, wool fibre has such a high surface tension that many materials are unable to wet it. Silicone polymers are the most significant group of materials found to be of value on untreated wool or wool treated in such a way that it retains its high surface tension. Other materials tend to 'bead' or form clumps on the fibre. This, of course, may be valuable if the intention is to 'spot weld' the fibres together. Fortunately, a wider range of materials are available to reduce the surface tension and to make the fibre wettable. Of these, the most common is oxidation using a chlorine source. Control of the physical properties and chemistry of the polymer employed can then meet the other requirements. It must also be understood that for polymers to adhere to the wool fibre surface, the surface must be clean and free from dirt, oil, wax, softeners or other materials that form an interfering surface coating. Due to excessive use of polymeric cationic dye fixatives, shrink-resistant treatments may fail.

Shrink-resistant treatments may be applied at all stages of production of wool articles. There are some basic constraints that limit the usefulness of any process and therefore its application at particular points in the production route. These vary according to the type of process. The application of a 'spot welding' polymer must take place after the final assembly of fibres has been made, or subsequent processing would break the fibre–fibre bonds. Similarly, any process that relies on an even deposition or exposure of the fibres to a chemical finish will be more successful in situations where the fibres are in a state such that treatment liquor can circulate sufficiently around them.

Woollen spun knitwear made from pre-treated yarn is much more difficult to mill to the correct handle and texture. Although knitwear has been manufactured by this route, the achievement of a satisfactory finish has been found to be problematic. In some cases, namely with woollen garments, it is more convenient to apply the shrink-resistant treatment at the same time as the application of colour by garment dyeing. For several decades now in the United Kingdom, this combined process has been found to be very effective. It is ecologically more acceptable in energy usage than the application of two separate wet processes in the production route and the consequent drying that is involved. The main drawback of this process route is that it does not allow the creation of ingrain effects in the garment, which are produced by the blending, carding and spinning of different coloured wool fibres. In general, it is a good practice to treat worsted wool at the top stage and woollen spun product at the garment stage, though there are many exceptions to this. 'Spot welding' resin processes are mainly used for woven fabric and are the main route for this type of product.

There are many processes available for imparting shrink resistance to wool, and the procedures can mainly be divided into three categories:

- (i) Resin processes
- (ii) Kroy–Hercosett processes of polymer application after chlorination treatments
- (iii) Oxidation processes

At present, the majority of the world's production of machine washable wool is wool treated with resin by applying polyurethane to garments. Although resin treatments are effective, extensional elasticity, softness, fabric handle and other properties characteristic of wool are lost. In the Kroy–Hercosett continuous processes for tops, wool is damaged by a lot of chlorine. Strong acid is used in this process, which necessitates a neutralisation/anti-chlorination procedure to remove residual chlorine, which causes AOXs (absorbable organic halogens) and pollution of wastewater. The oxidative treatment system is a chlorine exhaustion method developed by Bereck and Reincke. This is a two-step process comprising Basolan DC, which is a commercial dichloro-isocyanuric acid (DCCA) method of BASF Co. in Germany, and subsequent hydrogen peroxide treatments.

This is an interesting process performing a full exhaustion of chlorine within the texture of wool fabrics and results in little or no harmful active chlorine in wastewater, since peroxide as the antichlor agent acts with chlorine, thus the chlorine used can be significantly reduced. Wool

damage can also be reduced to give a softer handle by removing scales on the fibre surface. This process can be carried out easily by using a conventional batch-type, small-scale chlorination vessel at a lower temperature for a long time, and thus temperature control is sometimes difficult due to a change in climate, which leads to unevenness in processing. Anhydrous sodium dichloro-isocyanurate DCCA-Na, [2893-78-9], a white solid with 63% available chlorine, is recognised as a principal solid chlorine bleaching agent. It is useful for treating fibre surfaces to prevent shrinkage by modifying the scales (partial destruction) of wool's exocuticle through oxidative attack. Modification of the fibre surface by chlorination is also known to increase the amount of dye absorbed. A known DCCA formulation, Basolan DCTM (BASF), when applied by exhaust at 2.5% and 4.5% by weight of fibre (owf) controls shrinkage to ~8%. Early studies showed that the reaction of DCCA-Na with wool was most effective at pH 5.5 and lower when applied at 25°C or 30°C. Subsequent de-chlorination with 3 g/L sodium bisulphite applied at 50°C improved whiteness, but changes in wool properties after treatment were not characterised. Studies of chlorine pre-treatment with DCCA alone and followed by applications of cationic polymers, including those derived from silicone, polyamine and polyurethane, provided information on shrinkage and yellowness development, though little information on changes in wool properties.

Cardamone et al. used DCCA alone in a range of concentrations from 5% to 40% owf. Treatments with 5% applied at 30°C for 60 minutes from a citric acid (CA) buffered system, pH 4, followed by anti-chlorination with hydrogen peroxide or hydrogen bisulphite showed small increases in alkali solubility and in bursting strength. Less than 2% chlorine is detected in 5% and 20% DCCA/anti-chlorinated spent baths. The 5% DCCA/hydrogen peroxide treatment improves shrinkage resistance by 54% and whiteness by 63% when compared to untreated fabrics. In the case of 5% owf DCCA, the importance of anti-chlorination is to prevent the development of yellowness, improve whiteness, and improve shrinkage resistance with minimum chemical and physical damage. DCCA treatment with anti-chlorination is simple, effective without additives, and uniform. Controlled oxidation by 5% and 20% DCCA is important for modifying the fibre surface with an anionic charge. That DCCA oxidation is limited to the fibre surface is shown by confocal microscopy images. Scanning electron microscopy (SEM) reveals scale smoothing, consistent with little change in bursting strength. However, in the case of 20% DCCA, high alkali solubility indicates chemical damage from chlorination penetration known to cause sulphur sulphur bond cleavage. Cardamone and Yao reported a novel process, the so-called ARS (Agricultural Research Service, United States) method for wool bleaching and shrink-proofing treatment using alkaline H2O2 systems, followed by enzyme treatment at near-room-temperature conditions. In Mori Eco-friendly Treatment (MEFT) process, fabric samples were soaked with 0.05% non-ionic surfactant solution at 20–25°C with a liquor ratio of 1:20. The bath was rotated until the samples were soaked. Then, 4% (owf) calcium hypochlorite (Ca (ClO) 2·3H2O, which contains 70% as active chlorine) was added to the solution. After standing for 5 minutes, 12 mL/L of H2O2 (35%) with 2 g/L sodium pyrophosphate as a peroxide stabiliser and 1 g/L ethylene-diamine-tetra-acetic acid as a sequestering agent were further added under gentle stirring. The mixed solution (pH 5.0) thus obtained was maintained at 40°C for 20 minutes to complete the reaction of hypochlorite ions with peroxide and, subsequently, 6% (owf) sodium sulphite and 1 mL/L of formic acid (FA; 80%) were added to the solution and adjusted to pH 3.0. The treatment was further continued at 50°C for 10 minutes. The hydrogen peroxide species remaining in solution were completely reduced with sulphite ions in the acidic environment. Finally, after a gradual cooling, the samples picked up from the solution were rinsed in water and air dried.

By using calcium hypochlorite, which is easy to decompose compared to DCCA, the aim is achieved and the yellowing problem brought about by chlorination is also solved. If we compare the amount of chlorine used in the MEFT process with DCCA, it decreases from 3.0% to 2.4%, which means the reduction of 20% of effective chlorine. By applying the MEFT process, excellent properties in anti-felting and fabric handle were imparted to wool fabrics without perceptible change in the breaking strength, whiteness and dyeability. Other properties, such as hydrophilicity, descaliness and softness, are comparable with the other processes, such as chlorination.

# 5.3.5 Application in Top Form

Worldwide, any shrink-proofing process is mostly done in top form. The top may be conveniently processed as a web of parallel strands of wool top, ~1.0–1.5 m wide in most cases, through a multiple bowl backwasher (Fig. 5.2, where two bowls are shown). In each bowl, the web of slivers is fed into the bowl through a pair of rollers and around one or more perforated drum, which serves to immerse the web in the process liquor. At the same time, the liquor is sucked out of the end of the drum and recirculated into the edges of the bowl, creating a strong liquor exchange around the sliver web, as well as serving to press it against the drum. The wool then exits the bowl through a squeeze nip between two rollers to remove as much treatment liquor as possible. The suction drum backwasher usually comprises between five and seven of these bowls together with drying equipment of generally similar operating principles, except

that circulating hot air serves to hold the slivers on the perforated drums, which are of much wider diameter. This machinery has proven very effective in achieving consistent and level treatments over a number of decades. Attempts have been made from time to time to treat wool top in dyeing machines, with the wool wound in 'bump' form. But the level of shrink-resistance is not sufficiently high to be established as a commercial process of widespread interest. The dominant technology in this area is the chlorine-Hercosett process developed by McPhee as reported by Lewis, and named by reference to the trade name of the polymer originally used, which was produced by the Hercules Powder Company. In this process, wool is subjected to oxidation by chlorine in solution in water in a multiple-suction drum backwasher. The chlorine is generally produced by the action of acid on sodium hypochlorite and is carried out at an acid pH (usually 1.5) at relatively low temperature. Other variants on this theme have emerged, of

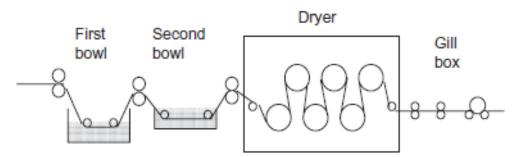


Fig. 5.2 Back washing machine.

which the main one is the use of chlorine gas dissolved in water, usually in a special applicator designed to contain the gas fumes generated and also to assist in a more level application of the chlorine to the fibre. Kroy produces the most widely used applicator machinery, in which the sliver web is held by two polypropylene belts and passed through a J-tube ~1 m in depth, where it is in intimate contact with the treatment liquor . In such processes, water temperature is very important, and in most warm climates, the water used for chlorination is cooled by refrigeration.

Step 1. Chlorination at 10–15°C at pH 1.5 with 0.3 g/L available chlorine, 5.0 mL/L sulphuric acid and 2.5 mL/L wetting agent.

Step 2. Antichlor at 40°C at pH 9.0 with 5.0 g/L sodium bicarbonate and 5.0 g/L sodium sulphite.

Step 3. Rinse at 30–40°C with water.

Step 4. Polymer treatment at 35°C at pH 7.5 with 5.0 g/L sodium bicarbonate and 5 g/L solid polymer

Step 5. Softener 40–45°C at pH 7.0 with 5 g/L sodium bicarbonate 2.5 g/L solid softener and 7.5 g/L lubricant.

A water-soluble polyamide is first produced by reacting adipic acid with diethylene-triamine, and the resultant material is then reacted with epichloro-hydrin to cross-link the precursor polymer, but more importantly to create reactive sites on the polymer backbone. The exact nature of the polymer and the reactive groups so formed varies depending on the exact reaction conditions used. Because of the cationic nature of the polymer, it has a tendency to exhaust onto the wool fibres, which have been made receptive to adhesion and spreading of the polymer by the chorine pre-treatment. The process is in part a padding operation and in part one of exhaustion during the brief period that the sliver spends immersed in the liquor (about 5–10 seconds).

The polymer is then cured on the wool as it is dried in the drum dryer. It is usual to arrange for the temperature in the dryer to increase from the start to the end of the process so that the sliver reaches 105°C, and the polymer cures properly; this is an essential feature of this type of polymer. Unfortunately, the wool so produced is very dry and difficult to process mechanically. Therefore moisture is added in some way, either by water spray, steam, or even by reimmersion in water and subsequent lower-temperature drying. Other polymer types have been used on wool, some of which have been promoted as needing no special curing step, but none have achieved any significance. One remaining drawback of this process is the affinity of dyes for the fibre, resulting in rapid dyeing and thus significant risk of unevenness. The fibres are therefore predominantly dyed in top form or as yarn in hanks. To avoid the use of chlorine, with the ensuing generation of AOX, a number of technologies have been developed and some have been commercialised, but to date none have yet achieved high popularity.

### 5.3.6 Application in Yarn Form

Wool, both in woollen and worsted form, has been successfully treated in yarn form, but only in the form of hanks in suitably designed machinery, where the liquor can be circulated in more than one direction so as to achieve the maximum degree of evenness of treatment. Despite this, the process is only of value for yarns that have already been dyed, because the shrink-resistant treatment is relatively uneven and therefore subsequent level dyeing is extremely difficult.

Dyeing may be carried out in the same overall wet process as the treatment with the shrink-resistant chemicals, provided dyeing precedes shrink-resistant treatment.

# 5.3.7 Application in Fabric Form

Many forms of equipment are available for the wet processing of woollen fabric and therefore for applying conventional oxidative shrink-resistant finishes. In fact, these usually result in high levels of relaxation shrinkage due to the tension induced by the processing and finishing machinery.

The only process to achieve widespread commercial utility is the application of 'spot welding' type polymers. This is a simple process using a vertical pad mangle to impregnate the fabric with a controlled amount of water containing the polymer in solution or suspension, followed by a drying and baking operation. More recent developments of this type of process are padding and drying, followed by garment making. The garments are then pressed and cured in a machine at higher temperature when a permanent- press effect is achieved.

## 5.3.8 Application in Garment Form

The garment processing of wool knitwear, both woollen and worsted, needs control to achieve the level results and restrictions on dye fastness, hence the woollen garment processing has been popular in the United Kingdom, though not worldwide. Due to the introduction of processes with greater control and also due to the development of better machinery for garment dyeing and wet processing, there has been an upsurge in popularity in recent years. The oldest machines for garment processing are paddle machines in which the garments are treated in a bath of water and agitated with a rotating paddle wheel. The simplest type of machine is the overhead paddle, where circulation is from top to bottom of the bath. A better development of this is the side paddle, where the garments are pushed around a steroidal channel by the paddle, thus achieving a circulation from top to bottom and side to side (Fig.5.3, P, paddle; V, vessel; I, internal perforated tank for additions).

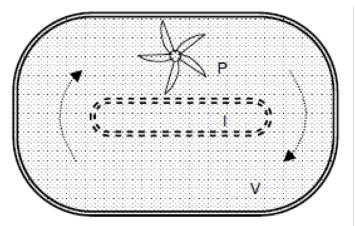


Fig. 5.3 Side paddle machine.

The main problem with paddle machines is that the garments cannot be effectively drained of process liquor, necessitating multiple rinses and prolonged process times. At the end of the process, the garments must be moved by hand into a hydro extractor in order to remove the considerable amount of water held in the knit structures. Alternatives exist in the form of the modern rotary dyeing machines, broadly based on the design of front-loading washing machines, but obviously with much greater capacity and control facilities.

They also operate at lower liquor-to-goods ratios than paddle machines, and this has a number of advantages for the dyer. However, the design of machine makes anti-cockle treatment of worsted knitwear much more difficult, and the lower liquor ratio is a disadvantage when attempting to chlorinate the garments. The latest non-chlorine processes lend themselves much more readily to this type of machine. After the wool top, the next largest volume of wool treated for shrink resistance is taken through the garment route, in one form or another of aqueous batch process. The wool, first scoured in a preparation step to remove contaminants, is oxidised with either potassium permono-sulphate or with chlorine, produced by the addition of acid to sodium dichloro-isocyanurate (DCCA). One or more polymers are then added; additional preliminary steps include anti-cockle for worsted garments and milling for woollen spun garments. The main process using chlorine in terms of volumes of wool treated over many years is the Dylan GRB process. This is usually performed in a side-paddle machine and has been used for treatment of both woollen and worsted garments. A substantial proportion of this production is dyed in the same wet process. The DCCA is carefully added in solution to the side paddle in such a way as to diffuse as evenly as possible into the goods, which are being agitated in a pre-acidified bath. The chlorine is slowly liberated from the DCCA by the careful control of bath pH and water temperature. Care must be taken to ensure that as little reaction

between the wool and the un-acidified DCCA occurs, otherwise the wool then becomes much yellower in appearance without achieving any significant shrinkage control.

## 5.3.9 Polymer addition

The bath is filled with cold water and paddle speed is set to 14–18 rpm.

1% acetic acid is added to set pH is 5.0–5.5.

2.0% polymer GE is diluted 10 times with water and added in the bath slowly for 10 minutes.

The paddle speed is reduced to 12–14 rpm, and the bath is run for 5 minutes.

The temperature of the bath is increased to 40°C and is run for a further 15–20 minutes.

1%–3% cationic softener on the weight of material is added.

0.5–2.0 mL/L hydrogen peroxide is added.

The bath is run for 10 minutes and then drained.

The main advantage of this type of process is that the action of this oxidant on wool is slower than chlorine at room temperatures, and therefore a more even treatment can be obtained. The limitation is in the lower level of protection afforded. The pressure to produce wool with good shrink resistance and better aesthetic properties coupled with less environmental impact will provide the driving force for new processes (Thomas et al., 1993). Attention is heavily focused on the value of plasma treatments for this purpose.

### 5.4 Fulling or Milling

Generally felting is undesirable for woollen and worsted apparels. However, felting is done purposefully in certain cases. Fulling or milling are processes where felting is done purposefully. The original term for felting process is 'fulling', which is still used in the United States. The British term 'milling' is used for all compressive processes, which cause felting. The woollen material coming from looms or the knitting machines are thin, porous in structure and lacking rigidity. Milling closes the interstices and makes a solid fabric with higher strength. Milling is continued until a specific density or weight per square yard/ metre is attained. Milling processes can be divided into three types:

- (1) Grease milling
- (2) Acid milling

## (3) Soap milling

Greasy woollens are often milled in sodium carbonate alone, which saponifies the grease into soap. Grease milling is a cheap but dirty method. The greasy fabric is run through a liquor containing 25–30 g/L soda ash and then squeezed to a liquor ratio of 1:1 or 3:2. The fabric is then milled to the required cover and dimensions before washing. This process is seldom used for medium and good quality fabrics. Some goods, such as felt hats and blankets, are milled under acidic conditions (pH 3) using dilute sulphuric acids. Acid milling is especially suitable for dyed goods, as many of the dyes applied on wool have poor resistance to neutral and alkaline treatments. Acid is preferred as a milling medium because

- (a) The resultant fabric has a greater tensile strength.
- (b) The process occurs more rapidly and to a greater extent.
- (c) There is a greater degree of extensibility and stretch of the milled fabric.
- (d) There is less fibre degradation and less flocking with heavier fabrics in with acid milling than with grease or soap milling.
- (e) There is less bleeding or cross-staining at low pH.
- (f) There is a minimised washing off, resulting in lower water consumption.

However, alkaline milling conditions are still largely used for woven piece goods, with the maximum milling taking place at around pH 10 using soap. The high-melting point soaps, such as those based on tallow and palm oils, are preferred to give the required gelatinous solution and lubricating properties. Sodium or potassium soaps are dissolved to give a 5% solution in hot water. Such solutions have to be applied warm, as soap solidifies on cooling. For carbonised fabric, thorough neutralisation is necessary, otherwise there may be flocculation of fatty acids. The soap acts as a lubricant and decreases the inter-fibre friction, permitting more rapid shrinkage than with alkali alone. For producing heavily consolidated fabrics such as meltons and billiards, soap is essential. However, during lengthy soap milling, certain fabrics cease to shrink further. In these cases the fabric is washed in a washer and then returned to the milling machine. Excessive lubrication by soap can block the felting process by reducing the influence of directional frictional effect. There is a gradual trend towards milling in almost-neutral conditions, for which milling aids based on non-ionic and anionic surfactants are useful. Some wool yarns are milled nowadays simply by tumble-drying wet yarns, whilst knitted garments

are milled in rotary-type machines using non-ionic surfactants with sodium bicarbonate or polyphosphates.

Normally, milling is carried out in a rotary milling machine (Fig. 5.4) in which the scouring and milling can also be done simultaneously. The fabric is processed in an endless rope form and passed through the milling liquor, followed by a pair of heavily loaded squeeze rollers and then into a spout, where compression takes places. The spout is rectangular in the cross-section, and the topside is pivoted at one end and free at the other. The pressure on the spout can be varied by applying weight to the pivoted top. The relaxation takes place as the fabric leaves the spout so that the necessary alteration of pressure and expansion is brought about.

# 5.5 Setting

Setting operations are the most important and critical of processes in the conventional finishing of wool fabrics. Depending on the finish required, the fabric may be 'set' two or three times during a complete finishing routine. Setting is carried out in loom-state or early in a finishing routine to stabilise the fabric by removing residual stress in the fibres and yarns imparted in prior spinning, twisting and weaving operations. In this way, when the fabric is subsequently processed in rope form during wet finishing, the formation of distortions due to uncontrolled relaxation of the fibres can be minimised.

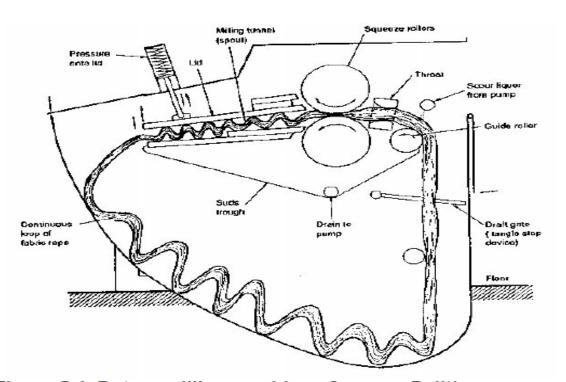


Figure 5.4 Rotary milling machine. Source: Pailthorpe

After wet finishing a wool fabric may be set to remove any unwanted distortions (such as rope marks) that have been introduced. In dry finishing, wool fabrics are set to ensure that they maintain their dimensions, flat appearance and surface geometry during any subsequent finishing operations or garment manufacture or while in use. Although their primary purpose is to stabilise the fabric in a flat form, permanent setting processes have many side effects. These operations improve the suppleness and smoothness of wool fabrics, with the modification of handle resulting from profound changes in the mechanical and physical properties of the fabric. Setting operations also modify the dimensional properties of wool fabrics; this depending on the nature of the fabric and its end use, this can improve or impair its overall dimensional stability.

Three types of sets have been defined in wool fibres:

- 1. Cohesive set: When the fabric is immersed in water at room temperature, the set is lost. The said cohesive set can be recovered by stenter drying and in continuous-dry finishing operations, such as pressing. The cohesive set is also the type of set associated with the formation of wrinkles in wool apparel during the wear and steam pressing of garments.
- 2. Permanent set: In this process, the deformation is stable to release in boiling water for 60 minutes. This type of set is conferred by wet and/or high-temperature operations, such as crabbing, piece dyeing and pressure decatising. The permanent set is now redefined as the deformation remaining after the wool is relaxed in water at 70°C for 30 minutes.
- 3. Temporary set: This is defined as the difference between the set released in water at room temperature and that released at a higher temperature used to assess a permanent set. There is some confusion between cohesive and temporary set, and in many parts of the wool industry, the terms are used interchangeably.

The disordered matrix component of wool fibres is normally associated with the setting phenomena. Movement of the macromolecules in this component is limited by a number of types of intra- and intermolecular interactions, namely:

- 1. Hydrogen bonds between amino, alcohol and amide groups
- 2. Polar interactions between ionised acid and basic groups
- 3. Disulphide and other covalent bonds

In wet wool fibres, there is also considerable evidence that there is interaction between the nonpolar side groups on the protein macromolecules, which contributes to the stability and mechanical properties of the fibres. These interactions have been called 'hydrophobic bonds'. Early research placed greatest emphasis on the permanent setting of wool fibres. As a result, less work was done on cohesive or temporary settings in spite of their importance in finishing as well as in the wrinkling, smooth-dry performance and pressing of wool garments. A cohesive set was originally described in terms of the rearrangement of hydrogen bonds (or the hydrogen bond network) in wool fibres under the influence of heat and/or moisture.

# 5.5.1 Wet setting

Wet setting operations are carried out to set wool fabric under flat condition permanently. The protein macromolecules are more mobile in the wet state; hence the permanent setting of wool can be done more rapidly or at lower temperatures than that at normal moisture regain. These operations do not impart a cohesive set because even at room temperature, the wet wool fibre is above its glass transition. A cohesive set is only imparted in subsequent drying operations. Crabbing and wet decatising are the most common forms of wet setting. Traditional 'crabbing' is a process in which the wet fabric is wound (often interleaved with a blanket or wrapper cloth) onto a drum which is partially immersed in hot or boiling water. The fabric is held in the hot water for 10-20 minutes, then rewound from the other end of the piece and retreated. The retreatment is necessary to avoid differences between the two ends of the fabric. The traditional Yorkshire crab also has additional rollers for steaming the wet fabric. The hot fabric is normally quenched when it is unrolled through a trough of cold water. Traditional crabbing imparts large amounts of permanent set (often in excess of 60% using crease-angle tests) to wool fabric. 'Potting' is a labour-intensive and time-consuming variation on traditional crabbing, which is designed to impart a strong permanent set and is reserved for very special applications, such as billiard cloth. The fabric is wrapped on a canvas-covered roller, which is then placed vertically in a tank of hot water ( $\sim$ 60°C) for up to 3 days. In some cases the fabric is cooled on the roll. A further variation on crabbing is 'beaming', in which the fabric is again rolled onto a beam but held in warm water (~40°C). The procedure is designed to remove crease marks formed in scouring or dyeing. Although beam dyeing is strictly a coloration process, it is also an effective crabbing operation. Under the conditions in the dye bath, large amounts of wet setting operations are designed to permanently flat set wool fabrics. The greater mobility of the protein macromolecules in the wet fibre allows a permanent set to be imparted more rapidly or at lower temperatures than that to the fibre at normal regain. In this operation, there is a danger of moiré

formation, so that beam dyeing is more often used on colour-milled woollen rather than clear-finished worsted cloths. 'Wet decatising' ('decating' in the United States) is also a type of setting operation in which wet fabric is rolled, or sometimes interleaved with a cotton wrapper cloth, onto a perforated drum. However, in this case saturated steam (rather than hot water) is passed through the roll to heat the wool and wrapper to temperatures around 100°C. Again, it is often necessary to rewind and retreat the fabric to avoid end-to-end differences.

The fabric is normally cooled before it is unrolled by drawing air through the roll. This avoids the possibility of distorting the fabric while it is still in a condition where a permanent set can be imparted.

Chemically assisted wet decatising is widely used in parts of Europe, especially Italy, to improve the handle and dimensional stability of wool fabrics. However, the use of reducing agents and wet fabrics can damage wrapper cloths and decatising machinery.

Before scouring, the woollen material is to be set with hot aqueous solution to prevent shrinkage during scouring and milling, resulting in cockling and crows footing (i.e., width distortion of the design and weave). For greasy materials, alkaline solution is preferred; the process is known as crabbing. In the simple crabbing process (Fig. 5.5), the fabric previously wound under tension on a roller (A) is wound onto a second roller (B) by passing it under tension through a trough of hot water (H). The second roller moves in touch with another roller fitted on its top (C). As the roll of fabric builds up on the lower roller, pressure is applied from the upper roller, which operates in slots to allow for the increasing diameter of the lower roller. Pressure is applied on the upper roller with the help of levers, wheels, weights, etc. The process may be repeated in a second trough. The application of heat and pressure in the moist condition is responsible for setting the fabric in a smooth and even manner. The duration of immersion in hot water may be 5–15 minutes, followed by natural cooling

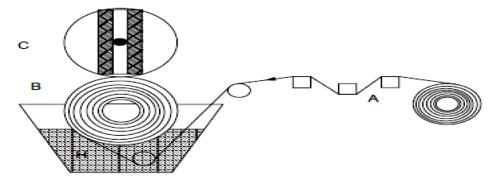


Fig. 5.5 Crabbing machine.

or by running it through cooled water. Double crabbing (i.e., making the upper layer of first crabbing as the inner layer during second crabbing) is often advisable for a more even treatment throughout the length of fabric. The crabbing process is often replaced or followed by a faster blowing process, in which the fabric is wound on a perforated roller through which steam is blown for 1–3 minutes under a pressure of about 0.1–0.5 MPa (1–5 atm). The process is repeated after rewinding the fabric so that the external layers form the interior of the roll. Machines are also available in which crabbing and blowing can be carried out consecutively.

In the crabbing and blowing processes, all fabric irregularities (e.g., creases) and all latent strains from spinning and weaving are removed and some widthways shrinkage occurs. The fabric is set in a smooth and regular state capable of withstanding the subsequent processes. The fabric becomes uniform and lustrous in appearance as well as thinner in handle. The weave is clarified, and where the weave is pronounced, a cotton wrapper is interposed between the layers of wool to avoid marking during the process. For most goods, an interlayer wrapper is not essential, and its use will increase the treatment time, as well as require frequent washing.

The potting process is similar to crabbing and is used for high-quality woollen fabrics. The fabric is wound carefully onto a cotton-wrapped perforated iron or steel roller, and the roll is placed in a tank of cold water. The temperature of water is raised slowly to either 75–80°C, or the boil and the treatment is continued for 3–5 hours. After cooling and rewinding to reverse the fabric, the operation is repeated. The traditional batch crabbing and wet decatising processes are labour intensive, suffer from end-to-end and batch-to-batch variation and have low production rates. Continuous crabbing machinery was first introduced in the 1960s. In these machines, the fabric is wet out in a trough containing hot water, squeezed to give the fabric the correct water content and then sandwiched between a heated drum (up to 160°C) and an impermeable belt. The wet fabric rapidly heats to temperatures at which a permanent set can be imparted. After its passage around the heated drum, the fabric is then quenched in a coldwater trough. The residence time of the fabric in the heating zone is short (typically 1 minute), so that unless the temperature of the wet fibres exceeds 100°C, only a little permanent set is imparted. Early continuous crabbing machines did not seal the fabric well and were relatively ineffective. In more modern machinery, improved belt technology allows higher belt tensions and a better seal, so that when combined with the higher drum temperatures (up to 180°C), higher temperatures can be achieved on the fabric and more a permanent set can be imparted on the wool. A number of machinery manufacturers offer this type of continuous crab as part of their range; for example, Supercrab-2 (M-Tec), Multi-set (Sperotto Rimar) and Vulcano (Biella Shrunk).

### 5.5.2 Dry Setting

Dry setting is the process of setting wool fabrics at a normal regain. In practice the regain of fabrics in a mill environment can vary from around 5% after drying to 15% after conditioning. Depending on the conditions, dry setting operations can impart a cohesive, temporary or permanent set (more often, all three) on wool fabric.

### 5.5.2.1 Decatising

Decatising is a type of setting operation in which fabric is rolled and interleaved with a cotton or cotton-blend wrapper cloth onto a perforated drum. Saturated steam is then passed through the roll to heat both the fabric and wrapper. If the steam used to set the fabric is vented to the atmosphere so that the fabric temperature is limited to around 100°C, the operation can also be called 'blowing'. 'Semi-decatising' describes the procedure where the fabric is rolled up with the wrapper and steamed only once. 'Full decatising' describes the procedure where the fabric is semi-decatised, unwound, reversed and then fed into the machine from the other end to be semi-decatised a second time. Two-drum decatising machines are designed to facilitate this rewinding and double steaming operation. In Germany the terms 'lustre (pressglanz) decatising' and 'finish decatising' are used to describe semi-decatising operations carried out on machines with different sizes of drums and under specified conditions (e.g., time, temperature, wrapper tension) to impart different surface effects to the fabric.

If the drum onto which the fabric and wrapper have been rolled is placed in an autoclave, the temperature of the steam (and fabric) can be increased to values as high as 135°C. The operation is then called 'pressure (or autoclave) decatising' or occasionally 'KD setting' (after the German, 'kessel dekatier'). In such machines the time and temperature as well as wrapper type and tension can be varied to impart different effects on the wool. Additionally, the steam can be passed from the outside to the inside of the roll, or from the inside to outside, which is known to change the effect of the process on the thickness, surface properties and ultimate handle of the fabric. All decatising machines have the facility to cool the fabric by drawing air through the roll before it is unwound, and it is used if a firmer, flatter handle is required.

The amount of permanent sett imparted on wool in decatising and its effect on the properties of the fabric have been extensively studied and reviewed. The amount of permanent set is

determined by the regain, pH and temperature of the wool and the time of the treatment. The moisture regain and pH of the fibre are critical in pressure decatising. The amount of the permanent set imparted is reduced dramatically if the pH of the fibres is low (pH < 3) or if their moisture content is below 10%. The critical moisture content depends on the temperature of the treatment and the extent of the increase of the moisture content due to condensation during heating of the fabric.

The decatising process improves wool and woollen materials especially with regard to the texture structuring and appearance of the product. During the process, steam without pressure in a cooled-down state is brought into contact with the material. Alternatively, controlled humidity may be introduced into the treating area and over the material, which is then converted into steam by pressure and the effect of heat. The continuous decatising machine (Fig.5.6) consists of two main cylinders fabricated out of stainless steel perforated plates mounted on ball bearings with specially designed gun metal nozzles for effective steaming and cooling. Another stainless steel dryer roller provided at the bottom between the two main cylinders having proper heat controlling arrangement to dry the wrapper cloth. Two hard-chromed pressure rollers press the fabric while feeding it to the main cylinder to ensure crease-free entry of the fabric into the machine. Pneumatic cylinders are provided to adjust the pressure and tension of the fabric, and plaiting-down arrangements with geared motors are made on either side for piling the fabrics.

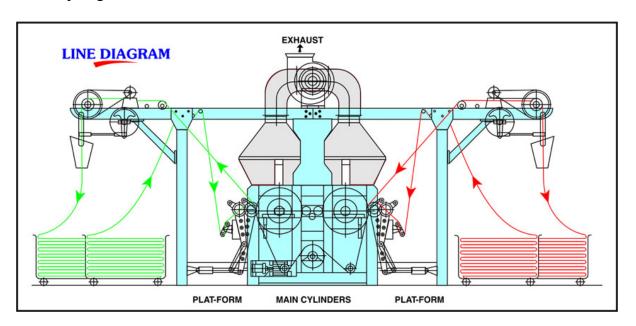


Fig. 5.6 Continuous decatising machine.

5.6 Fundamentals of Silk Finishing

Silk, the queen of textile fibres, possess excellent properties and require little finish. Silk is

available in four varieties, namely mulberry, tussah, muga and eri; the most popular is the

mulberry variety. Silk consists of a fibrous portion called fibroin and the gummy portion called

sericin, apart from small percentage of minerals, waxes etc. Silk possesses a high lustre. Many

finishing chemicals lower the quality of the fabric; hence, minimum finishing agents are

applied on silk. The creases form easily in silk during home laundering or when wet. Among

various chemical modifications, the epoxy treatment for silk looks promising, with

experimental results showing that the physical properties are improved.

Silk is the only continuous-filament natural fibre. The unique qualities of silk come in the form

of its high texture, fineness, strength, lustre, scroopy feel and good draping quality. Combining

these qualities with its eco-friendly usage makes silk among the more favoured textile fibres.

However, these qualities can only be achieved after removal of the silk gum (sericin) which

protects fibre from damages during silk production.

For silk articles, the 'silky handle' is most desired, and so the finish must take account of this

age-old demand. The desired finish for silk is similar to that of other fibres and is mainly

comprised of:

✓ lustre: discreet, not greasy

✓ handle: soft but firm-textured

✓ gall or draping of the material

The finish must take into account the following aspects:

✓ The inherent handle and draping properties of silk should not diminish.

✓ The right combination and sequence of the finishing machines depends on the silk

article.

The wash-and-wear finish is now becoming more important in the silk industry, which

can be achieved using softeners, elastomers and synthetic resins.

✓ The undesirable properties of silk should be improved by finishing techniques.

✓ The demand for soil-resistance and flame-retardant silk is increasing.

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Antimicrobial finishing is applied in order to maintain hygiene and freshness. In earlier days, the finishing of silk articles was primarily a matter of the finisher's craftsmanship. Experienced finishers used to manage batches as per the requirement of individual customer using their skill and experience. Since ancient times, there were very few chemicals, such as acids, oils and filling agents (e.g., gum Arabic or tragacanth), that were used for the finishing of silk. With the passage of time, better wear and care properties of silk were needed to compete with other fibres in performance quality. Higher wear and care properties are now more important in the silk industry and are achieved by the use of softeners, elastomers and synthetic resins.

However, silk need not be as durable as other textile materials. The future will dictate whether we really need machine-washable silk and whether the technical problems of finishing can be solved without affecting fibre or fabric properties to a large extent.

# 5.7 Mechanical Finishing of Silk

Mechanical finishes are ecofriendly, but the effects obtained are mostly temporary. The most important mechanical finishes are calendaring, decatising, breaking, etc. Calendaring is necessary to improve appearance after the completion of the final wet processing. Various types of calendaring are applied on silk.

#### 5.7.1 Calendering

Calendering is a mechanical levelling and segmenting process for 'finishing' fabrics or webs to obtain or to produce special effects. Such special effects could be flattening, lustre, compacting, glazing, moiré, Schreiner, smoothing, texturing and other embossed patterns. Calendering is a finishing process used on cloth where fabric is passed between rollers at high temperature and under high pressure. Calendering is used on fabrics such as moiré to produce a watered effect and also on cambric and some types of sateens. The fabric is run through rollers that polish the surface and make the fabric smoother and more lustrous. High temperatures and pressure are used as well. Fabrics that go through the calendering process feel thin, glossy and papery. The calendering finish is easily destroyed and does not last well. Washing in water destroys it, as it wears with time. Calenders are used to influence the handle and appearance of the fabric.

With most articles a three-bowl calendar with steel and paper rollers are sufficient. Additional calendar rolls may be needed, such embossing or schreiner calenders (e.g., moiré effects). The three basic mechanical parameters of this operation, in which the fabric is passed between or around the calendar rolls, are

- (1) Speed (calculated on the basis of dwell time in the nip).
- (2) Precise pressure (PLI or pounds per lineal inch).
- (3) Heating—rolls are often heated to a predetermined temperature to obtain the desired or more lasting effect.

The three mechanical operating parameters of speed, temperature and pressure must be controlled. In addition to these three parameters, consideration must also be given to the following:

- ✓ A back-up roll design and type.
- ✓ The wear resistance of top rolls and resilient rolls.
- ✓ The effects of fabric finishing chemicals on rolls.
- ✓ Web tension.
- ✓ Control and profile of roll temperature.

The function of a rolling calender is to provide a smooth or glossy fabric surface as well as to improve the fabric's hand. The basic mechanical action of this type of calender is to cause the fibres and webs to not only reshape but to also possibly flatten or deform around one another, as it also causes the fibres to nestle or stack more tightly around one another. This function can process all types of cloth/webs. Normally, the unit comes as a three-roll calender with alternate steel and filled rolls, although it may also come with two, four or five rolls. The intermediate resilient roll is of wool felt paper, cotton, khaki wool and resilient wool and cotton blends. The main function of the silk finishing calender is to provide a smooth fabric surface, light lustre and improved hand. The basic mechanical action for this type of calendar can be the same as for the rolling, but with the selection of special fibre rolls that are comparatively softer. When loaded against a smaller diameter steel, the 78 shore hardness fibre rolls will create a very wide nip between the rolls, which will not only extend the web but also bend bonds from coatings or remove stiffness from the weaving, knitting or yarn forming process. A silk finishing calender can process all types of fabrics, but it is used mostly for high content cotton and coated or impregnated webs. It may run at a speed of 100 yards per minute, although most nonwovens are normally run between 30 and 35 yards per minute. Nip loading is between 400 and 700 PLI. The wide nip flexes and releases the web, causing a disruption between the bond points which not only serves to 'soften the hand' of the web, but also to strengthen the web

by enhancing the inter-fibre cohesion. The silk finishing calender has a three-roll configuration, with a top- and bottom- filled roll and a steel middle roll. The filling is of cotton/wool blends. Some synthetic-covered rolls can be used, but they afford far less mechanical displacement to the web. The main steel roll, which is positively driven by a variable speed motor and a roller chain, can be heated by gas or steam to surface temperature of 175°C. Optionally, the two auxiliary filled rolls can be driven off nip drives. The unit has a circulation system for the bearing boxes, which are required for roll heating systems over 150°C. In most cases, silk is only calendered in a cold state, which produces a soft handle. A higher lustre is obtained with hot calenders, but it must be determined in each case whether such treatment has a negative influence. In exceptional cases (e.g., furnishing fabrics), chinz effects are desired.

#### 5.7.2 Palmer or Felt Calender

This is used for delicate fabrics like cotton mulmul, voiles, silk, synthetic and knitted fabrics. The machine provides a smoother handle and more lustrous appearance of silk fabrics. It is mainly used for taffeta-like woven fabric and for serge articles. Fig. 5.7 shows the line diagram of a felt calender. The combined drying and finishing is achieved by using an iron or stainless steel cylinder with about a six-and-a-half feet diameter (C) moving together with an endless blanket (B) while the fabric (A) passes in between. The tension of the blanket is controlled by a small roller D, though very little tension is put on the fabric. The production of felt calendar is  $\sim 30-50$  yards per minute.

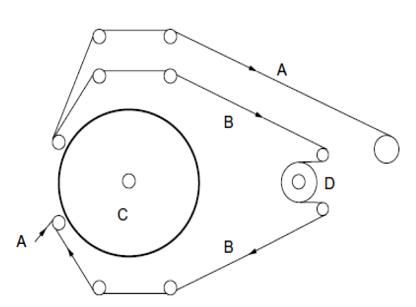


Fig. 5.7 Schematic diagram of a felt calender: (A) fabric, (B) blanket, (C) large heated cylinder and (D) small roller to control tension in the blanket.

# 5.7.3 Decatising

Decatising improves the dimensional stability of silk fabric. It removes creases to make the fabric smooth. Various types of silk fabric, such as woven crêpe fabric, are decatised. The wool felt fabric used for decatising silk is not too hard, and the silk fabric is not pressed flat. Superheated steaming during decatising gives the desired appearance and a smoother feel on various fabrics. Both discontinuous and continuous types of decatising machines are available on the market. Fig. 5.8 shows the principle of decatising.

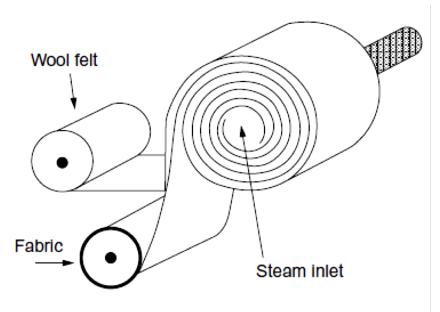


Fig. 5.8 Principle of decatising.

The fabric to be decatised is wound with a woollen felt fabric (as the external layer) on a perforated stainless steel roller. Steam is passed from the inside of the perforated roller through the wound layers for specific time (e.g., 30 minutes). The fabric is rewound by changing the upper surface of the fabric as the inner layer and steamed again. Kawahara et al. covered the fabrics with the wrapping cloths and wound it all into a roll. The roll was steamed from the centre of the roll for 5 minutes, then cooled. The radial expansion of the roll by steaming was increased by a small percent. The silk necktie fabrics with 63 denier weft yarns (manufactured by twisting 3 silk yarns of 21 denier) in doubles showed higher values of the shear and bending rigidity as well as their hysteresis, as compared with the fabrics with 126 denier weft yarns (manufactured by twisting 6 silk yarns of 21 denier) in singles. For the fabrics with methacrylamide-grafted 126 denier weft yarns, the shear rigidity and its hysteresis decreased, but the bending rigidity of satin fabrics increased. The decatising treatment was effective in reducing the shear rigidity and its hysteresis.

# 5.7.4 Breaking Machine

A breaking machine is used to confer a particular type of soft handle when calendaring is insufficient. Breaking is a final treatment for fabrics with an excessively harsh handle, such as crépe. Breaking machines or breakers are of two types:

- ✓ Button type—the piece is passed several times rapidly back and forth over small rollers studded with brass buttons.
- ✓ Knife type—the fabric is drawn over the edges of slanted knives.

The older button type has recently been replaced by knife breakers.

# 5.7.5 Tamponing

Silk is very sensitive to mechanical friction when it is subjected to various stages of treatment. Even if sufficient precautions are undertaken, silk fabrics are chafed in places. Chafing may originate during boiling off, then show up only after colouring or dyeing. The cause in most cases is the rubbing of the fibre/fabric surface against harder surface. Light chafe marks look like white spots, as if the material has been sprinkled with white powder or flour. During chafing the fibre surface gets roughened, whereby the individual silk fibrils are split open. The nap or pelt thus formed produces a different refraction of light and generally take up a lesser quantity of dye, thereby producing a 'white effect'. Chafing may be aggravated during finishing due to

- (1) Too high of a dyeing temperature
- (2) Too long of a treatment time
- (3) Extreme pH values above pH 10 and below pH 4

The dyeing temperature is the most serious factor. It is, therefore, recommended to dye sensitive fabrics at low temperature and to prolong the treatment time somewhat to obtain good exhaustion. Depending on the degree of chafing, this damage can be removed in some cases by wiping lightly with oil, known as tamponing, which pastes down the raised hairs on the fibre fibrils. In former days, faulty portions of fabric were treated by hand with a so-called tamponing cushion which carries an extremely fine film of oil. Nowadays, tamponing machines can apply a fine film of oil homogeneously on several rollers, which in turn are evenly transferred onto the fabric. The correction of chafing requires considerable experience, and the fabric may have to be run more than once through the machine.

# 5.7.6 Shrinkage and Relaxation

Horizontal screen steamers or shrinkage machines are used to produce shrinkage and relaxation of the fabric. Crêpe-like fabrics sometime appear too flat as they come into an insufficiently relaxed state from the previous treatment. In the screen steamer, the fabric is laid without tension (i.e., relaxed state) on a continuous screen. Steam is passed through the screen and from the fabric below. The shrinkage machines may be open or closed.

# 13.8 Chemical finishing of silk

For many years, silk finishers used classical finishing agents and took a conservative view of modern finishing chemicals. However, as new end use standards are set and new finishing chemicals are developed, modern products have now made headway in the silk finishing sector. Though finishes based on elastomers and synthetic resins are developed, specialists are often critical about their use, due to the fear that the characteristics of silk, particularly its handle, may be hampered.

# 5.8. Weighting of Silk

Unlike most fabric/yard goods which are sold by the yard (or metre), silk is sold to the wholesaler by weight. The first step in processing the silk fibre is 'degumming the fibre', with the gum being a by-product of the production of the silk fibre by the worm. Approximately one-fifth of the weight of the silk fibre is lost in the degumming process, and manufacturers felt that they had the 'right' to replace this lost weight with a filler of some sort. Silk has an affinity for several metallic salts, the more common of which are iron, lead and tin. It was discovered to be an easy process to return this weight lost in the degumming process by soaking the fibres in a bath of these metallic salts. This process is called weighting, and by increasing the weight of the raw silk, the merchant increased his profit. Weighting with some metallic salts did improve the drapeability of the fibre; however, greedy merchants soon began adding more weight than the lost one-fifth, and sometimes the final weight was increased 10-fold. The finishing treatment of 'weighting' on silk has been commonly used for the last 300 or more years throughout the world. This practice was widespread in the 19th century and decreased somewhat in the 20th, but it is still used to some extent. The most commonly used process of weighting is tin/phosphate/silicate, where tin silico-phosphate is finally formed on the fibre along with lot of intermediate and by-products. Weighting gives fullness and richness of feel and handle, whereas heavy weighting can make the fibres hypersensitive to rubbing, resulting in a rough handle. Generally, the finer and more expensive reeled silks are weighted rather than the less-costly spun silks. Heavily weighted silk must be made into garment as soon as it is made. Saltwater, perspiration and tears cause spots to be formed that seem as if the silk is eaten by acids. Sunlight also attacks weighted silk.

The silk industry makes a distinction between pure-dye silk and weighted silk. In the pure-dye process the silk is dyed and subsequently finished with water-soluble substances such as starch, glue and gelatine, but it is not weighted. If weighting is not executed properly, it may lose much of its strength and durability. The metallic salts used for weighting silk may also cause health risks.

The weighting improves the handle and drape of the material, and with some polymer weighting systems, flame-retardant and easy-care properties can be introduced. The increase in fabric weight is measured in 'par', either 'below par' or 'above par', and can be as high as 300% above par, based on the raw weight of the silk. Excessive weighting can be detrimental, as this diminishes the affinity of the fibre for dyestuffs and causes brittleness and a loss in fibre strength. A variety of methods have been used over the years to weight silk, which can be classified into three categories:

- (1) Vegetable weighting
- (2) Mineral weighting
- (3) Polymer weighting

# 5.8.1. Vegetable Weighting

The use of tanning agents is a simple operation that causes improvements in hydrophobicity, but due to their inherent colour, the agents cannot be applied on light-coloured dyed fabric or white fabrics. The method is now rarely used.

# 5.8.2. Mineral Weighting

Tin phosphate/silicate is the most popular weighting process. The application is made in several stages, and careful monitoring is conducted to ensure minimum undesirable side effects. The silk is soaked in stannic chloride at room temperature for about 1.5 hours. It is then hydro extracted and washed in cold water. Washing removes any unfixed stannic chloride and hydrolyses-fixed stannic chloride into meta-stannic acid. The silk is further treated with dilute disodium phosphate for 1 hour at 60–70°C; it is then washed and acidified to give an insoluble tin phosphate compound. The whole process is repeated until the required increase in weight is obtained. Finally an insoluble tin silico-phosphate is formed by treating silk with dilute

sodium silicate at 70°C for 1 hour. Deposition of the metallic salt takes place in the amorphous regions of the fibre with little alteration of fabric appearance.

$$SnCl4 + 4H2O \rightarrow Sn(OH)4 + 4HCl$$
 (5.1)

$$\operatorname{Sn}(OH)_4 + \operatorname{Na_2HPO_4} \to \operatorname{Sn}(OH)_2 \operatorname{HPO_4} + 2\operatorname{NaOH}$$
 (5.2)

$$Sn(OH)_2 HPO_4 + 3Na_2SiO_2 \rightarrow (SiO_2)SnO_2 + 3NaOH + Na_3PO_4$$
 (5.3)

Mineral weighted fabric can be identified by a burning text. Untreated silk melts partially and forms little char, whereas mineral weighted silk burns well enough to leave ash.

The weighting process includes the application of 30%–300% solutions of inorganic salts of aluminium, iron, lead, tin or zinc to the fabric in order to increase the body and weight. These weighting compounds are highly acidic, lowering the pH of the solution below 3. The pH of the solutions used to treat silk during manufacturing processes is an important factor in the light stability of silk fibres. Silk is less resistant to light between pH 6 and 8. It has also been reported that silk shows its maximum light fastness at pH10. Accordingly, it is obvious that weighted silks are more susceptible to photo-degradation. The damage caused by strong acids to silk fibres is quite severe, and it has been shown that the effects of weighting on silk are much more deleterious under exposure to light than in darkness.

## 5.8.3 Polymer Weighting

Organic weighting by grafting has recently been introduced in order to decrease costs and to avoid the deleterious effects of metallic weighting. This method of weighting is based on a treatment of methyacryl-amide and ammonium per-sulphate. It is reported that polymer weighting of up to 50% can be achieved without impairing the fabric handle. The disadvantage of this method is that it alters the dyeing properties of the fibre, but it is preferred, as it is less harmful to the environment than mineral weighting.

# 5.9 Sandwash Finishing of Silk

The sand-wash finish produces a machine washable fabric with a soft, velvet-like feel. The sand-washing process itself gives rise to a rough handle, but this is modified by using softeners to produce the sand-wash feel. The effect is produced by treating silk under harsh conditions, when the fabric surface roughens breaking the surface fibrils to create a peach-skin-like texture. Depending on the method used, the fabric also shrinks, making it dimensionally stable. The traditional handle of silk is lost, along with as much as 50% of the initial fabric strength.

A sand-wash finish can be produced in several ways. Silk may be dyed in overflow machines under harsh conditions (e.g., for longer time or at higher temperatures), either directly during dyeing or as a prewash treatment. The process can sometimes be carried out in a washing machine with the addition of pumice stones or small pieces of expanded clay. The disadvantage of these methods is the inadequate uniformity of the finish and the risk of chafe marks. Other methods include the mechanical raising of the fibre surface using an emerising machine, although with this method, there is the risk that whole fibrils may be broken. Uniform surface modification may be achieved by using a protein-active enzyme such as Bactosol SI (Clariant).

#### 5.10 Softeners/Lubricants

Softeners and lubricants are used to improve handle, drape, cutting, sewing, etc. and to help retain the traditional feel of silk. Fabric softening is carried out during the final stages of wet processing, usually in the final wash bath. A wide variety of softeners can be applied to silk; recently the use of multifunctional silicone softeners has become popular, particularly aminofunctional poly-siloxanes in micro-emulsion form. Silk develops extraordinary properties with the application of softeners/lubricants. These softeners help to improve hand, drape, cutting, sewing and other desirable qualities.

In recent years, with the popularisation and application of silicone softeners in the textile industry, the silk industry widely uses organic silicon as the main body of the finishing process and combines it with mechanical finishing in order to improve the performance of silk fabric. Silicone-finished silk fabric has improved handle, lustre, elasticity, <3% shrinkage, and maintain the natural style characteristic of silk. Various types of silicone-finishing agents impart different degrees of smoothness to silk fabric. The film-forming silicone products provide the best flexibility. But, for real silk-finishing silicone, preferably using single-component, low-temperature elastic membrane species, amino-modified silicone finishing agents are used. The non-ionic products include poly-ethoxylated esters of long chain alcohols, acids and oils. The non-ionic types are easy to mix with other finishing agents.

## 5.11 Scroopy Handle

Scroop is the ability to make a rustling sound added to silk or rayon fabrics during finishing by treating with certain acids. To measure the scrooping sound of silk objectively, a sound generator was developed to rub two fibre bundles at right angles just as violin strings. It was observed that

- ✓ The fibres can generate an effective frictional sound with little mechanical or outside noise.
- ✓ The measured sound is intermittent due to stick-slip vibration.
- ✓ The intermittent vibration contains both the string transverse vibration and the vibration of about 8 kHz frequency.
- ✓ The scrooping sound found in silk handling also contains an intermittent vibration, which has also about 8 kHz in frequency.

According to the prevailing fashion, the final handle of the fabric may be adjusted to be harder or softer, depending on the type of silk article. The fibre itself should always retain the classical feel of silk. This must be taken into account while formulating finish liquor. In some cases a scroopy handle finish is given as early as the final rinsing bath after dyeing or printing. This preliminary finish is given by adding oil emulsion or Merseilles soap. In a subsequent bath without intermediate washing, these are precipitated on the fibre surface in a finely dispersed form with an organic acid such as tartaric, oxalic, FA or acetic acid. It was long believed that a good handle can be produced by this double deposition.

However, it is stated (Sandoz, 1988) that the addition of oil and soap do not contribute to producing the scroopy handle; rather, it is attributed to the acid alone. The natural scroopy handle can be restored after various treatment stages. It is believed that the acid treatment causes the formation of a very fine skin on the fibre surface by reorientation of the fibroin molecules at the outermost layer of the fibre. Various acids influence the handle in different ways. For a scroopy finish (Sandoz, 1988), silk is treated in 1–3 g/L oil emulsion (e.g., winding oil or Clariant) or 1–3 g/L Marseilles soap solution for 15–20 minutes, then treated without rinsing in a second fresh bath containing 2–4 mL/L FA, acetic acid, lactic acid, or CA for 15–20 minutes at room temperature, then hydro extracted and dried. For the softening of a scroopy-handle silk, fabric is padded with 10–20 g/L cationic softener (Ceranine SG paste, Clariant) and 1 mL/L acetic acid. The fabric is dried at 120°C after padding.

# 5.12 Oil- And Water-Repellent Finishing Of Silk

Traditionally, metallic acid salts or oxides, proteins or nitrogenous compounds were used; however, the current trend is for combined polymeric finishes that offer resistance to both oil and water, based on silicones, acrylamide, acrylic acid (AA) or fluoro-chemicals. A combined treatment with a fluorocarbon derivative, a reactive resin and a water-repellent finish produces

a versatile finish. Grafting with the vapour of hexa-fluoro propylene, vinyl fluoride, vinyl chloride and acrylonitrile produced an increase in water repellency, oil repellency, mineral acid resistance and photo-stability. An example of such a finish is as follows:

# Oil- and water-repellent finish with spot protection

Fluoro-chemical compound

Sandoflur K or GTC (Clariant) 20-30 g/L

Cerol EWL liquid (Clariant) (as extender) 40-60 g/L

Finish MH liquid (Clariant) 5–10 g/L

Zinc chloride or zinc nitrate 0.6–1.2 g/L

Adjust pH to 5–5.5 with acetic acid, pad at room temperature, dry at 120°C and cure for 4 minutes at 150°C.

## 5.13 Stain Repellent/Release Finish

Soiling generally means smearing or staining of a large surface of the fabric with dust, dirt. oil, grease or any of these. A fabric gets soiled mainly by three types of mechanism:

- (1) Mechanical adhesion of soil to the cloth
- (2) Adhesion by electrical forces due to an attraction of dust particles from the air by an electrically charged fibre surface
- (3) Re-deposition of soil during washing

Some of the principal reasons for discarding textile articles include the inability to remove stains. Stains, whether on fabrics or on garments, are unwelcome. In fabrics, they reduce the consumer appeal, and pieces containing stains have to be marketed at low prices as seconds or fents. One Teflon fabric protector from DuPont (marketed by INVISTA) is a water-based, fluoro-chemical finish that resists substances that are present in oil- and water-based stains. It has been studied on woven silk union fabric by Desai. While many stain resistance products are merely a surface coating for fabrics, Teflon textile finish works through sophisticated molecular engineering on the nanometre scale to protect each fibre in fabrics. When a repellent finish is applied to fabrics, it provides superior water/oil repellency and protection against spills and stains. When a stain-release finish is applied, the invisible stain release technology improves the ability for ground-in stains to be easily removed during laundering. Teflon fabric

protector delivers durable, long-lasting protection by forming a molecular shield around the fibres, guarding against oil- and water-based stains, dust and dry soil. Repellent products lower the critical surface tension of the fabric, so the fabric won't attract stains or soil. Oil and water bead up and roll off the fabric, so spills can be easily blotted up with a clean cloth. Advanced Dual Action Teflon fabric protector combines stain repelling and stain release.

Additional important advantages of silk blend/union fabrics are easy-care and moderate prices. The fabrics thus woven were finished with a stain-repellent/release finish, and the easy-care properties are seen to be enhanced. The finish applied is seen to be compatible to the antimicrobial finish applied to the fabrics. The finish combines the best properties of two stain fighters into one finish; that is, both stain repellency and stain release have been imparted effectively to the fabric. Two additional important advantages of silk blend/union fabrics are their easy care and moderate prices. The fabrics thus woven were finished with a stain repellent/ release finish, and the easy care properties was enhanced. The finish applied was compatible with the antimicrobial finish applied to the fabrics. The finish applied has combined the best features of two stain fighters into one finish; that is, both stain repellency and stain release properties have been imparted effectively to the fabric. A spot- (water-) resistant finish is as follows:

Cerol ZN liquid (Clariant) 20-40 g/L

Ceranine HCS/HCL liquid (Clariant) 10-20 g/L

Adjust pH to 5–5.5 with acetic acid, pad at room temperature and dry at 120°C.

#### 5.14 Antistatic Finishing of Silk

Silk has a fairly low static charge build-up property, but static charge build-up may increase if silk is grafted with a monomer used to improve certain fibre properties. Treatment with selected water-soluble vinyl monomers (e.g., N, N'-methylene bis-acryl-amide, tri-acryl-oyl polyethylene glycol methacrylate etc.), under acidic condition produces an increase in antistatic and hygroscopic properties. The deposition of certain metallic particles has also been found to increase the antistatic and conductivity properties of the fibre.

# 5.15 Crease-recovery finishes

Commercially an easy-care fabric or product is one that does not crease readily while in use, can be washed and requires no or minimum ironing to restore its original appearance. In addition, the traditional comfort, handle, texture, drape and colour of the product should not be

affected. Technically an easy-care finish enhances the fabric resistance to and recovery from creasing without any adverse effect on other intrinsic fabric properties. Resistance to creasing depends on rigidity, and recovery from creasing depends on elasticity (Marsh, 1962). In today's market, easy-care products are applicable to almost all clothing materials. In silk finishing, there is a gap in the market for a product that can impart true easy-care characteristics.

The easy-care properties are inadequate as compared to synthetic fabrics, so a good creaserecovery (CR) finish becomes quite desirable for silk. Suitable resin pre-condensates are used to achieve this effect. These products are such that they either react with one another or crosslink the fibroin backbone to form water-insoluble products under the action of heat and catalysts. Silk has been used as a luxury fibre for many years due to its soft hand, characteristic scrooping, excellent drape and good lustre. However, it has a very low fibre resilience that causes wrinkling, especially when wet. This poor wet resiliency is due to silk's lack of intermolecular chemical cross-linkages. When the fibres absorb water and swell, the salt linkages between polymers, which are responsible for the high dry CR of these fibres, are broken. Silk fabrics have low wet and dry resiliency, hence the fabrics wrinkle easily during home laundering or when wet. One of the most important factors that influence the quality of the fabric is the ability of to recover from wrinkles. Wrinkles are defined as the fabric deformation based on its viscoelastic properties. They are classified into two categories, namely desirable wrinkles for a fashionable look and undesirable wrinkles that occur during wear. The crushing and creasing of textile materials is a complex effect involving tensile, flexing compressive and torsional stresses. The tensile extension plays a relatively small part in the elastic recovery, and measurements of the modulus of elasticity are of little help to the crease-ability of the various fibres, as the more elastic materials in the engineering sense are apt to crease more readily. The bending elasticity seems to be of the greatest importance in the phenomenon of creasing. A crease appears when the material is distorted in such a manner that part of it is stretched beyond its small power of elastic recovery. The bending of fibres or filaments which takes place during crease leads to an extension of the fibre on the upper surface and a compression on the under surface. The relative positions of the polymer chains may change due to the above phenomenon, resulting in a formation of creases. However, if the adjacent polymer chains are bonded by cross-links, the polymer chains can return back to their original locations and prevent crease formation. The crease-resistant finish on cotton fabrics improves comfort, ease of maintenance, dimensional stability and pilling performance. Silk has

less wrinkle formation when compared to cotton, and it is altered by applying poly-carboxylic acid (PCA) and di-methylol urea (DMU).

Fig. 5.9 Chemical structures of (A) BTCA and (B) CA.

Various chemical methods have been examined to improve the poor performance properties of silk. In general, these methods may be roughly divided into three groups: grafting of vinyl monomers on silk, cross-linking through formaldehyde- containing reagents and cross-linking through non-formaldehyde reagents. The grafting of silk with many different types of monomers has been extensively examined by Tsukada et al. However, this method uses organic solvent systems in many cases; therefore there are some doubts for its commercial application. The most popular cross-linking agents in crease-resistant finishes have been N-methylol agents or N-methyl-amides because of their efficiency and low price. Formaldehyde-free crosslinking agents for producing crease-resistant properties are of interest to replace N-methylol compounds. Poly-carboxylic acids, which are non-formaldehyde reactants, are possible replacement for conventional finishing reactants. The main advantage of poly-carboxylic acids is that they are formaldehyde free, do not have bad odour and produce very soft fabric hand. Non-formaldehyde di- or multifunctional cross-linking reagents included aromatic acid anhydrides, dibasic anhydrides and epoxide. Anhydrides also require organic solvent. To obtain the optimal improvement in a crease angle of silk, about 20% of aqueous epoxide was needed. This can adversely impact hand and drape properties of the treated silk. In addition the increase in the wet crease angle for tussah silk was not very high.

More recently, the butane-tetra-carboxylic acid (BTCA, Fig. 5.9A) treatment of silk was found to improve considerably the resilience of silk. However, to form an anhydride molecule, as a precursor for the esterification reaction between silk and acid, a high curing temperature was needed. This can adversely affect the hand and drape of the treated silk. BTCA is expensive

and the catalyst used; i.e., sodium hypophosphite (SHP), caused some shade changes for the dyed fabrics due to its reductive nature. Therefore a search for a cross-linking agent for silk finishing must be continued.

As mixed poly-carboxylic acids have been an interesting research area for saving money with BTCA by reducing the required amount of BTCA in durable press (DP) finishing, the finishing of BTCA mixed with CA, (Fig. 5.9B) was investigated.

It was observed that the wet-wrinkle recovery angle (WRA) decreased when a smaller amount of BTCA mixed with CA was used in the DP finishing, although the breaking strength retention did not vary too much. As CA was considered to have a lower activity than BTCA, in the same condition, CA could not have a complete cross-link with silk fabrics. To obtain better finishing results with CA, other methods should be considered. The combination of a hydroxylfunctional organo-phosphorous oligomer (HFPO) and 1, 2, 3, 4-butanetetracarboxylic acid as a formaldehyde-free flame-retardant finish was applied on silk. When BTCA is applied alone, most of BTCA reacts with the hydroxyl group of silk by a single-ester linkage. In the presence of HFPO, BTCA is able to bond HFPO onto silk by either a BTCA 'bridge' between silk and HFPO or a BTCA-HFPO-BTCA cross-linkage between two silk protein molecules. The treated silk fabric demonstrated a high level of flame retardancy with a modest loss of tensile strength. The treated fabric passed the vertical flammability test after 15 hand-wash cycles. The thermal analysis data demonstrated that HFPO reduces silk's initial thermal decomposition temperature and promotes char formation. Glyoxal is a non-formaldehyde reagent which is readily available. It has been reported that in the presence of aluminium sulphate, glyoxal provided the high reaction rates for cotton required in commercial processes. By using various co-reactant additives, such as glycol and  $\alpha$ -hydroxyl acid, a mild curing condition such as 120°C for 2 minutes may be suffice. The formation of cross-links in cotton by glyoxal was achieved through hemi-acetal formation. A similar chemistry can also be applied for the reaction between silk and glyoxal. The feasibility of using glyoxal as a crease-resistant finishing agent for silk was examined by Choi et al. (1998). Results indicated that glyoxal considerably improved the conditioned WRA of the silk in the presence of a Bronsted-Lowry acid, such as sulphuric acid and FA, and aluminium sulphate. The presence of such a strong acid did not greatly reduce the tear strength retention of the treated silk. After the treatment the dyeability of silk with acid dye slightly decreased. The FA-CaCl2 solubility test substantiated the presence of cross-linking in the treated silk. X-ray diffraction indicated that the reaction mainly occurred in amorphous region. Thermal stability at a high temperature of the treated

silk was generally better than that of the untreated control. The conventional treatment is conducted with an epoxide solution in tetra-chloro-ethlene at 60–80°C, which is applied on an industrial scale in Japan. In a conventional epoxide finishing of silk, an organic solvent has to be used, which is hazardous to the health of the exposed workers as well as the environment. With the family of silicone epoxy cross-linking agents, crease resistance occurs because chemical cross-links occur between the silk fibroin strand and the epoxy groups.

Cai et al. used EPSIA (epoxy silicone cross-linking agent), a new modifying agent consisting of silicone-containing epoxy cross-linking agents to improve the wet resiliency of silk; the structure of this is shown in Fig. 5.10, where R1, R2, R3 are alkyls and n is either I or 2. The silk fabric samples were treated with a pad-dry-steam process from an aqueous bath containing EPSIA, the dispersion agent (Invadine JFC) and the catalyst (potassium

$$R_1$$
 $R_1$ 
 $R_3$ 
 $R_1$ 
 $R_3$ 
 $R_4$ 
 $R_5$ 
 $R_5$ 
 $R_5$ 
 $R_6$ 
 $R_7$ 
 $R_8$ 
 $R_9$ 
 $R_9$ 
 $R_9$ 

Fig. 5.10 Structure of EPSIA (epoxy silicone cross-linking agent).

thiocyanate). The sample is then dried at 60°C for 3.5 minutes and finally steamed at 110°C for 7 minutes. After the chemical finishing, both the dry and wet resiliencies of the silk fabrics were distinctly increased, and the finished silk showed good durability to home laundering. Results also indicated that any shrinkage in the laundering process was well controlled after the silk fabric was finished with EPSIA. Breaking strength of the fabric decreased slightly, whereas tear strength increased. Furthermore, there was a 20% decrease in the bending stiffness and more than a 200% increase in abrasion resistance. These results showed that the treatment of silk with EPSIA could significantly reduce the inter-fibre or inter-yarn frictional force, as well as being capable of enhancing resiliency. However, there was a 12.6% reduction in moisture regain. Researchers made a comparative study of the application of three cross-linking agents, namely glyoxal, CA and 1, 3-dimethylol-4, 5-dihydroxyethylene urea (DMDHEU). The catalysts used were aluminium sulphate, sodium hypophosphate and magnesium chloride, respectively. The optimum concentration for treatment with glyoxal and CA were found to be 5% and 6%, respectively. The aluminium sulphate was found to be the most suitable catalyst for glyoxal. The optimum curing temperature and curing time were found to be 120°C and 120 seconds. Ecofriendly, crease-resistant finishes were found to have less of deleterious effect on mechanical properties, i.e., tensile strength, abrasion resistance, etc. when compared with

DMDHEU. The percentage of increase in the CR angle was found to be more in tussah fabric than with mulberry fabric. Untreated silk fabrics have poor easy-care characteristics. They cannot be machine washed, as they shrink. It is also impossible to remove all the creases inserted during washing by ironing. A zero-formaldehyde, easy-care finish for silk fabric was developed using a PCA finishing system, as reported in a PhD thesis submitted. The treatment of silk fabric with a PCA, namely 1,2,3,4-butane tetra-carboxylic acid, C8H10O8 (BTCA), CA or 2-hydroxypropane-1,2,3-tricarboxylic acid, C6H8O7 in the presence of SHP catalyst improved the easy-care properties of the silk fabric when compared with the untreated control. The thesis confirmed that the treatment of the silk fabric with the selected PCA-finishing systems improves the stability of silk fabric. The treated fabric had improved stability to heat, reduced swelling in solvents, and improved elastic recovery in the dry and wet states. A number of interesting mechanistic possibilities are proposed for improving the easy-care performance of silk fabrics with a PCA-finishing system. It is postulated that the improvement in elastic and stability properties of the treated silk fabrics could be as a consequence of new fibre inter-chain cross-links and/or a new intra-fibre PCA-polymer network being formed within the silk fibres. Three different types of possible bonds between the PCA and silk fibre are:

- ✓ Ester links formed via acid and/or base catalysis with hydroxyl containing amino acids, namely serine, threonine and tyrosine, in silk fibroin.
- ✓ Amide links may also be formed between the basic amino acids, which are arginine, lysine and histidine, in silk fibroin and acid anhydrides formed during curing.
- ✓ Hydrogen bonds formed with acid, basic and hydroxy side groups, as well as with the silk fibroin backbone.

Irvin extends previous research with respect to PCA finishing. It proposes the use of a new analytical method to predetermine the effectiveness of an acid/catalyst system as an easy-care finish. It recommends the use of combination acids to compete with 1, 2, 3, 4-butanetetracarboxylic acid and proposes the use of monosodium citrate as an alternative catalyst to SHP when cured at 165°C. A water-soluble anionic bifunctional polyurethane polymer, WPU200 was prepared from the reaction of PEG200 and hexa-methylene diisocyanate at 60°C in butanone in a stirred reactor (Hu and Jin, 2002). It is reported that WPU200 can improve remarkably the wet resiliency of silk fibres, so it is an effective wash-and-wear finishing agent for silk fabrics. The WPU200 polymer has the advantages of water solubility, low-reaction temperature, soft handle and no hazards to human health. Traditional

pad-dry-cure technology can be used in the wash-and-wear finishing of silk fabrics with WPU200. The optimum finishing conditions include 4% WPU200 in the finishing bath at pH 9–9.5, curing temperature of 100°C, and curing time of 3 minutes. Both the breaking strength and elongation of the finished silk fabric drop slightly. Whiteness and moisture regain (65% RH. 25°C) of the finished fabric are little lower than those of the control samples, and the stiffness is a bit higher. The dry CR angle and the handle can be improved by anionic and nonionic softeners.

The post treatment with various softening agents further improved the easy-care characteristics of the treated silk fabric. Treatment with a reactive silicone softener gave rise to a fabric with the greatest improvement in elastic properties, as well as a super soft and luxurious yet floppy handle. The treatment of silk fabrics with a BTCA/SHP finish even with a silicone softener after-treatment did not improve the elastic properties of the silk fibroin sufficiently for this finish to be recommended as a viable easy-care finish for silk fabrics for commercial use. However, the inclusion of a fibre-swelling agent FA or AA into the PCA pad bath vastly improved the easy-care properties of the treated samples. It is proposed that the increased swelling brought about by the inclusion of a fibre-swelling agent into the pad bath was able to wet-set the silk through stabilisation in its new swollen state. The after-treatment with a silicone softener improved the dry elastic recovery; this should help prevent creases being formed in the dry state. The finish produced a super soft fabric with minimum ironing requirement and enhanced resistance to fabric abrasion. The treated fabric was machine washable. The treatment of silk fabrics with a BTCA/FA/SHP or BTCA/AA/SHP finish, followed by an after-treatment with a silicone softener results in a good easy-care performance. The reactions of the epoxy compound (tri-glycidyl ether of glycerol, TEG) with silk under pad-dry-steam or pad-dry-bake conditions have been reported by Peng et al. In the presence of the catalysts, thio-sulphates or thio-cyanates, the epoxy-descould rapidly react with silk. Crease-proofing has been conferred on the fabrics as a result of epoxy cross-linking. However, yellowing of silk is intensified by this process, and the bath solution containing epoxy resin and catalytic salt is rather unstable, turbiding or even precipitating after short time storage. Epoxy resins, especially TEG and diglycidyl ether of ethylene glycol, are the effective silk finishing agents. NaH2PO4 plays a role as a buffer for the alkali released in the finishing processes catalysed by potassium thiocyanate (KSCN). A two-bath pad process (i.e., padding epoxy resins and a catalyst (KSCN) in separate baths) is more appropriate in practice in modifying silk fabrics to have higher light resistance and excellent crease-proof, without impairing its natural lustre and hydrophilicity. The fault of the 'water spot' on tussah silk fabric is greatly reduced by such treatments.

A silicone-containing epoxide named as EPSIB was prepared for silk crease-resist finishing by Cai et al. (2001). An aqueous silicone-containing epoxide EPSIB was prepared by the prepolymerisation of cationic emulsion and a subsequent reaction with 0.5 mol of epoxy chloropropane at the optimum pH value of 9 at a temperature of 65°C for 90 minutes. A two-dip-twonip process can be applied to finish silk fabric with the liquid at 100% pickup. Then the sample was dried at 60°C for 2.5 minutes, and finally steamed at 110°C for 5 minutes. Both dry and wet resiliencies of the silk fabric finished with EPSIB increased with an improvement of fabric dimensional stability, whereas other properties were hardly influenced. Clearly, there is a scope for considerable commercial exploitation for EPSIB in silk finishing. The anti-wrinkle properties of silk fabrics dyed with four bifunctional reactive dyes and three polyethylene polyamine cross-linking dyes (cross-linker 2-chloro-4, 6-di(aminobenzene-4'-β-sulfato-ethylsulfone)-1,3,5-s-triazine (XLC) were investigated by Dang et al. (2010). Compared with the undyed silk fabrics, the WRA of silk fabrics dyed with bifunctional reactive dyes was increased by 14.9%–15.3% when the dye concentration was 0.67 g/L. With the same concentration of synthesised cross-linking dyes, the WRA of dyed silk fabrics was increased by 17.9%–21.3% with a high dye reactivity. The anti-wrinkle properties of silk fabrics dyed with the crosslinking dyes were as good as those finished with 1, 2, 3, 4-butaneteracarboxylic acid. Moreover the anti-wrinkle properties of silk fabrics were improved with the increase of the cross-linking dyes and cross-linker XLC concentration; the handle of these silk fabrics was still satisfactory. The improvement of the WRA of the silk fabrics dyed with cross-linking dyes was about the same as those finished with glyoxal or epoxide. It may be concluded that these cross-linking dyes could lead to not only a high dye fixation, but also an excellent wrinkle-resistant properties of silk fabrics. Additionally, it is feasible to dye and finish silk fabrics with cross-linking dyes simultaneously. Poly-carboxylic acids which are non-formaldehyde reactants are possible replacement for conventional finishing reactants like N-methylol agents or N-methyl amides. The main advantage of poly-caboxylic acids is that they are formaldehyde free, do not have bad odour, and produce a very soft fabric hand.

Ecofriendly crease resistance finishes were carried out using synthesised poly-carboxylic acid and dimethylene-urea. Chemicals were synthesised for betted durability and applied on cotton and silk fabrics for improved dimensional stability. Poly-carboxylic acid and dimethylene urea were synthesised using sonication treatment, with ultrasound waves passing through it at

10,000 decibels and centrifugation treatment is given for even dispersion of the converted particles. After centrifugation the size particles were analysed using Dynamic Light Scattering method. Analysed particles were applied on the fabric using a padding process and then curing. As a result the controlled sample and the treated sampled are compared, and the CR angle is checked using the Shirley Crease Recovery Tester. The CR of the PCA-treated cotton and silk fabrics were better than untreated fabric. The PCA-treated cotton and silk samples showed better results in the crease angle than the untreated sample. Cloth weight (grams per metre, GSM), thickness and strength of these samples were also increased. Silk fabric is padded with 70%-80% pick-up, then it is dry and cured for 90 seconds at 150°C. Traditional chemical anticrease treatments with silk are subject to increasing criticism. Fortunately, science development has provided alternative enzymatic processes and other methods to address this issue. On the basis of the microbial trans-glutaminase (MTG) catalysis principle and the theory of improving crease resistance through cross-linking, MTG probably has a special effect on silk fibroin and plays a role in silk crease resistance. MTG is a type of amino-acyltransferases, which can catalyse an acyl transfer between peptide-bond glutamine (acyl-donors) and a suitable primary amine (acyl-acceptors).

They are found widely in a host of different organisms, including mammals, plants, fish and microorganisms (Lilley et al., 1998). In most cases, the result of their reaction is forming the cross-link of proteins via intra- or interε (γ-glutamyl) lysine isodi-peptide bridges, if the primary amine is the ε-amino group of the peptide-bond lysine. The effect of MTG on silk fabric investigated through the Fourier transform infrared spectroscopy (FR). The microcosmic structure of the fibroin through the SEM was analysed. Solo MTG treatment, as well as compound treatments of MTG followed by hydrogen peroxide, protease and ultrasonic, all showed that MTG can improve the crease resistance of silk fabric. It also enhanced its tensile breaking strength or amended damage in the tensile-breaking strength caused by pretreatments. Simultaneously, a comparison with other treatments showed that compound treatment of MTG followed by ultrasonic exerted a better-coordinated effect and conferred better performances, which increased the WRA by 17.4% and improved tensile-breaking strength 11.2%, respectively. At the same time, other performances were still maintained well. However, the effect of solo MTG finishing is not very ideal and needs further enhancement.

# 5.16 Flame-Retardant Finish

According to statistics, about 50% of fires are caused by textiles in the world. Silk fabric is widely used in making pajamas and domestic decoration materials for its lustre, soft handle,

wearing comfort and aesthetic appearance. So it is vital that silk is endowed with a flame-resistant property. Silk has low flammability with a limiting oxygen index (LOI) value of about 23%, which can be attributed to its high nitrogen content (about 15%–18%). But it still needs added flame resistance finish to fulfil some commercial requirements.

Flame-resistant finishing on silk fabrics can be traced back to 200 years ago, when silk fabrics were immersed in the mixture of borax and boric acid in solution, but this mixture is easily removed by water. In the mid-1980s, Achwal reported that silk fabrics treated with a urea phosphoric acid salt by a pad-dry process had high flame resistance with an LOI value higher than 28%; the flame resistance finish was fast to dry cleaning but not fast to washing. To this day, there is still no suitable flame retardant for silk fabrics. When fire accidents occur, the primary hazard is the ignition of clothing by contact with hot surfaces or small open-flame sources. So fabric with a good flame-retardant character should be chosen for garments in order to reduce the unreasonable risk of burn injuries and deaths from fires associated with clothing. Many countries enacted flame retardancy legislation for apparel such as evening dress and pajamas. Presently, many related regulations have been enacted around the world to state the flammability performance and standardise testing methods for flammability of textiles, such as S.I.1985 No. 2043.

# 5.17 Nightwear (safety) Regulations of the U.K and CFR Standard for U.S.

The test procedure under CFR 1610 US Standard provides a method for testing and establishes three classes of flammability performance of textiles and textile products used for clothing, thereby restricting the use of any dangerously flammable clothing textiles (those designated Class 3). The test method requires that a 16 mm (5/8 in) flame impinge on a specimen mounted at a 45 degree angle for 1 second. The specimen is allowed to burn its full length or until the stop thread is broken, a distance of 127 mm (5 in). The results of several specimens are averaged, and a class designation is made based on the flammability performance and surface characteristics of the sample.

Samples resulting in a designation of Class 1 or 2 meet the requirements of the standard. If a sample fails to meet the requirements of the standard, then the sample is designated as Class 3 (rapid and intense burning). For plain surface fabrics, a Class 3 designation results from an average flame spread time of <3.5 seconds. For raised surface fabrics, a Class 3 designation results from an average flame spread time of <4.0 seconds and the ignition or fusing of the base fabric. A flame retardancy treatment of silk fabric by immersion in a mixture of borax and boric

acid solution can be traced back some 200 years. Crosslinking by introducing N-hydroxymethyl (3-dimethyl phosphono) propion-amide and tri-methylol-melamine (TMM) or hexamethylol melamine (HMM) onto silk fabric can improve laundering durability to 30–50 hand wash cycles, and the LOI of the treated silk fabric was above 30%. But the treated silk fabrics would become stiff and yellow and had the problem of formaldehyde releases .Silk, along with other natural fibres, is rendered fire resistant and self-extinguishing by coating with a reaction product of poly-halogenated acid with a cyclic nucleus, such as chlorendic acid and thio-urea. Titanium hexa-chloride, titanium tetrachloride and zirconyl chloride are found to give a good flame-retardant finish for silk because they lower the pyrolysis onset temperature and increase char formation. A multifunctional finish that imparts flame resistance, dimensional stability and easy-care properties has been reported based on N, N'-ethylene-bis (P, Pbis [aziridinyl]-N-methyl phosphine-amide) (Fig. 5.11).

$$\begin{pmatrix}
H_2C \\
N
\end{pmatrix}
=
\begin{pmatrix}
O & CH_3 & H_3C & O \\
P-N-CH_2CH_2-N-P
\end{pmatrix}
=
\begin{pmatrix}
CH_2 \\
CH_2
\end{pmatrix}$$

**Fig. 5.11** Structure of *N*,*N*-ethylene-bis (P,P-bis[aziridinyl]-*N*-methyl phosphinamide)

The well-known flame retardant N-methylol dimethyl-phosphono-propion-amide (MDPA), known as 'Pyrovatex CP', is effective for cotton. Researchers applied it onto silk fabrics and gained a good flame resistance property, but it came at the loss of some physical properties of silk fabrics, such as whiteness, strength, etc. They also applied a self-prepared novel reactive-organo-phosphorus flame retardant named diethyl-2-(methacryloyloxyethyl) phosphate (DEMEP) onto silk fabrics. Only less phosphorus content can make silk fabrics gain excellent flame resistance. Silk fabrics treated with DEMEP can endure 15 laundry cycles. While MDPA can be fixed onto silk fabrics effectively only with the help of a cross-linking agent, hexakis (methoxy-methyl) melamine (HMM) MDPA-treated silk fabrics have better laundry endurance. Both flame retardants can cause silk fabrics to become yellow and damaged, though strength losses are negligible. DEMEP-treated silk fabrics have better elasticity and wear comfort than MDPA-treated silk fabrics. Developing a durable flame-retardant and formaldehyde-free process for silk fabric is still a challenge. Recently, a series of phosphorous-based flame retardants have successfully synthesised and applied them to silk fabrics to produce durable and formaldehyde- free effects. However, there is little research reported on

in the literature about the properties of apparel and sewing ability for silk fabrics after being treated with flame retardant. Such parameters are imperative for garment manufacturers.

Three kinds of silk fabrics with different weaving styles for apparel uses were treated with a vinyl phosphorus monomer dimethyl-2-(methacryloyloxyethyl) phosphate (DMMEP) by a graft copolymerisation technique using potassium per-sulphate as an initiator (Fig. 5.12). The treated silk fabrics can be self-extinguished after being ignited with a candle-like fire, can pass the vertical flammability test and might show some decrease in permeability. The flame-retardant silk fabrics became little stiff, which is helpful for a flat seam appearance, and are more difficult to distort in fabric laying up, panel cutting and making. The formability and sizing stability during sewing, wearing, and pressing process was improved. The flame-retardancy treatment had different effects on the drape property of silk fabrics with different weaving styles. The seam strength decreased for different weaving styles after being treated with the flame retardant

$$CH_2 = C - C - C - CH_2CH_2 - O - P < OCH_3$$

$$CH_2 = C - C - C - CH_2CH_2 - O - P < OCH_3$$

Fig. 5.12 Structure of dimethyl-2-(methacryloyloxyethyl) phosphate (DMMEP).

### 5.18 Antimicrobial Finishes

The textile materials and clothing are susceptible to microbial attack because they provide the basic requirements for microbial growth. The materials made of cellulose and protein natural fibres provide moisture, oxygen, nutrients and temperature for bacterial growth and multiplication. This often results in objectionable odour, dermal infection, product deterioration allergic responses and often similar diseases.

The control of microorganisms (e.g., bacteria, mildews moulds, yeasts and viruses) on textile fabrics extends to diverse areas such as hospitals, the environment and everyday households. Neither natural nor synthetic fibres have resistance to microorganisms; thus various antimicrobial finishes and disinfection techniques have been developed for all types of textiles.

The functions of antimicrobial finishes applied to textiles are

(a) To control the spread of disease and the danger of injury-caused infection

- (b) To control the development of odour from perspiration, stains and dirt on textile materials and
- (c) To control the deterioration of textiles, particularly fabrics made of natural fibres caused by mildew

Antimicrobial finishes protect the fibre from microbial attacks and protect the owner against transfer of pathogenic germs. Silk is relatively resistant to attack by mildew, fungi and other bacteria. But if stored in moist conditions, then damage can occur. The fungal resistance can be improved by treatment with compounds like N-(2, 2'-dichlorvinyl) salicyl-amide and a 0.01%–0.25% solution of benzal-konium chloride. This increases the resistance of silk to mildew during long storage (up to 2 years) even at high relative humidity.

Silk is a biocompatible biopolymer applicable in biomedical and biotechnological devices. In this field, the assignment of antimicrobial activity has great importance. However a general problem related to all antimicrobial chemicals is to find the balance between high biocide activity and the requirements of safe handling, including nontoxic finish processes, and nontoxicity to humans at usual concentrations and environmental demands.

Antimicrobial agents that have been used industrially in recent years include quaternary ammonium salts, metal salts solutions and antibiotics. Unfortunately, some of these agents are toxic and ineffective, which leads to special attention for 'green chemistry' from worldwide research. Chitin, the second most abundant biopolymer in nature next to cellulose, is a high molecular weight linear polymer of 2-acetamide-2-deoxy-d-glucopyranose units linked by 1, 4-glucosidic bonds (Dutta et al., 2002). Chitosan is a de-acetylated derivative of chitin, which is characterised by its nontoxicity, biodegradability and compatibility with other ingredients.

Previous studies (Hussein et al., 1997) reported that d-glucose-amine hydrochloride (chitosan monomer) does not show any growth inhibition against several bacteria in which chitosan is effective. The antimicrobial activity of chitosan is, therefore, related to not only its cationic nature, but also to its chain length. Hebeish et al. treated cotton and silk fabrics with chitosan of low, medium and high molecular weights (210,000, 100,000, and 1800, respectively) at different concentrations. The padded samples were dried at 100°C for 3 minutes, followed by curing at 150°C for 3 minutes, then washed and dried. This was followed by screen printing using a natural dye extracted from prickly pears. Thus, the obtained fabrics were monitored for nitrogen, colour strength after dyeing/printing and antimicrobial activity. The results signify

that the chitosan concentration and its molecular weight determine the magnitudes of both the antimicrobial activity and colour strength of the printed fabric. The silk pre-treated with chitosan followed by dyeing with natural dyes assumes high colour values. Hosseini et al. studied the effects of chitosan on dyeability and the antibacterial properties of silk yarns against two kinds of bacteria: 'Staphylococcus aureus' and 'Escherichia coli' were investigated. The treated silk samples were found to have an antibacterial potential due to the antibacterial property of chitosan. SEM photographs reveal the deposition of chitosan on the treated yarns.

Chitosan is used in the textile field. It is applied by wet thermal curing involving relatively high temperature with energy consumption, costs and possible fabric degradation; moreover, the addition of toxic reagents, such as glutar-aldehyde, is required as a cross-linking agent (Alonso et al., 2009). In UV curing, however, radical species are generated by the interaction of UV light with a suitable photo-initiator, which induces the curing reaction of reactive monomers and oligomers at low temperature and quickly, with a lower environmental impact and lower process cost than thermal process.

Silk samples were prepared with a chitosan add-on of about 2% in order to maintain silk hand characteristics. An antimicrobial activity test was performed according to ASTM E2149-01 method, using E. coli ATCC 8739 as microbes. A test on treated samples revealed the antimicrobial efficiency, with a complete elimination of microorganism. The best results in terms of fastness were found with 1-hour impregnation at 50°C, with a 75% microorganism reduction. The partial loss of antimicrobial activity after washing was due to the loss of chitosan from fibres, as confirmed by dyeing tests with an acid dye to reveal chitosan presence on fabrics. The surface morphology of the treated fabrics was examined by SEM, and the acquired images revealed a good homogeneous distribution of chitosan on the fibres. It was shown that the polymer does not form a coating on the textile, but it covers every single fibre without gluing them.

The worldwide demand for colourants of natural origin, especially yellow or red pigments, is rapidly increasing in food, cosmetic and textile sectors. Among the species examined, common madder (Rubbia tinctorum L) and woad (Isatis tinctoria L) proved to be quite interesting sources of red (alizarin) and yellow (luteolin) dyes, respectively, either for their agronomic characteristics or dyeing properties. Their main disadvantage lies in the order of magnitude of their extraction yield factors (i.e., a few grams of pigment per kg of dried raw material). To overcome this limitation, it has been suggested that other biological sources be exploited, such

as fungi (both moulds and yeasts), bacteria, algae and plant cultures, since appropriate selection, mutation or genetic engineering techniques are likely to significantly improve the pigment production yields with respect to wild organisms. Among the several pigment-producing microorganisms described in the literature, the fungus 'Thermomyces' has been thoroughly studied.

Using the natural fungal extract 'Thermomyces', 100% silk fabric was coloured, and the effects of colouring behaviour and fastness results were analysed. The process parameters, like pH, temperature and time, were varied and the results were optimised. The results were examined by colouring the protein fabric samples at 2% of the fabric weight, and the optimum conditions for coloration and antibacterial finishing were found to be 40 minutes, 30°C and pH 3. The process proves to be more eco-friendly in nature and can be used for medical application to develop wound dressings, face masks, bandages, etc.

# 5.19 UV-Protection Finishing of Silk

One of the major problems with silk fibres is their susceptibility to photochemical degradation. UV-absorber organic compounds that absorb UV-radiation can impart some protection by dissipating the energy as heat. UV absorbers are organic or inorganic colourless compounds with a very strong absorption in the UV range of 290–360 nm. UV absorbers incorporated into the fibres convert electronic excitation energy in to thermal energy. They function as both radical and oxygen scavengers. The high-energy shortwave ultraviolet radiation (UVR) excites the UV absorber to absorb high energy. The absorbed energy may then be dissipated as longer-wave radiation. Alternatively, isomerisation can occur, and the UV absorber may then fragment in to non-absorbing isomers. Screening agents, energy transfer agents and pigments can also be used to deactivate the fibre degradation process.

Organic UV absorbers are derivatives of o-hydroxyl benzo-phenones, o-hydroxyphenyl-triazes and o-hydroxy-phenyl hydrazines. The ortho-hydroxyl group in the molecule helps in absorption and to make the compound soluble in alkaline solution. Organic compounds like benzo-triazole, hydrobenzo-phenone and phenyl-triazine can be used by normal padding or coating applications. Ortho hydroxy-phenyl and diphenyl-triazine derivatives have excellent sublimation fastness and a self-dispersing formulation. It can be applied by a pad thermosol process and also in print pastes. The presence of organic pigment in the fibres helps in better diffusion of light from the substrate, thus providing better protection. Titanium dioxide and

other ceramic materials have an absorption capacity in the UV region of 280–400 nm and reflects visible and IR rays.

# 5.20 Summary

The improvement of functional characteristics of textile has remained important to the textile industry. Economic forces, market demands and environmental concerns will shape the direction that chemical development for functional characteristics will take. Global competitions are requiring textile chemicals at a lower cost by reducing the amount of water to be shipped. Often, the use of a textile chemical involves generating undesirable side effects. Products that require less energy and water to perform these functional properties, such as plasma, will be preferred. With such environmental and energy conservation benefits, textile industries see a promising future for plasma technology in developing high-performance materials for the world market. Cyclodextrins are nontoxic and biodegradable, thereby offering 'green' solutions to enhance the properties and providing new functionalities to textile products. Biopolymers such as sericin are preferred to be used in order to improve functional properties due to their cheaper price than other chemicals and they are environmentally safe. In the future nanotechnology will overcome the limitations of applying conventional methods to impart certain properties to textile materials. The improvements on the application areas of nanotechnology in textile industry such as antibacterial textiles, antistatic textile, flameretardant textiles, etc. will be increasing. Wool has the potential to bring unique properties to nonwovens in areas as diverse as electrostatic air filters and domestic wipes. Anti-felt or shrinkresistant finish processes have been developed for decades in order to obtain easy-care (machine- washable) wool products. Commercially successful shrink-resistant processes used by the textile industry in the past, as well as technologies currently being developed, can be divided into four groups:

- 1. Combination of oxidation and additive polymer processes.
- 2. Additive polymer-only processes.
- 3. Plasma treatment followed by resin
- 4. Enzymatic processes.

It is widely believed that the use of plasma treatment for the production of shrink-resistant wool will become an alternative to the existing processes. Silk materials are luxurious and have many excellent qualities, such as high lustre, comfort in wearing, fine and smooth texture, high silky

softness and excellent drapability; hence it needs little finish to improve its aesthetic properties. However, it does lack several functional properties. For example, silk materials are prone to creases and wrinkles. It gets easily stained and is susceptible to microbial attack and ultraviolet light. It has a high water absorbency, hence it has poor oil and water repellency. Silk is also flammable. Despite of all these deficiencies, one must be very careful in performing physical and chemical treatments on silk, as this delicate fibre may easily lose its lustre and/or softness for which it is so attractive. Therefore the processing of silk is a very skilful job. Until recently the processing of silk was restricted among limited small-scale skillful processors, and classical finishing methods were in vogue. However, in recent years, diversified uses of silk materials have compelled processors to search for newer processes and chemicals. On the other hand, the use of chemicals is getting more and more restricted due to objections from an environmental point of view. In spite of the above fact, many newer chemicals have proved to be promising for the finishing of silk.

# **Chapter 6 Chemical Finishing Processes**

#### 6.1 Introduction

Chemical finishing can be defined as the use of chemicals to achieve a desired fabric property. Chemical finishing, also referred to as 'wet' finishing, includes processes that change the chemical composition of the fabrics that they are applied to. In other words, an elemental analysis of a fabric treated with a chemical finish will be different from the same analysis done prior to the finishing. Typically chemical finishing takes place after coloration (dyeing or printing) but before fabrics are made into garments or other textile articles. However, many chemical finishes can also be successfully applied to yarns or garments. Chemical finishes can be durable, i.e. undergo repeated launderings or dry cleanings without losing effectiveness, or non-durable, i.e. intended when only temporary properties are needed or when the finished textile typically is not washed or dry cleaned, for example some technical textiles. In nearly all cases, the chemical finish is a solution or emulsion of the active chemical in water. Use of organic solvents to apply chemical finishes is restricted to special applications owing to the expense and the real or possible toxicity and flammability of the solvents employed.

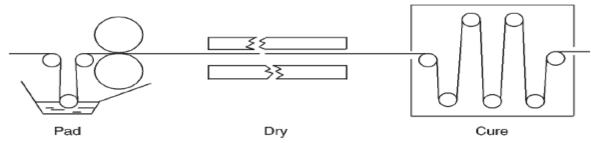
The actual method of finish application depends on the particular chemicals and fabrics involved and the machinery available. Chemicals that have strong affinities for fibre surfaces can be applied in batch processes by exhaustion in dyeing machines, usually after the dyeing process has been completed. Examples of these exhaust applied finishes include softeners, ultraviolet protection agents and some soil-release finishes. Chemicals that do not have an affinity for fibres are applied by a variety of continuous processes that involve either immersing the textile in a solution of the finishing chemical or applying the finishing solution to the fabric by some mechanical means.

After application of the chemical finish, the fabric must be dried and if necessary, the finish must be fixed to the fibre surface, usually by additional heating in a 'curing' step. A schematic diagram of a pad–dry–cure process is shown in Fig. 6.1. Various methods of finish application, drying and curing will be discussed in the following sections.

### **6.2** Application of Chemical Finishes

# **6.2.1 Concentration Relationships**

In batch processes, the amount of chemical finish to be applied is usually expressed as a weight percentage based on the original fabric weight. This relationship is often abbreviated as % owf (percent on weight of fabric) or % owg (percent on weight of goods) as seen in Equation 6.1:



6.1 Pad-dry-cure process. Reproduced from *Cotton Dyeing and Finishing: a technical guide*, 1997, p152, courtesy of Cotton Incorporated, Cary, NC.

$$\% \text{ owf } = \frac{\text{wt chemical} \times 100}{\text{wt fabric}}$$
 [6.1]

For example, if a softener is to be applied at 3 % owf to 500 kg of fabric, then 15 kg of softener will be used (3 % of 500 kg). It must be recognised that since nearly all chemical finishes are provided as an aqueous solution or emulsion, a knowledge of the actual solids concentration of the supplied chemical is needed to determine the actual increase in fabric weight after drying. If the solids concentration is not known or provided, it can be determined by careful evaporation at moderate temperature followed by weighing the residual. But this weight ratio (residue related to the original product sample) is only the upper limit or the maximum concentration of the active finish product. The presence of dispersing or emulsifying agents, salts, unreacted components and by-products may reduce the actual percentage of the active agent compared to the measured weight ratio. A low value of the active products may be determined if they are not solids but liquids and if they partially evaporate with the water during drying.

In continuous processes where a chemical solution or emulsion is applied to a fabric, the amount of chemical actually applied to the fabric depends on the amount of finishing solution applied, the concentration of the supplied chemical in the finishing solution or emulsion and the solids or active compound concentration of the supplied chemical. The amount of finishing solution or emulsion applied is referred to as the 'wet pickup' (wpu) of the fabric and is usually expressed as a percentage on the weight of the dry untreated fabric (Equation 6.2):

$$\% \text{ wpu } = \frac{\text{wt of solution applied} \times 100}{\text{wt of dry fabric}}$$
 [6.2]

To determine the amount of supplied chemical added to the fabric, the '% add-on' is given by Equation 6.3:

% add-on = 
$$\frac{\% \text{ conc in solution (wt/wt)} \times \% \text{ wpu}}{100}$$
 [6.3]

where % conc. is the concentration of the finishing chemical in the applied solution or emulsion expressed as percentage by weight. Since most finishing formulas are given in terms of grams per litre (g l–1), Equation 2.4 can be used to convert the g l–1 concentration to weight percent:

% conc in solution (wt/wt) = 
$$\frac{\text{conc in g l}^{-1}}{10 \times \text{density (g ml}^{-1})}$$
 [6.4]

where the density is the applied solution or emulsion density.

When the actual solids level added to the fabric is desired, the percentage of solids add-on can be found from Equation 6.5.

% solids add-on = 
$$\frac{\% \text{ solids of chemical} \times \% \text{ conc in solution} \times \% \text{ wpu}}{100 \times 100}$$
 [6.5]

## 6.2.2 Pad Application of Chemicals to Dry Fabric

Chemical finishes are often pad applied to dyed or printed fabrics after a drying step. In this situation, dry fabric is passed through the chemical finish solution and the process is called a 'wet on dry' process. The wet pickup of a chemical solution in a pad mangle is influenced by many factors such as fabric characteristics, machine settings and solution or emulsion properties. Table 6.1 summarises some of these factors.

In order to obtain consistent chemical application, the nip pressure should be uniform across the fabric width, the solution level and temperature in the pad should be constant and the fabric speed should not vary throughout the application process.

# 6.2.3 Pad Application of Chemicals to Wet Fabric

To avoid the costs of a drying step after dyeing, chemical finishes are often pad applied to wet fabric in a process called 'wet-on-wet'. In this case, the wet pickup of the fabric exiting the pad must be maintained at a higher level than that of the incoming fabric, usually at least 15–20 % higher. An additional complication is the fact that water entering the pad on the incoming fabric can interchange with the finishing solution, in effect diluting the concentration of the components and causing tailing of the finish effect. Therefore, a chemical feed more

concentrated than the pad solution must be employed. Several authors have addressed this problem and have derived equations to allow the calculation of the required parameters.

Table 6.1 Factors affecting fabric wet pickup

Factor	Effect on wet pickup
Fibre type	Higher wet pickup with hydrophilic fibres
Yarn construction	Higher wet pickup with low twist and/or open end yarns
Fabric construction	Higher wet pickup with loose constructions (knit vs. woven)
Wettability	Higher wet pickup with more easily wetted fabrics
Pressure of squeeze rolls	Higher pressures lead to lower wet pickups
Nature and hardness of squeeze roll coverings	Harder coverings lead to lower wet pickups
Length of immersion time	Higher wet pickup with longer immersion time
Viscosity of solution or emulsion	Higher wet pickup with higher viscosity
Surface tension of solution or emulsion	Higher wet pickups with faster wetting solutions
Temperature of solution or emulsion	Viscosity and surface tension change with temperature, changing wet pickups
Concentration of solution	Viscosity and surface tension change with component concentrations, changing wet pickups

The following is a simplified summary of the necessary equations. In most cases a defined chemical add-on is desired. In order to determine the pad solution or emulsion concentration of the finishing chemicals, an effective percentage wet pickup, wpueff, is calculated from Equation 6.6:

$$wpueff = (wpu0 - wpui) + wpui \times f [6.6]$$

where wpu0 is the percentage wet pickup of the fabric exiting the pad, wpui is the percentage wet pickup of the fabric entering the pad and f is the interchange factor, a measure of interaction between incoming water and the pad solution that can vary from 0 to 1 depending on fabric and machine parameters. Typically f is between 0.7 and 0.8. An initial interchange factor is assumed and then corrected, if necessary based on analysis of the treated fabric.

# 6.2.4 Low Wet Pickup Methods

Typically, pad applications of chemical finishes yield wet pickups in the 70–100 % range. These high pickups necessarily require the removal of large amounts of water during drying. The evaporation of this water can lead to uneven finish distribution in the dried textile owing to migration of the finish to the fabric surface during drying. The high rate of evaporation at the fabric surface leads to movement of the finish solution from the wet fabric interior to the

drier fabric exterior resulting in a higher concentration of the finish at the fabric surfaces with a corresponding lower concentration in the fabric interior regions. This migration is reduced as the finish solution becomes more and more concentrated and viscous as drying progresses. Therefore, reducing the amount of water initially applied will tend to reduce finish migration. However, too low a wet pickup can be equally problematic and also lead to uneven finish distribution if the liquid phase is discontinuous. The concept of a 'critical application value' (CAV) is useful when discussing optimal wet pickups. The CAV is defined as the minimum amount of durable press finish liquid that can be applied to a given cotton fabric without producing a non-uniform distribution of crosslinks after drying and curing. Dye staining tests can be used to determine these distributions. For non-cellulosic fibres, other methods of finish distribution analysis can be used.

The CAV for a particular process is dependent on fibre type and fabric construction and absorbency. A finish application below the CAV may result in a non-uniform speckled treatment, while an application above the CAV could lead to finish migration. Cellulosic fibres, because of their inherent hydrophilicity, have CAVs in the range of 35–40 % wet pickup. Hydrophobic fibres like polyester can have CAVs of less than 5%, allowing much lower wet pickups than hydrophilic fibres.

In order to minimise finish migration during drying and reduce the energy costs associated with drying large amounts of water, various techniques have been developed to reduce the amount of water used in finish applications. An additional benefit is that some applications will allow precise placement of chemicals, leading to the possibility of fabrics with different finishes on their face and back. Another advantage is the recovery and reuse of the finish liquor removed by some of these techniques. To reduce the danger of uneven finish distribution on the fabric, caused by low wet pickup, a thorough preparation is necessary by any method that provides a very good and uniform absorbency. There are two main types of low wet pickup applicators. The first is the saturation—removal type where the fabric is completely saturated with the finish liquid and then the excess liquid is removed mechanically or with a vacuum before drying. With the second type, a precise amount of finish liquid is uniformly applied to the fabric using transfer roll, spray or foam techniques. Table 6.2 gives a survey on low wet pickup finishing application methods, some of which will now be discussed in more detail.

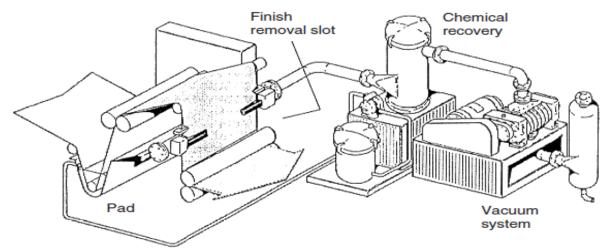
Table 6.2 Low wet pickup finish application methods (adapted from Greenwood and  $Holme)^4$ 

Type of method	Examples
Saturation-removal	Vacuum extraction Porous bowl techniques Air-jet ejectors Transfer padding (squeeze–suck technique)
Topical application	Kiss-roll Loop transfer system Engraved roll Nip padding system Spray system Foam application

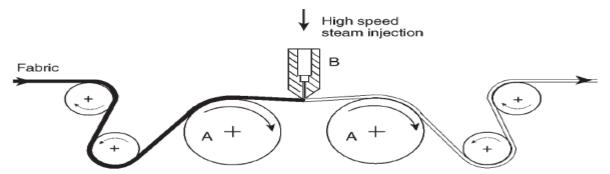
One of the simplest approaches to the saturation–removal has been to place a vacuum extraction device after the application pad and prior to dryer entry. By pulling a vacuum through the wet fabric and returning the extracted liquid to the pad, an effective lower pickup can be achieved, usually in the order of 40 %. Figure 6.2 shows a typical vacuum extraction installation. Another relatively simple method of reducing wet pickup is the use of the Machnozzle system, Fig. 6 3, a machine similar in principle to air-jet ejectors. In this device, high pressure steam is used to push excess liquid out of the fabric, leading to very low wet pickups, especially for synthetic fabrics. In the area of topical application, several methods have been used to apply chemical finishes using transfer rolls. The kiss roll, Fig. 6.4, picks up the chemical finish and transfers it by direct contact to the fabric. The amount of finish picked up is dependent upon how well the finish wets the roll, the absorbency of the fabric, and to a lesser extent, the surface speed of the roll relative to the fabric speed.

Another version of finish application with transfer rolls is the loop transfer system, Fig. 6.5. A loop of fabric is immersed in finish liquid and then squeezed with the fabric to be treated between squeeze rollers. The finish is transferred to the fabric at a much lower wet pickup than possible by direct immersion. These roll transfer techniques are especially useful for the backside application of finishes, for example hand builders and flame retardants, to pile fabrics (without crushing the pile).

One interesting modification of the kiss roll applicator is the Triatex MA machine which uses on-line monitoring to control wet pickup. Figure 6.6 shows a schematic of the system. As the fabric passes through the system, two  $\beta$ -gauges are used to determine the fabric weight intensity of electrons that pass through the fabric. The kiss roll rotational speed is then automatically adjusted relative to the fabric speed to maintain the desired wet pickup.

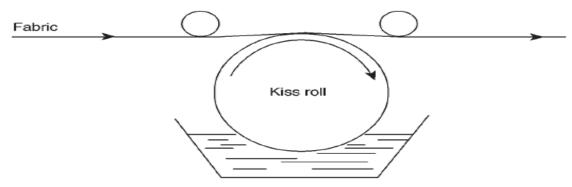


6.2 Vacuum extraction system. Reproduced from *Cotton Dyeing and Finishing: a technical guide*, 1997, p 157, courtesy of Cotton Incorporated, Cary, NC.



63 Machnozzle system. Reprinted from *Textiles Sciences and Technol ogy 11: Textile processing and properties: preparation, dyeing, finishing and performance*, T.L. Vigo (ed.), 1997, Chap 4, p 275, with permission from Elsevier.

difference before and after the fabric has passed over a kiss roll. The  $\beta$ -gauges measure mass per unit area based on the



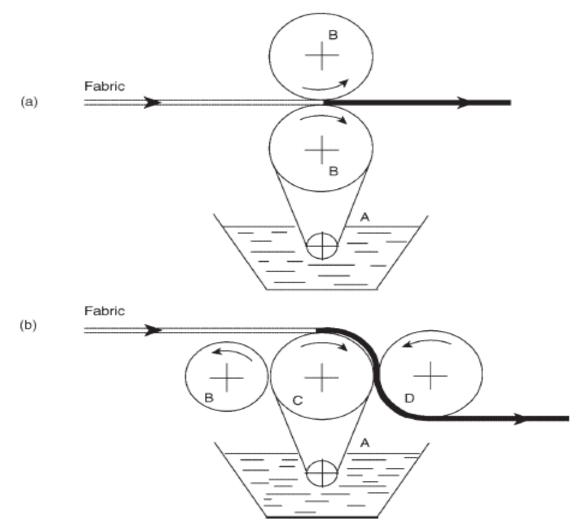
6.4 Kiss roll applicator. Reproduced from Cotton Dyeing and Finishing: a technical guide, 1997, p 153, courtesy of Cotton Incorporated, Cary, NC

An engraved roll (Fig.6.7) can transfer precise amounts of chemical finish to fabrics since the engravings can be made in various depths and designs. A doctor blade removes any excess

liquid from the roll surface before fabric contact insuring that only the liquid in the engraved areas is transferred to the fabric. A disadvantage of engraved rolls is that a roll will deliver the same amount of finish, regardless of the fabric being treated. Therefore multiple rolls are needed if different fabrics are to be treated to the same wet pickup. Chemical finishes can also be applied by spraying (Fig. 6.8). By controlling the flow rate through the spray bars, the amount of applied finish can be set to the desired add-on. Care must be taken to avoid overlapping spray patterns that could lead to an unacceptable uneven finish distribution. Special care is needed with aerosols from fluorocarbon sprays (inhalation, followed by repellency of the inside of the lungs is a deadly danger). One important application method for chemical finishes is the use of foam to apply the finish to the fabric. By replacing part of the water in the chemical formulation with air, the amount of water added to the fabric can be significantly reduced. In addition, surfactants are included in the formulation to be foamed. Even if they are carefully selected, they may cause effect reduction of repellent finishes. The chemical formulation is mixed with air in a foam generator producing high volumes of foam that can be applied to fabrics in a number of ways. The ratio of liquid to air in a foam is referred to as the 'blow ratio', conveniently determined by Equation 6.7:

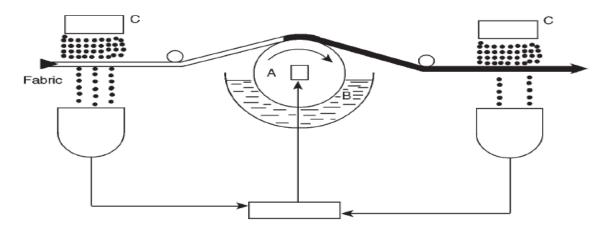
blow ratio = 
$$\frac{1}{\text{foam density}}$$
 [6.7]

Foam densities in the order of 0.1 g cm-3 are routinely used. The stability of the foam is influenced by the components of the chemical system, the viscosity of the foam and the method of foam preparation. The half-life of a foam is the time in which 50 % of the liquid in a given foam volume has been drained from the foam. Foams for textile applications can have half-lives from a few seconds to several hours. Some of the foam application methods are shown in Fig. 6.9 and Fig. 6.10. The one side applicators apply foam to only one side of the fabric, leaving open the possibility of two different finishes on different sides of the same fabric. The two side applicators, on the other hand, apply the same foam to both sides of the treated fabric.

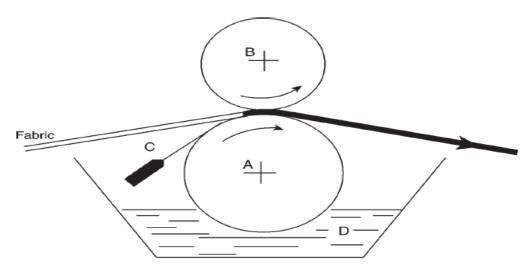


6.5 Loop transfer applicators. Reproduced from Textile Finishing, D. Heywood (ed.). Bradford, SDC, 2003, by permission of The Society of Dyers and Colourists.

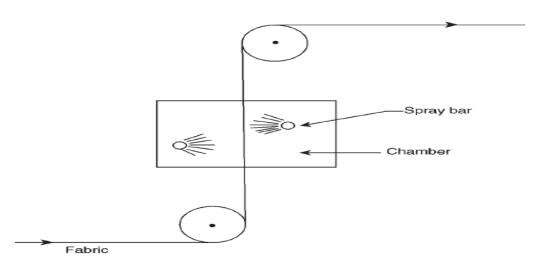
Another two side foam application method is illustrated in Fig. 6.11. This applicator employs two slots to apply the foam to the fabric. Two distinctly different finishes can be applied to different sides of the same fabric simultaneously. Foam application on fabrics with large open spaces or non-uniform porosity often causes uneven finish distribution. Foam application systems also include horizontal pad mangles, kiss coating systems, knife-over-roller or knife-on-air systems, screen printing, and slot applicators.



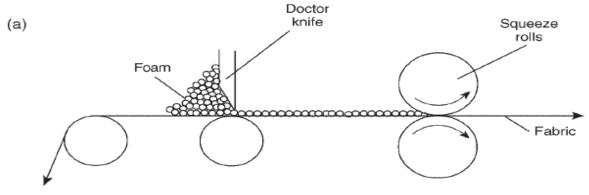
6.6 Triatex MA (minimum application) system. Reproduced from *Textile Finishing*, D. Heywood (ed.). Bradford, SDC, 2003, by permission of The Society of Dyers and Colourists.



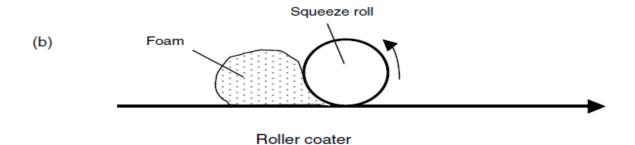
6.7 Engraved roll applicator. Reproduced from Textile Finishing,D. Heywood (ed.). Bradford, SDC, 2003, by permission of The Society of Dyers and Colourists.



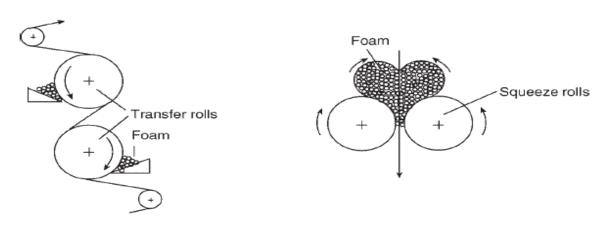
6.8 Spray applicator. Reproduced from Cotton Dyeing and Finishing: a technical guide, 1997, p 156, courtesy of Cotton Incorporated, Cary, NC.



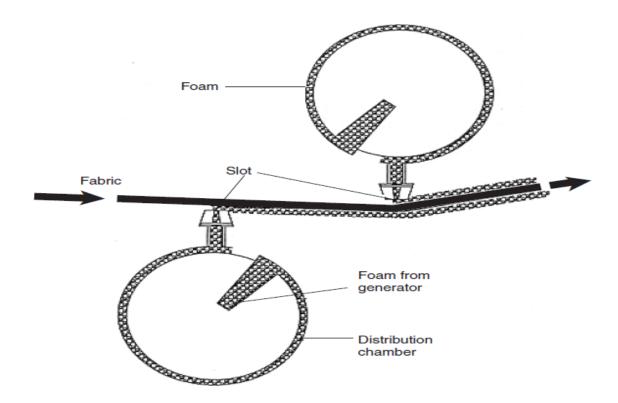
Doctor blade coater



6.9 One side foam applicators. (a) Reproduced from *Cotton Dyeing and Finishing: a technical guide*, 1997, p 155, courtesy of Cotton Incorporated, Cary, NC.



6.10 Two side foam applicators. G.H.J. van der Walt and N.J.J. van Rensburg from *Textile Progress*, 1986, **14**(2), 16–17. Reproduced by permission of The Textile Institute, UK.



6.11 Foam slot applicator. Reproduced from *Cotton Dyeing and Finishing: a technical guide*, 1997, p 155, courtesy of Cotton Incorporated. Carv. NC.

In all these application methods, proper fabric preparation is required in order to achieve uniform finish distributions. A well-absorbent fabric is the best guarantee of a proper finish application.

### **6.2.5** Application of Finishes to Garments

Applying chemical finishes to garments is usually accomplished using exhaustible finishes (softeners, antimicrobials, ultra violet (UV) absorbers, and so on), which are added to the bath of the garment processing machine after all other garment wet processing steps have been completed. However, occasionally, a non-exhaustible finish such as an easy care finish is desired. Special procedures have been developed for this situation. One approach is to extract as much water as possible from the garments and then immerse them into the finish solution, either in a separate trough or in the garment machine itself, followed by further extraction, drying and curing. A second approach is to spray a precise amount of finish solution into the garment processing machine after water extraction. Even distribution of finish is accomplished by rotating the garments long enough to allow the finish to migrate throughout the garment. Drying and curing complete the process. Both methods have been demonstrated commercially, but the spray procedure requires fitting existing garment machines with the precision spraying

attachment, while the immersion procedure involves less capital investment, but consumes more chemical finish.

# 6.3 Drying Wet Textiles

Water in a wet textile resides in three different areas. The most loosely bound water is on the fabric surface and interstices. Much of this water can be taken out by mechanical means such as squeezing, centrifugation or vacuum extraction. The remaining water, the water held in the yarn capillaries and the water absorbed internally by the fibre, must be removed through vaporisation by thermal means. There are three heat transfer mechanisms used to dry textiles. Conduction methods involve direct contact of the wet textile with heated surfaces. These are the most efficient heat transfer methods, but do not allow for control of fabric width during drying. Steam heated cylinders are examples of conduction drying methods (Fig. 6.12). High pressure steam inside the cylinders provides the energy necessary to dry the fabric.

Convection methods involve contact of the wet textile with hot air and are the most common method used in textiles since they combine high process speeds with control of fabric dimensions during drying. Examples include tenter frames (Fig. 6.13). Air is heated to the desired temperature by gas- or oil-fired burners or steam heat exchangers and passed over the fabric by high velocity blowers. Fabric tensions are adjusted in both the width and length directions, allowing for complete control of final fabric dimensions. The third heat transfer mechanism is radiation, examples of which are infrared and radio frequency dryers. Radiant heaters are often used as predryers, removing much of the moisture from wet fabric prior to entering the actual drying process (Fig. 6.14). Use of predryers minimises finish migration and increases dryer productivity since less water must be removed in the dryer. A more detailed description of drying methods and machines is given by Miles.

## **6.4 Curing Chemical Finishes**

The same heating equipment used to dry wet textiles can also be used to heat the fabric and finish to the temperatures desired for optimal curing. For all equipment, it must be remembered that the temperature of the fabric cannot exceed  $100\,^{\circ}\text{C}$ 

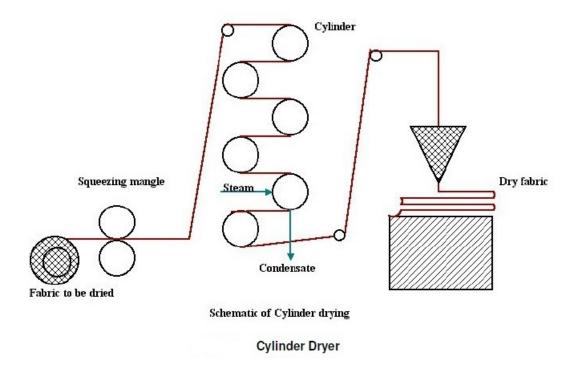
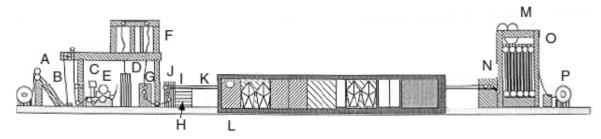


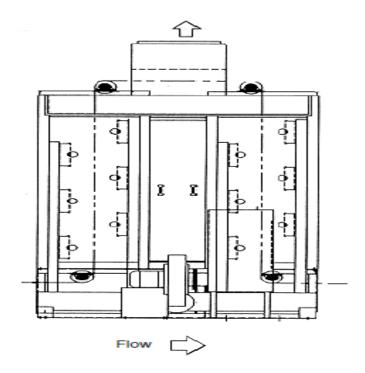
Fig.6.12 Steam heated drying cylinders.



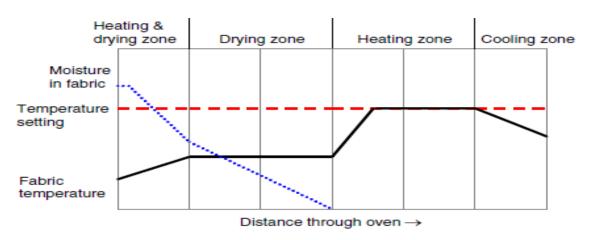
6.13 Tenter frame. Reproduced from *Cotton Dyeing and Finishing: a technical guide*, 1997, p 166, courtesy of Cotton Incorporated, Cary, NC.

until all of the water has been removed. Figure 6.15 demonstrates this effect. The fabric temperature does not rise to the set temperature until after all the water has gone. When drying and curing are done separately in two steps, the curing time can be controlled easily. As speed is defined by distance divided by time, the curing time can be calculated by Equation 6.14:

For example, if the fabric content of the machine is 20 m and the fabric speed is 40 m min-1, then the curing time is 0.5 min.



6.14 Predryer. Reproduced by permission of Aztec Machinery Company, USA.



6.15 Temperature and moisture profiles in the tenter.

Often drying and curing are combined in one process, for example the so-called shock-condensation or shock-curing processes. As the end of the drying phase is not easy to determine, there is a risk of over- or under-curing with many disadvantages. The best available solution for this problem is curing controlled by the temperature of then fabric. As shown in Fig. 6.15 only when all the water is evaporated, will the temperature of the fabric rise from the wet-bulb temperature to the temperature of the surroundings and the curing process can start. With radiation pyrometers the surface temperature of the fabric is exactly measured free of contact. Thereby the end of the drying step and the time of the curing step can be determined and monitored. As radiation pyrometers are relatively expensive, often not all the sections of

long tenters are completely monitored by pyrometers; they are concentrated in and most important in the tenter section where drying ends and curing starts.

# 6.5 Coating and laminating

A short survey on coating might be of interest, because there is a smooth transition between chemical finishing with one-side application of greater amounts of products, for example stiffeners or flame retardants, and fine or thin coating. Fine coating is characterised by adds-on of about 4–50 g m–2, mostly 15–20 g m–2. Also some application techniques are similar for one-side finishing and for coating, for example those using doctor knifes, rolls, rotary screens, foam or spray applications. The chemical finishing processes discussed in the following chapters have the goal, for the most part, of producing a finish uniformly distributed throughout the textile material. Except for specific performance properties, the treated fabric is nearly indistinguishable from untreated fabric since the physical appearance of the textile is seldom changed by conventional chemical finishing. However, other forms of chemical finishing are practiced where the goal is to produce a textile with a layer of chemical finish on, in or in between substrates leading to nonhomogeneous structures. Coating and laminating are two such processes. With coating, the intent is to add the chemical finish to a substrate, while with laminating, the purpose is to join two textiles into one structure with the chemical acting as the adhesive.

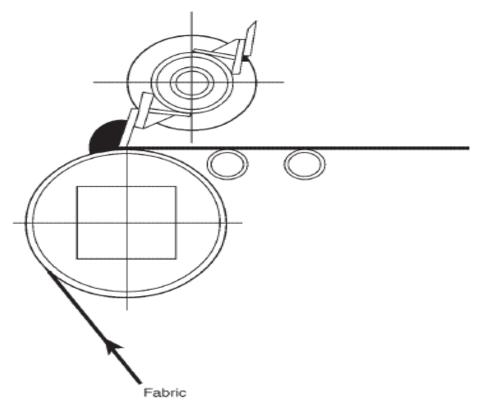
A variety of useful products are produced by coating and laminating. Coating and lamination technology provides products for automotive air bags, footwear, interlinings, upholstery, hats, labels, umbrellas, adhesive tapes, rainwear, protective clothing, artificial leather articles, window blinds, tents, sleeping bags, curtains, floor coverings, luggage, sails, mattress ticking, flexible fuel tanks, abrasive products, awnings, filter fabrics, geotextiles, hoses and many others.

The entire market sector of technical textiles benefits from coated and laminated products. Both coating and laminating require a textile substrate to be treated. The substrate plays a major role in establishing the final properties of the finished article. In addition to the chemical and physical properties of fibres themselves, yarn construction and fabric formation are significant factors. Yarns made from staple fibres provide rough surfaces that enhance adhesion to chemical coatings. Filament yarns generally must be pre-treated with chemicals to generate a more reactive surface prior to coating or laminating. Fabric structure determines the extent of textile-finish interfolding as well as influencing the final mechanical properties of the treated

material. Knitted and non-woven structures are especially useful for coating and laminating but when strength and dimension stability are required, wovens are preferred.

The chemicals used for coating and laminating are polymeric materials, either naturally occurring or produced synthetically. These include natural and synthetic rubbers, polyvinyl chloride, polyvinyl alcohol, acrylic, phenolic resins, polyurethanes, silicones, fluorochemicals, epoxy resins and polyesters. Coating formulations typically include auxiliaries such as plasticizers, adhesion promoters, viscosity regulators, pigments, fillers, flame retardants, catalysts and the like. The combination of the textile fabric and the polymer matrix on it results in interesting new properties. Thus coated textiles can be both flexible (similar to fabrics) and water- and air-tight (similar to films). Generally, the textile component provides strength and/or flexibility, the polymer coating delivers thermal insulation and barrier functions against liquids, gases and light. Both components contribute to various aesthetic requirements. The most common coating application method is the knife over roll system (Fig. 6.16). The shape and angle of the coating blade, the gap between the blade and the fabric and the viscosity of the coating all affect the amount of coating applied and the penetration into the fabric. Usually a direct coating consists of two or three layers. The first base or tie coat delivers adhesion to the fabric, the main layer (top or cover coat) consists of the dominating type of polymer with all the additives necessary for the required properties, and often there is a final or finish coat for protective and aesthetic demands. Reverse or transfer coating follows a contrasting order of the layers. It is used when the textile component does not have enough dimensional stability or has a structure which is too open for the direct coating process. Therefore a support foil, called release paper is used, first coated with the finish layer, then with the main layer, followed by the tie coat and at last the textile, for example, a knit-wear or a thin non-woven. Every coat step needs a short pre-gelation. After the final heating and end-gelation the release paper is separated. After the coating is applied, the fabric can be heated to evaporate water and other solvents and cured if required by the polymer system. Some coated fabrics are embossed or printed, depending on the fabrics intended end use.

The laminating process involves applying an adhesive coating to the surface of the primary substrate, bringing the second substrate together with the adhesive layer, thereby making a three component composite, and finally with heat and pressure forming the final laminate. The adhesive can be applied by a variety of techniques including the knife over roll method mentioned earlier. Other application methods include scatter coating of thermoplastic polymers and rotary screen printing of adhesive emulsions or solutions.



6.16 Knife over roll coater. Reproduced from Textile Finishing,D. Heywood (ed.). Bradford, SDC, 2003, by permission of The Society of Dyers and Colourists.

According to the immense market importance of coatings and related fibre composite products, there are many special processes and products, including front-, back- and double-side coatings, water vapour permeable coating (incorrectly called breath-active), foam and spray coating, flame laminating, bonding, flocking, hot-melt and paste-dot coating for fusible interlinings, pre-pregs and other textile composite materials for wide technical usage.

# **Chapter 7 Softening of Textiles**

#### 7.1 Introduction

As a general rule, each fibre has its specific softness value, which depends on its chemical composition and physical structure; the lesser the crystallinity, the greater the softness. The fineness of the fibre or of the filament also directly affects the softness of the yarn (e.g. woollens, worsteds, micro-fibres, etc.). The yarn twist ratio is inversely proportional to its softness. The weave also contributes to the fabric softness; softness reduces while making the weave more compact and increases for a more loose or open-structured weave. Furthermore, a greater number of yarns per centimetre increase the stiffness of the fabric, thus reducing its softness. While softening, the composition and properties of the substrate should be carefully considered. It is also worth noting that no standard methods have been developed or established to determine exactly what the softness of a fabric is. This evaluation is therefore almost personal and carried out on the basis of the operator's experience. It is possible to distinguish the following types of softness:

- (1) Surface softness
- (2) Surface smoothness
- (3) Elasticity (compression and stretching)

To change the hand properties of a fabric, we can apply mechanical, physical, chemical or combined techniques. As a general rule, the softening agents are hygroscopic or lubricating agents, which facilitate the fibre sliding within the fabric structure, thus granting easier deformation and creasing of the fabric. Softener is an auxiliary that, when applied to textile materials, brings about an alteration in the handle, resulting in the goods being more pleasing to the touch. Just as it is difficult to define the term 'softener', the definition of 'handle' also presents problems. 'Handle' is such a subjective property that the choice between two different softeners is usually made by actually handling the treated goods rather than on the basis of quantitative measurement. One of the more important attributes used in the textile and apparel supply chain is fabric handle. Evaluation of fabric quality has traditionally been assessed subjectively in both the textile and clothing industries. The simplest and most widely used method of subjectively assessing fabric quality is through the notion of fabric handle. The term 'hand' has been defined as 'the subjective assessment of a textile material obtained by the sense of touch'. Hand is thus a physiological and psychological phenomenon; it implies the ability of the fingers to make a sensitive and discriminating assessment and of the mind to integrate

and express the results in a single valued judgement. Such a judgement cannot be obtained objectively, since the relevant mechanism of the sensory organs, the nervous systems and the brain are not known and vary with each individual and also vary on successive judgements for each individual.

The systematic evaluation of handle, based on a definition that is specific to the sense of touch, might be expected to involve taking precautions in order to exclude bias caused by fabric appearance and possibly even by fabric odour, as well as any rustling sound made by some fabrics. The hand of the textile material is most important quality for most buyers and users. Softness has always been an important aspect in textile finishing, and it has become even more important when the synthetic detergents entered both industry and the household. Due to the removal of natural fats and waxes which had given the fibres a natural softness and smoothness, the usage of a lubricant at the end of the finishing process became necessary.

Handle correction is absolutely necessary for new processes that came up, such as resin finishing, or for the touch of fabrics which should possibly be like synthetic fibres, as well as for an increased comfort consciousness in general. Due to innovations in the textile industry as well as in the processing stages, the demand for softening agents has increased. Textiles produced from open-end yarns showed a distinctly harder, more brittle handle than ring-spun yarns, and classical softeners could not sufficiently compensate to handle such differences. A new problem came up that was caused by the increasing number of jet dyeing machines and overflows. In view of the high shear forces in the short liquor technique, softeners with a low foaming tendency and a high shear resistance are required for this purpose.

As far as the making up of knit goods is concerned, the usage of extremely fast running sewing machines leads to more and more problems with loop damages. The requirement for better sewing properties could not be met in an optimal manner by classical softeners. Almost all apparel and home furnishing are treated with softeners. Only a few varieties of textile, namely, wall coverings, carpets and industrial textiles, are not softened. Softeners are the most important global textile finishing chemicals in terms of value (19.9%) and amount (22.1%).

Fabric softeners have been in use since the 1930s. However, their demand increased greatly in the 1950s due to the introduction of synthetic fibres and synthetic detergents, as well as the modern washing machine and automatic dryer. The mechanical agitation of automatic washers causes abrasion and surface fuzz formation in fabrics, as well as distortion and entanglement of fibres, more rapidly than earlier wringer washing machines. The entangled fibres were

further set by the automatic dryers; this produced a stiff and harsh hand which prompted interest in and created a market for home laundry softeners. Moreover, synthetic detergents remove lubricating oils and waxes in natural fibres, thereby resulting in very clean but harsh and scratchy fabrics that are uncomfortable to wear. Fabric softeners counteract these effects. Synthetic fibres are prone to static build-up, which can be objectionable to the wearer. Fabric softeners act as antistatic agents by enabling synthetic fibres to retain sufficient moisture to dissipate static charges. Household softeners, both liquid and dryer sheets (for impregnation of sheets used in dryer), work by forming a coating of yarn and fibres with lubricants and humectants. The lubricants make the fabrics feel smooth, soft and flexible by an internal lubrication of the fibres. Humectants help fibres retain moisture, a factor that is crucial in antistatic treatment.

The softeners are mainly used for textile processing for the following:

- (1) To provide desired softness often described as smooth, supple, super soft, elastic, dry or slashy.
- (2) To influence or to improve technical properties, namely, antistatic, hydrophilicity, elasticity, sew-ability and rubbing fastness.
- (3) To confer natural touch on synthetic fibres and to improve their wearing comfort by the regulation of moisture content or smoothness.

The handle is a subjective sensation felt by skin when the textile material is touched by a hand or finger and gently pressed and moved over its surface. The perceived softness is due to a combination of several measurable physical phenomena such as elasticity, compressibility and smoothness. The textile materials become harsh after the preparatory processes due to removal of natural oils and waxes. The finishing with the softeners restores original suppleness or enhances softness to a higher degree. The additional improvements by the softening treatment are fullness, antistatic properties and sew-ability. However, the treatment may impair certain other properties like increased yellowing of goods, reduced crock fastness and change of hue of dyed fabrics and instability of fabric structure.

Linen fabric is a quite stiffer fibre when compared to cotton and polyester, and it is also stiffer than its blends. Linen is very sensitive to the applied finishing processes, and a significant decrease in tensile properties occurs during the processes. However, the breaking load of the cotton and polyester fabric constituents did not have a significant decrease.

The combination of chemical-mechanical and enzymatic-chemical-mechanical finishing technologies were used in large-scale production for improving the softness and hand of linen and fabrics such as cotton/linen and polyester/linen blends. Results prove that a mechanical treatment has the most significant effect on fabric stiffness, whereas an enzyme application yields fabrics with a good appearance and luxurious hand. Both mechanical and enzymatic treatments weaken the linen considerably but do not alter the breaking load of cotton and polyester constituents. Crease recovery of linen and linen/cotton fabrics improves only slightly with the applied finishing, whereas polyester/linen end products have good crease recovery properties.

## 7.2 Properties of Softeners

The essential properties of a textile softener are as follows:

- (1) Available in forms convenient to handle: stable liquid, pre-dilutable and dosable
- (2) Compatible with common textile auxiliaries
- (3) Non-volatile and stable in high temperature
- (4) Non-yellowing
- (5) Should not affect colour fastness of dyed materials
- 112 Principles of Textile Finishing
- (6) Low foaming and shear stable; does not deposit on rollers
- (7) Preferably applicable by exhaust processes with good properties
- (8) Ecofriendly, nontoxic and dermatologically safe
- (9) Biodegradable
- (10) Good price-performance relationship

The requirement profile for textile softeners is:

- (1) Textile characteristics: handle, volume, softness, fall and odour
- (2) Mechanical properties: stretch, elasticity, abrasion resistance, tensile strength, tear strength, smoothness, pilling tendency and sew-ability

- (3) Functional properties: moisture management (hydrophilic/hydrophobic), antistatic, flame retardant, dirt resistant, sew-ability, rope crease prevention and antimicrobial
- (4) Factors specific to production: environmentally acceptable (in manufacture and use); resistant to acids, and alkalis; stability to heat and frost during storage; jet suitability (reduced foaming and stable shear force; viscosity suitable for metering; good solubility and compatible with bleach liquor, dye liquor, optical brighteners, synthetic resins and other chemical finishes

# 7.3 Chemistry of Softeners

The major share of softeners for the final finishing being available in the market today is at least partly based on fatty acid amine condensation products. A variation of the reaction partners and reaction conditions allows for a variation of the ionic character (e.g. cationic, pseudo-cationic, non-ionic or amphoteric) and the molecular weight to a wide extent.

By the ionic character, the molecular structure and the molecular weight and essential properties of the softener, such as yellowing, softness and shear resistance, can be controlled. Such fatty acid amine condensation products had satisfied the market requirements for many years. In the 1980s, the functionality of lubricants started to increase by putting additives into the softener formulation. Due to the market requirements for softer and softer handle effects, the silicone chemistry thereby was of great importance. An intelligent formulation of fatty acid amine condensates with selected additives not only results in better handle in general but also additional benefits. Improved softeners like true hydrophilic, low foaming, jet stable and sewability enhancer for use in modern short liquor jets have been developed. The softeners are surface active agents; that is, their molecules contain both hydrophilic and hydrophobic components. Typically, the molecules contain a long alkyl group, sometimes branched, of 16–22 carbon atoms, but most have 18 corresponding to the stearyl residues; silicones, paraffins and polyethylene softeners are exceptions. About one-third of the textile softeners are silicone based.

The softening agents are classified according to their ionic nature, namely, anionic, cationic, amphoteric and non-ionic. These are further classified according to their chemical compositions as follows:

Anionic

- (a) Sulphates
- (b) Sulphonates

(c) Sulpho-succinates
Cationic
(a) Quaternary ammonium salts
(b) Amino-amides
(c) Cyclic cationics (e.g. imidazolines)
Amphoteric
(a) Sulpho-betaines
(b) Fatty amino acids
(c) Amine oxides
Non-ionic
(a) Ethoxylates
(b) Esters
(c) Poly-ethylenes
(d) Silicones
(e) Waxes
<b>7.3.1 Anionic Softeners</b> Anionic softeners are being used less because of their somewhat poor handle and low substantivity, but they are still of importance for certain fields:
(1) As sanforising assistance due to their good rewetting properties
(2) As crease-preventing agents in dyeing processes (anionic products do not retard at all, or only to a small extent)
(3) For a one-bath application with other anionic auxiliaries such as optical brighteners
(4) As a raising assistant due to good smoothing and antistatic efficiency

(5) As a sizing fat

They have general formula RSO3·M or ROSO3·M (where R = alkyl or aryl and M = Na or K). Generally, the group contains products like:

- (1) Alkyl aryl sulphonates
- (2) Alkyl sulphates
- (3) Sulphated or sulphonated amides and
- (4) Alkyl ether sulphates

Anionic softeners have limited use in textile processing at present. They are used in raising and sanforizing processes, resulting in the improvement of smoothness/antistatic and rewetting properties, respectively. They are also used for combined finishing with other anionic substances. Sulphonates, in contrast to sulphates, are resistant to hydrolysis. They are often used for special applications, such as medical textiles, or in combination with anionic fluorescent whitening agents.

## Anionic softeners are:

- (1) Heat stable at normal textile-processing temperatures
- (2) Compatible with dye and bleach bath additives
- (3) Nondurable (i.e. easily washed off)
- (4) Able to provide strong antistatic properties
- (5) Capable of rewetting properties, as their anionic groups are oriented outwards and are surrounded by a thick hydration layer

Clariant (www.clariant.in) marketed Ceranine AS Liq., a non-yellowing anionic softener for white fabrics that is compatible with optical brightening agents (OBAs). It is a derivative of an amide compound and can be applied both by exhaust and padding methods. Exhaust method: 2–3% on the weight of fabric exhausted at room temperature for 30–45 min at pH 4.5–5.5 followed by drying. Padding method: 15–30 g/L padding with 70–80% pickup, followed by drying at 110–130°C (Sandoz, 1985).

### 7.3.2 Cationic Softeners

The cationic products were first introduced in the late 1930s for improvement fastness of direct dyes on cotton. But later it gains popularity mainly as softener. The worldwide consumption

of cationic projects in 2005 is about 390,000 tons, with an average annual growth of 4%. This class of softeners is most widely used because of its high degree of substantivity (especially to cotton and acrylic fibres) and effectiveness at low concentrations. Cationic softeners show the best soft handle and are therefore used for household articles as well as for industrial articles. They have affinity to almost all fibres and are usually applied by the exhaust method in acid environment (pH 4–5). The only problem is the incompatibility with anionic auxiliaries (optical brighteners, dyeing auxiliaries) as well as their tendency to yellow in comparison to non-ionic products. Cationic softeners are mainly used for coloured textile substrates. Until recently, household fabric softeners were formulated from three main chemical classes of cationic surfactants: dialkyl-dimethyl ammonium compounds, imidazolinium compounds and diamido alkoxylated ammonium compounds. In the 1990s, esters of dimethyl ammonium compounds (so-called ester quats) displaced all other softener types in Europe due to a higher biodegradability of dialkyl quaternary compounds.

According to their chemical nature, pseudo-cationic softeners have a pH-dependant positive charge. The organic quaternary compounds (QUATs) have pH-independent positive charge. Due to their affinity for the textile fibres, the cationic products can be applied by both padding and exhaust processes even from baths with high liquor to goods ratio. They are exhausted in most of the textile fibres, imparting the best softness and durability. As hydrophobic groups are oriented away from the fibre surface, they provide a good hydrophobic surface but poor rewetting properties. They are precipitated in the presence of anionic products.

A classical cationic softener can provide good handle to the fabric and possess good exhaustion properties, on the other hand, however, it shows weaknesses when it comes to whiteness compatibility and hydrophilicity.

Some other disadvantages of cationic softeners are:

- (1) Poor compatibility with anionic products such as optical brighteners and dyeing auxiliaries
- (2) Higher soil retention
- (3) Yellowing when exposed to high temperature
- (4) Lowering of light fastness of some direct and reactive dyes
- (5) Fish toxicity and poor biodegradability of many quaternary ammonium compounds. The various chemical classes used for producing cationic softeners are as follows.

## 7.3.2.1 Salts of Primary Amines

General formula: RNH2·HX (where R = alkyl, X = chloride, acetate, glycolate, etc.) (e.g. octadecyl ammonium chloride). The manufacturing stages are:

Fatty acids + ammonia  $\rightarrow$  fatty amides  $\rightarrow$  (dehydration) fatty nitriles  $\rightarrow$  (hydrogenation) fatty amides  $\rightarrow$  (neutralisation with acid) cationic products.

For adequate lubricity and softness, long-chain derivatives are required. Due to poor solubility, this group is relatively unimportant as textile softener.

## 7.3.2.2 Salts of Tertiary Amines

General formula:  $RN(R/)(R//)\cdot HX$  (where R = alkyl, X = chloride, acetate, glycolate, etc.). The manufacturing stages are:

Secondary amines  $\rightarrow$  (alkylation with fatty chlorides/alcohols) tertiary amines  $\rightarrow$  (neutralisation with organic/inorganic acid)  $\rightarrow$  salts of tertiary amines. Numerous members of this group are used as emulsifiers, scouring and wetting agents.

# 7.3.2.3 Quaternary Ammonium Salts

General formula: RN(R/)(R//)(R///)X (where R = alkyl, X = chloride, alkyl sulphates), such as distearyl dimethyl ammonium chloride. Despite having better solubility than the above two classes, their uses are limited due to low biodegradability.

### 7.3.2.4 Salts of Amino Amides

General formula:  $RCON(R/)(R//)[N(R///)(R///)]n\cdot HX$  (where R = H and/or alkyl, X = acetate, glycolate). These important types of softeners can be prepared by the amidation of a polyamine with fatty acids or by aminolysis with triglycerides (e.g. hydrogenated fat or oil). The use of hydrogenated tallow or palm oil is very common.

The most common polyamines are amino-ethylethanol (Fig. 7.1), diethylene triamine (Fig. 7.2), tri-ethylene tetramine and tetra-ethylene pentamine. The solubility of water and mild alkali increases along with the number of amino groups. The amidation results a complex mixture. The amidation of the polyamine (Fig. 7.1) with fatty acid is found to be a mixture of mono-amide, diamide and ester amido-amide (30%, 55% and 12%, respectively), ester diamide and imidazoline also form in small quantities. All products are neutralised with acids to form the corresponding salts, which are pseudo-quaternary. The products show excellent softness on

various textile materials. Alkali solubility can be improved by the alkylation of the amino amides with alkyl halides/sulphates to form quaternary ammonium salts. The degree of hydrogenation of the fat is very important; the lower the iodine value, the better the softness and the less yellowing during drying.

Fig. 7.1 Aminoethylethanol.

Fig. 7.2 Diethylene triamine.

### 7.3.2.5 Salts of Imidazolines

Imidazolines are prepared by reacting fatty amides with polyethylene polyamines with simultaneous removal of water. These are further alkylated with alkyl halides/ sulphates to form quaternary imidazolines. The residual amino groups can be neutralised with organic acids to form corresponding ammonium salts.

### 7.3.2.6 Salts of Amino Esters

General formula: RCOOR/N(R//)(R///)(R///)X (where R = alkyl, hydroxy-alkyl and X = chloride) can be obtained by reacting fatty acids with amino alcohol. Mono-, di-or triethanol amines may be used. The corresponding quaternary salts can be produced with alkyl halides/sulphates.

## 7.3.3 Amphoteric Softeners

Formulations based on amphoteric substances are usually for special products of certain applications. Amphoteric products give an average handle, are normally compatible with white and give the fabric a good hydrophilicity, as well as excellent antistatic properties. Furthermore, amphoteric softeners are very sensitive to skin and are often biodegradable. The main application range is hygiene and terrycloth articles.

They are characterised by good softening and high antistatic effects due to a strong ionic character, but they have poor durability in washing. The chemical structure of amine oxide type amphoteric softener is shown in Fig. 7.3 (alkyl-dimethyl-amine oxide, where R1 is a long alkyl

chain), whereas betaine-type amphoteric softeners are shown in Fig. 7.4A–C. Amphoteric softeners compounds are compatible with human skin and are often used for hygiene articles. They are widely used for finishing of terrycloth towels.

Fig. 7.3 Alkyldimethylamine oxide amphoteric softeners.

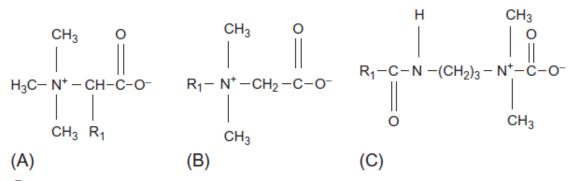


Fig. 7.4 Betaine type amphoteric softeners.

### 7.3.4 Non-ionic Softeners

Non-ionic softeners do not carry any electrical charge and therefore do not possess any distinctive substantivity. Such products are applied by means of forced application (i.e. usually in padding mangle procedures). Non-ionic softeners can be combined universally, are stable to temperature and do not show yellowing. This is the reason that this product class is perfect for finishing optically brightened, highly white articles. The soft handle of pure non-ionic products is only average. Compared to cationic softeners, a classical non-ionic softener shows a superior yellowing resistance. With respect to wash fastness, handle and exhaustion behaviour; however, such a product is disadvantageous when compared to cationic softeners, which are generally based on etho-xylates and esters. The compounds of fatty esters are probably most widely used. In addition to softening, they impart a high degree of lubrication.

For etho-xylates, the fatty radical confers the softness of handle and the etho-xylate chain confers the antistatic properties and absorbency. Unfortunately, the situation is not as simple as this. Acrylic fibres, the molecule will align itself so that high fibre—fibre static friction will be generated, resulting in a very scroopy handle.

Esters, especially linear esters such as methyl stearate or oleyl oleate, tend to have very good lubricating properties and do not have the disadvantages associated with the ethoxylates. However, they do not have antistatic properties and, being nonpolar, usually need dispersing in an aqueous medium.

Poly-ethylenes are widely used when crease-resistant or wash-and-wear resins are applied to cotton and its blends to improve their tensile properties. The resins tend to increase fibre friction, thus restricting the movement of individual fibres within the fabric, which the lubricant overcomes. Poly-ethylenes are very suitable because of their ability to surround individual cotton fibres and give this reduction in friction. Further, they are compatible with the resins and catalysts commonly used, do not affect resin— fibre interaction and are stable to heat treatment.

Waxes used in softener formulations are essentially solids which confer a degree of lubricity to the fibre. Paraffin waxes are very commonly used, and others include solid esters and the synthetic Fischer–Tropsch waxes. Paraffin waxes are used to provide low fibre-metal dynamic friction, such as for knitting or sewing aids. They can be left on many knitted garments and give a 'waxy' handle to the fabric. Paraffins are well known for their application in smoothening agents. Very interesting are paraffins with a carbon chain length of C24–C32. Paraffins with a shorter C-chain vaporises rapidly, whereas those with longer chains can be converted to usable emulsions only under pressure.

Except for the ethoxylates, all the non-ionic softeners give good lubricity, but none confers a soft, bulky handle. Hence, a product that would give good stability or good physical properties to a fabric could easily be formulated from non-ionic agents alone, but if any degree of softness were required, then an anionic, cationic or amphoteric component would be needed.

In the case of non-ionic products, a fatty chain of high molecular weight (i.e. saturated compound of C16–C18) provides the softening effect, and the solubility is provided by condensation with an ethylene oxide chain (EO). They have a general formula, R(OC2H4)nOH or R(C2H4)nOOH (where R = alkyl), and contain different non-ionic components such as fatty alcohols, ethoxylated fatty alcohols and fatty amines, paraffins and oxidised polyethylene waxes as active ingredients. The emulsifiers may be needed to obtain stable emulsions. Some of the production routes for EO condensation are:

- (1) With fatty acids forming a fatty acid poly-glycol ester (R-COO-(CH2-CH2-O)n-CH2-CH2-OH) imparting a soft silk-like, scroopy handle.
- (2) With fatty acid amines and amides forming alkyl-amine poly-glycol ether (R–NH–(CH2–CH2–O)n CH2–CH2–OH) or fatty acid poly-glycol ether (R–CONH–(CH2–CH2–O)n CH2–CH2–OH). These compounds provide a distinctively softer handle.
- (3) Fatty alcohols provide intermediate softness and have the general formula of R-CH2O-(CH2-CH2-O)n CH2-CH2-OH. Non-ionic softeners can be applied on all fibre types, both natural and synthetics.

They can be combined with anionic and cationic products, and for pale shades they can be added directly in the dye bath with no after-soaping or rinsing. Though they can be applied universally, they have less softening effects than anionic and cationic products.

Polyethylene melt can be oxidised by air at high pressure to add hydrophilic groups (e.g. carboxyl). Highly stable emulsions can be prepared in the presence of alkali. These economic softeners are compatible with most of the textile chemicals and provide high lubricity stable in extreme pH conditions and heat, but not durable in dry cleaning. Non-ionic softeners have no electric charge and have no significant affinity for textile fibres. Normally, they are used for forced application methods; namely, padders, spray and foaming applications. Occasionally, they are used by exhaust processes in yarn dyeing machines, winches, jiggers and jets. They can be combined with other non-ionic and ionic active products carrying any electric charge. They are stable at high temperature and almost non-yellowing. They are especially suitable for optically brightened white goods. In comparison to anionic and cationic softeners, as a group they show the best resistance to heavy metal and alkaline earth salts. Pseudo-quaternary compounds are not purely non-ionic, but they do behave analogously and provide the best compromises concerning softness and bath stability/compatibility.

There are a great many non-ionic compositions described in the literature for softening textile materials. These non-ionic textile softening compositions possess many advantages, but many also suffer from several disadvantages. Many of the non-ionic softeners are waxy or gummy in nature, making them difficult to weigh and measure or to mix and disperse with other textile agents, as well as to place in a form, such as an aqueous dispersion, which may be readily handled and applied to textiles. Many softener compositions are diluted with water to form products of 10–30% activity, resulting in viscous liquids or pastes that are difficult to pour or

pump and which cannot easily be diluted further in cold water. Many of the non-ionic softeners are readily capable of imparting softness to textile material treated therewith, but they suffer from the disadvantage that textile materials treated with softeners tends to yellow on ageing or when exposed to elevated temperatures. Many of the textile compositions do not suffer from the aforementioned disadvantages but they do suffer from another disadvantage, such as being too costly, so that they are not suitable for use in the industry. For these reasons, there is a need in the industry for a non-ionic textile softening composition which is economical, imparts improved softness to textile material treated there with, does provide improved resistance to yellowing upon ageing and heating to elevated temperatures and is readily dispersible in water.

It has now been discovered that this need may be met by an improved non-ionic textile treating composition comprised:

- (1) About 70–80% by weight of a blend of (i) a mixture of linear and branched C18–C32 aliphatic, monohydric alcohols and (ii) a mixture of C24–C40 aliphatic hydrocarbons, said blend of alcohols and hydrocarbons having a melting point between about 43°C and 50°C;
- (2) About 15–25% by weight of the etherification reaction product of about 30–50 mols of EO with an amount of blend (1) sufficient to furnish 1 mol of hydroxyl groups;
- (3) About 3–10% by weight of poly-oxyethylene ether of oxodecyl alcohol, wherein the mole ratio of oxyethylene groups to oxodecyl alcohol groups is from about 5–10. The non-ionic textile treating compositions of this invention may be converted to fluid, pourable, easily handled dispersions of 10–30% dispersed solids that dilute readily in cold water for application to textile materials.

#### 7.3.5 Reactive Softeners

The softeners described above are merely deposited on the materials (i.e. they are washed fast). Reactive softening agents can partially react with cellulose on account of their chemical structure and thus give permanent effects. The most common reactive softening agents are:

- (1) N-methylol compounds of higher fatty acid amides (e.g. N-methylol stearic amide)
- (2) N-methylol compounds of urea substituted with higher fatty acid (e.g. octa-decylethylene urea). These are popularly used in resin finishing and have a water repellent as well as softening effect. The characteristics of various classes of softeners are listed in Table 7.1.

#### 7.4 Silicone softeners

Silicones were classified as a separate class of man-made polymers derived from silicon metal in 1904. They have been used to formulate textile softening chemicals since the 1960s. Initially, unmodified poly-dimethyl-siloxanes were used. In the late 1970s, the introduction of aminofunctional poly-dimethyl-siloxanes opened new dimensions of textile softening. The term 'silicone' refers to artificial polymer based on a framework of alternating silicon and oxygen (siloxane bonds). The larger atomic radius of silicon atom makes the silicon–silicon single bond much less energetic, hence silanes (SinH2n+1) are much less stable than alkenes. However, silicon–oxygen bonds are more energetic (about 22 Kcal/mol) than carbon–oxygen bonds. Silicone also derives from its kitone-like structure (silico–ketone) similar to acetone. Silicones are free of double bonds in their backbones and are not oxo-compounds. Generally, the silicone treatment of textiles consists of silicone polymer (mainly poly-dimethyl-siloxanes) emulsions but not with the silane monomers, which may liberate hazardous chemicals (e.g. hydrochloric acid) during treatment.

Silicones exhibit some unique properties including thermal oxidative stability, low temperature flowability, low viscosity change against temperature, high compressibility, low surface tension, hydrophobicity, good electric properties and low fire hazard because of their inorganic—organic structure and the flexibility of the silicone bonds. One of the key features of silicone materials is their effectiveness at very low concentrations. Very small amounts of silicones are required to achieve the desired properties, which can improve the cost of textile operations and ensure a minimum environmental impact.

The mechanism of softening by silicone treatment is due to a flexible film formation. The reduced energy required for a bond rotation makes the siloxane backbone more flexible. The deposition of flexible film reduces inter-fibre and inter-yarn friction. Thus the silicone finishing of textile produces an exceptional soft handle combined with other properties such as:

- (1) Smoothness
- (2) Greasy feel
- (3) Excellent body
- (4) Improved crease resistance
- (5) Improved tear strength

- (6) Improved sew-ability
- (7) Good antistatic and anti-pilling properties

Because of their inorganic—organic structure and the flexibility of the siloxane bonds, silicones have the following unique properties:

- (1) Thermal/oxidative stability
- (2) Low-temperature flowability
- (3) Low change of viscosity with temperature
- (4) High compressibility
- (5) Low surface tension (spread-ability)
- (6) Low fire hazard

Silicones have very wide application in textile processing, such as fibre lubricants in spinning, high-speed sewing machinery, winding and slashing, as binders in nonwoven manufacturing, as antifoam in dyeing, as softeners in print paste, finishing and coating. Rochow in the USA and Müller in Germany independently discovered the direct synthesis of methylchloro-silanes via the reaction of metallic silicon with methyl-chloride. The hydrolysis of dimethyl-di-chlorosilane leads to polymers with a 'Si-O-Si-O' backbone and methyl side groups. The silicone is treated with methyl chloride in the presence of a catalyst, which forms chloro-silanes. The chloro-silanes are a mixture of about 60-70% dimethyl-di-chloro-silane, 20-30% methyl-trichloro-silane, 6-7% tri-methyl chloro-silane, 2-3% methyl hydrogen di-chloro-silane and 0.5% unstable chloro-ilane. This mixture on condensation with water gives a monomer, silanol which polymerises into poly-siloxanes (silicones) (Fig. 7.5). This polymer may be linear, branched, cyclic and elastomeric form. The demand for shear stability is constantly increasing due to the development of machinery for shorter liquor ratios, higher production rates and stronger pumps. The micro-emulsions are mostly preferred due to their higher shear stability, but they can only be obtained from amino-silicones. Due to the introduction of hydrophilic amino groups in a silicone basic chain, the products are easily emulsifible and such emulsions are thermodynamically stable. Their spontaneous formation and optical transparency differentiates them from macro-emulsions. Electron microscope studies show no separate droplets, but rather micelles with no sharp boundary.

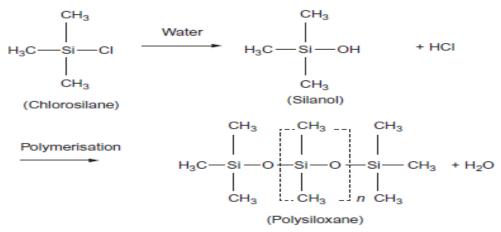


Fig. 7.5 Conversion of chlorosilane to silanol and polysiloxanes.

The water-clear transparency indicates that there are no separate particles with sharp boundaries. Micro-emulsions are generally easier to make, but with an increased addition, the softening increases to a limited extent. As more is added, the resulting higher amount of the emulsifier may adversely affect the handle. The ideal softener should be molecularly dispersible, but still cationic for exhaustion. One possible solution is to use amino-glycol-silicones, although they provide inferior softness. The recent development is a nano-form of silicone in which the particle size is less than 10 nm. Their improved penetrability into textile structures produces a favourable yet unusually soft handle and other properties. The advantages of nano silicone emulsion are:

- (1) Gives an inner softness with a unique cool, natural and dry handle
- (2) Provides hydrophilic properties
- (3) Absolutely stable emulsion in a wide range of pH range and shear forces
- (4) Less negative effect on seam-slippage than traditional silicone emulsions
- (5) Because of hydrophilic properties, oily dirts cannot develop a good adhesion to the silicone film. Hence the soil release property is much better compared to traditional silicones
- (6) Suitable for both white and coloured goods
- (7) Suitable for all kinds of fibres

Saraf and Alat assessed the performances of various types of commercial softeners. It was observed that the micro-silicone emulsion gave a very good handle as it imparts surface smoothness. The whiteness was not affected, but hydrophobicity did increase.

A study by Chattopadhyay and Vyas showed that the nano-emulsion softener improves the feel, softness and crease recovery to a higher extent when compared to the conventional emulsion softener. However, the nano form of emulsion causes a higher loss in strength with increased elongation-at-break. The water absorbency of nano-emulsion-treated fabric is found to be poorer than that of the conventional emulsion.

In a study by Roy Choudhury et al. (2012), cotton fabric was treated with macro, micro and nano silicone softeners, each at three levels of concentration separately. The treated fabric samples were tested for hydrophilicity and various physical properties to evaluate the performance of the softeners. By using ANOVA, it was established that there were significant changes in the degree of hydrophilicity, crease recovery, stiffness, bending modulus and drape as well as tearing strength. All the softeners rendered the fabric hydrophobic to some extent. This effect was marginal for nano softener but significant for macro and micro softeners. For both macro- and micro-finished samples, the trends are similar in that hydrophobicity increased significantly even at the lowest concentration, while for the nano-finished sample, hydrophobicity increased with concentration at a very low rate. All the softeners improved the crease recovery property of the fabric in varying degrees; the improvement was highest in the case of the macro-softener, intermediate for the micro and lowest for nano-softener. For all three softeners, the crease recovery angle increased along with the concentration. The untreated raw sample was the stiffest of all the specimens. This was followed by macro, nano and microsoftener-treated samples, respectively. It is indicated that macro-softeners mainly deposit on the fabric surface, while nano-softeners penetrate into the core structure. The micro-softeners have some sort of combined effect and probably strike a balance to render the lowest bending length. For all softeners, the bending length decreased linearly in both warp and weft directions, with an increase in the softener concentration indicating increasing flexibility. The same trend was observed with drape coefficients. In general, a softener application reduced the drape coefficient. With an increase in concentration, the drape coefficient decreased linearly. The application of the softeners improved the tearing strength of the fabric. This improvement was highest for micro-softener and least for the nano-softener. The softeners greatly increased the abrasion resistance. The average strength loss due to abrasion cycles was highest in the case of macro-softener-treated samples and lowest for nano-softener-treated samples. The macrosoftener-treated samples were the least soft and the nano-softener-treated samples were the softest but in general, softness increased with the increase in the concentration. The macrosoftener-treated samples had the least durability to the laundering and micro-softeners had a

medium level. The best performance in durability was obtained with nano-softeners, indicating that the finishing with a macro is a surface phenomenon while that with a nano is an intrinsic one in the fibres. As the nature of the chemical constitution of the three softeners is not identical, some of the observed differences in properties could also be due to chemical as well as physical effects. The SEM photographs confirm that the macro-softener mostly deposits on the fibre surface, while the nano-softener penetrates inside the fibre and the micro-softener takes up an intermediate status.

# 7.4.1 Organo-Modified Silicones

Dimethyl-siloxane homo-polymers show a very poor softening and hardly reduce fibre wettability. This is due to poor interaction with the fibre and consequent uneven distribution on the surface.

A by-product of the hydrolytic reaction is methyl hydrogen silane. The direct Si–H bond can be used to add  $C \square C$  unsaturated molecules in a reaction called hydro-sililation (Fig. 7.6). The reaction, normally catalysed by platinum or rhodium complexes, enables the introduction of organic side chains that may contain functional groups. The organo-functional silane thus obtained can then be introduced into an existing chain of dimethyl-polysiloxane by the socalled equilibration reaction. An equilibration step exploits both the ease with which Si-O-Si linkages are cleaved in the presence of catalysts and the rapidity with which the new Si-O-Si bonds form. Chain cleavage occurs concurrently with poly-condensation and polymerisation. Thus inhomogeneous mixtures of siloxanes of various chain lengths are converted to polymers whose chain lengths are uniform and follow a Gaussian distribution. The final product may have reactive group like -OH (called a reactive softener) or may have nonreactive methyl or other group (called nonreactive or stopped silicone). The reactive softeners can be applied along with crease-resistant (durable press or wash and wear) finishes when a high degree of durability is obtained. A reaction between the cross-linking agents and the reactive end groups or amino side chains of silicone is likely. The treated fabric surface is significantly more hydrophobic than non-cross-linked silicones with similar structures. Reactive softeners are preferred when normal smoothness and resilience, but not a 'super soft' handle, are required. Methyl oils or poly-dimethyl-siloxanes are the group of silicones first used for the softening of textile materials. The advantages are a glossy surface and the possibility of cross-linking a Si-OH or Si-OR terminating group on the fabric to obtain an interpenetrating network. These are

called 'lubes' and are still used for lubricating of sewing threads. Silicones are a special class of softeners that provide the following:



Fig. 7.6 Hydrosililation.

- (1) Very high softness
- (2) Unique hand
- (3) High lubricity
- (4) Good sew-ability
- (5) High elastic resilience
- (6) Improved crease recovery
- (7) Increased abrasion resistance and tear strength
- (8) Good temperature stability
- (9) A high degree of permanence for products capable of forming cross-links
- (10) A range of properties from hydrophobic to hydrophilic

Besides softeners, they are used as: wetting agents; water repellents; lubricants in spinning, winding and sewing; defoamers and as agents in elastomeric finishes and coatings.

#### 7.4.2 Amino-silicones

Amino oils or amino-silicones clearly dominate the current softeners market due to their elegant handle. The most popular members of this class of softeners are amino-propyl (Fig. 7.7A) and amino-ethylamino-propyl silicones (Fig. 7.7B). More than 90% of all commercially available amino-silicone softeners are amino-ethyl-amino-propyl silicones.

$$CH_3$$
  $CH_3$   $CH_3$   $CH_3$   $CH_3$   $CH_3$   $CH_3$   $CH_3$   $CH_3$   $CH_4$   $CH_5$   $CH_5$   $CH_5$   $CH_5$   $CH_6$   $CH_6$ 

Fig. 7.7 Aminosilicones—(A) aminopropyl and (B) aminoethylaminopropyl silicones.

The nitrogen content of common amino-silicones is below 1%. An ideal distribution of side chains is superior to block structure. The amino-polysiloxane is prepared by hydrolysing and condensing an amino-functional dialkoxy-silane with water in the presence of heat and a base catalyst. The amino-polysiloxane is hydrophobic and has a molecular weight of at least 30,000. The softener properties of amino-silicones can be altered in a number of ways by changing the following:

- (1) The functional groups
- (2) The molecular weight, mainly determined by X
- (3) The amount of substituted groups, Y (amine number)
- (4) The distribution of the substituted groups in the chain
- (5) The chain end X, which can be made reactive/cross-linkable (R = OH or OR) or terminating. The amount of substituted groups determines substantivity, hydrophobicity and the disposition to yellowing. The molecules with reactive chain ends tend to crosslink on the fabric to give elastomeric effects that increase fabric smoothness and hydrophobicity. Normally, it takes place very slowly, but with the use of suitable cross linkers, a network can be achieved in a stenter. Amino-ethyl-amino-propyl and cyclo-hexyl-amino derivatives give a basically similar softness, but the former show slightly better performance. The latter imparts less hydrophobic because of weaker interactions with the fibre

The former is superior to any other known functional silicone containing amino or its derivative (e.g. amido) groups in terms of imparting softness, most probably because the pair of amino groups locates in close vicinity. Silicones with primary amino groups or amine derivatives provide more softness than secondary amino groups, as the latter are not very effective in increasing attractive interactions.

Amino-silicones are prone to thermo-yellowing; the extent of colour changes depends on the type of the amino side chains, amino content, curing temperature and time. The yellowing can be minimised by alkylation, acylation or by trapping the N-atom in a ring structure. Cyclohexyl-amino-silicones exhibited excellent overall performance (i.e. good softening and whiteness together with reasonable wettability).

When non-yellowing silicone softeners are needed, amino fluids with low amine number are generally used. Silicones containing amide group (-NH-(C=O)-R) instead of amine groups have been developed which are non-yellowing and provide a dry handle as compared to low-amine amino-silicones.

It was generally believed that silicone softeners boost the yellowing of blue jeans caused by polluted air. But recently it was proved that ethoxylated emulsifier (used for preparation of silicone emulsion) and not amino-silicones is responsible for such yellowing. The use of fatty acid poly-glycosides emulsifier solves the problem.

The chemical structure versus activity relationship between the arrangement of pendant aminofunctional side chains and important finish responses such as whiteness, hand, water absorbency and soil release was explored by Lautenschlager et al.

Dramatic effects were observed which suggest that the optimisation of the structure of an amino-functional side chain could lead to a substantial finish Improvement.

# 7.4.3 Epoxy-silicones

In the similar way, epoxy modified, cationic silicones can be prepared having structures similar to Fig. 7A, with R = R/-CH (CH2) O and R = (CH2)3OCH2CH (OH) CH2N+ (CH3)3Cl-, respectively. Epoxy-silicones, due to polar interactions between polymer side chains and cellulose, are much better softeners than dimethyl-siloxanes and give measurable hydrophobic effects. They have strong interactions with the fibre, giving it a durable finish. Amino-silicones are superior to epoxy-silicones, most probably due to better orientation and better distribution on the surface due to strong dipole–dipole hydrogen bonding and electrostatic interactions with the fibres, especially with cellulose. The basically continuous encapsulation of the treated fibre results in low fibre friction and more hydrophobic surface.

# 7.4.4 Carboxy-silicones

These have structure similar to 7(a) with R = COOR. These types impart a silky finish, along with a bounciness and bulkiness to the fibre. The emulsions are non-yellowing and are generally applied to nylons and polyesters.

# 7.4.5 Hydroxy-silicones

They have structures similar to Fig. 7.7A, where R = OH. They impart an elastomeric feel to the fibre.

# 7.4.5.1 Acrylic-modified silicones

Acrylic-modified silicones (R = CH=CH2 in Fig. 7.7A) impart a durable finish with a smooth handle and drape. The properties of different functionally modified silicones are compared in Table 7.2.

Table 7.2 Properties of functionally modified silicones

		Functional group						
Properties	Amino	Ероху	Carboxy	Hydroxyl	Acrylic			
Internal softness	Excellent	Poor	Poor	Poor	Good			
Surface smoothness	Fair	Good	Good	Very good	Good			
Hydrophilic	Fair	Poor	Fair	Poor	Poor			
Water repellency	Fair	Very good	Fair	Good	Good			
Durability	Excellent	Very good	Fair	Excellent	Excellent			
Nonyellowing	Fair	Excellent	Very good	Excellent	Good			

A number of other silicone structures have been patented for commercial use (US Patents, 1989–1996). A few are shown in Fig. 7.8A–D.

# 7.4.6 Hydrophilic silicones

Poor wettability is highly undesirable for various textile materials like towels, underwear, sportswear, etc. Most of the highly effective softeners, including silicones (especially all conventional amino-silicones), impart a considerable hydrophobicity to textiles. In the case of silicones, the stronger the interaction between the functional (anchor) groups of the silicone

and the fibre surface and the greater the frequency of the anchor groups in the polymer chain, the better the coverage and hydrophobicity of the fibre surface. On the other hand, a uniform fibre coverage favours a soft handle so long as lubrication is ensured by the flexible polymer segments. The wettability of silicone-treated goods can be improved in a number of ways. The first generation of hydrophilic silicones, available commercially for decades, contains polyether, namely, poly-ethylene-oxide (PEO) and/or poly-propylene-oxide segments as a side chain or as part of the main polymer backbone (e.g. silicone wax, Fig. 7.9A), amino-polyether siloxane (Fig. 7.9A) and polyether siloxane (Fig. 7.9A). These silicones with poly-glycol groups show excellent hydrophilicity and act almost as wetting agents. The tendency of yellowing is lower and almost zero for some structures. However, the bulky PEO groups disturb the ideal orientation on the fibre, resulting in inferior softness as compared to classical amino-silicones. Modified amino-silicones containing hydrophilic moiety are an ideal softening additive in fluorocarbon-based soil-releasing agents. An invention relates to a textile treatment composition comprised a combination of an epoxy-glycol-siloxane and an amino-functional compound.

Fig. 7.8 Acryl-modified silicones.

The amine copolymer formed from the combination imparts hydrophilicity and good hand to 100% cotton textiles. The composition is durable to shelf ageing. The composition comprises the following ingredients:

- (a) An epoxy-, glycol- or siloxane ingredient
- (b) An amino-functional compound selected from the group consisting of amino-silanes and silicone quaternary ammonium compounds (optional)
- (c) A surfactant (optional)
- (d) An acid (optional)
- (e) A carrier

Ingredient (a) has the general formula:

$$E_{3}SiO - \left(R^{1}_{2}SiO_{2/2}\right)_{k} - \left(R^{1}R^{2}SiO_{2/2}\right)_{j} - \left(R^{1}R^{3}SiO_{2/2}\right)_{k} - SiE_{3},$$

wherein h is 25–1000, j is 1–500, k is 1–500 and each R1 is independently a monovalent hydrocarbon group. R1 can be an alkyl group, such as methyl, ethyl, n-propyl, isopropyl and t-butyl, or an aromatic group such as phenyl, tolyl and xylyl. Preferably, R1 is methyl.

Each E is independently selected from the group consisting of R1, an epoxy-functional group, a poly-oxy-alkylene group and combinations thereof. Each R2 is independently a poly-oxy-alkylene group having the formula:

$$-C_m H_{2m} (OC_2 H_4)_x - (OC_3 H_6)_y - OR_4$$

wherein m is an integer with a value of 2–8, x is an integer with a value of 0–200, y is an integer with a value of 1–200, each R4 is independently selected from the group consisting of hydrogen, acyl groups of 1–8 carbon atoms and R1.

By incorporating quaternary ammonium groups into silicones, softness, hydrophilicity, exhaustion properties and bath stability against electrolytes can be improved. However, some quaternary containing silicones showed high toxicity. The problem now can be avoided by avoiding structures not containing quaternary groups at distance close to the critical range. Silicone softeners with quaternised ammonium end groups as shown in Fig. 7.10 exhibit better soil-release and antistatic performances than classical amino-silicones.

Fig. 7.9 Hydrophilic polyether softeners (A) silicone wax, (B) polyether siloxane and (C) aminopolyether siloxane.

$$\begin{array}{c|cccc} CH_3 & CH_3 & CH_3 & CH_3 \\ | & | & | & | & | \\ X-N^+-Z-[Si-O]_xSi-Z-N^+-X \\ | & | & | & | \\ CH_3 & CH_3 & CH_3 & CH_3 \\ Z=CH_2CH(OH)CH_2O(CH_2)_3 \\ X=(CH_2)_yCH_3 \end{array}$$

Fig. 7.10 Silicone with quaternised ammonium end-group.

However, they have poor wash fastness and the handle they impart is described as unsilicone-like. One or two numbers of quaternary groups per molecule show poor wash durability. The presence of multiple groups (polyquat structure) improves wash durability. The levelness of such compounds is strongly dependant on silicone structure. Any common chemical modification (e.g. alkylation or acylation) of an amino-ethylamino-propyl group aiming to reduce yellowing actually improves hydrophilicity, probably because of the weakening silicone—fibre bonds. Cotton-knitted goods, meant for sportswear, casual wear and children's wear, were traditionally finished by cationic softeners by the exhaust method and are now finished in open width by the padding method. This opened up avenues for silicone and other softeners, which because of their low or no affinity and poor shear stability could not be applied on soft-flow machines. Though a poly-dimethyl siloxane (PDMS) based softener provides a better feel and higher fastness to home laundering as compared to conventional cationic

softeners, but those are also hydrophobic, like cationic softeners. Another problem is the dulling of the shade or yellowing of whites during or after finishing due to softeners. In a study by Saraf and Alat, the performances of various types of commercial softeners are assessed as under:

- (1) Though a conventional cationic softener provides the softest handle among organic softeners, the reduction of whiteness and hydrophobicity of the finished fabric are maximum.
- (2) A weakly cationic softener performed better than conventional cationic, but the whiteness is still lower.
- (3) A pseudo-cationic softener retains whiteness better, and the finished product is somewhat hydrophilic.
- (4) A reactive polyurethane gave the best hydrophilicity and unaltered whiteness, but the feel is not very soft.
- (5) Micro-silicone emulsion gave a very good handle as it imparts surface smoothness. The whiteness is not affected, but the hydrophobicity increases.

# 7.4.7 Application Methods

Pre-condensated poly-siloxanes (as solution in organic solvents or as aqueous dispersions) are normally applied to the fabric together with catalysts. After drying on the stenter, the actual condensation takes place with temperature (150-160°C, 0.5-3 min). Zinc, tin, lead, magnesium, and zirconium compounds are all suitable as catalyst for condensation. Ceraperm MN Liq. is macro-silicone emulsion for the permanent finishing of cellulosics, synthetics and blends. It is non-ionic in nature, hence it is compatible with all finishing chemicals and OBAs. It imparts exceptionally smooth surface softness with wash resistant effect, fast to dry cleaning. It is mainly applied by the padding method (dry or wet on wet) and also in garment form. It is mostly applied (10–30 g/L) by padding with resin (Finish NEC Plus) followed by a shock cure at 180-190°C for 45 s Ceraperm 3P Liq. (Clariant) is a colourless, highly concentrated amino micro-silicone. It is weakly cationic in nature. It can be applied by both the exhaust and padding method. In the exhaust method, 1-2% (owf) softener is applied on drum washers and winches. In the padding method, 10-20 g/L Ceraperm 3P is applied at pH 5.5-6. After padding, it is shock cured at 170°C for 45 s. Sandoperm SE 1 oil liq. (Clariant) is a nano-silicone emulsion that is slightly cationic in nature. 10 g/L Sandoperm SE1 oil liquid is padded (pickup 80%) along with 0.35% acetic acid (60%) and dried at 140°C. A study was made to find the best pH for application of amino-silicones by the exhaust method. It had been observed that the maximum exhaustion and softness of amino-silicones occur at pH 5–5.5, as in this pH range the amino groups are ionised to the maximum extent. Often silicone-treated fabrics shows moist appearance during storage. The problem has been studied thoroughly by the author and the problem is probably due to insufficient curing as discussed below.

Most of the silicone emulsions are mixtures of unreactive polymer poly-dimethyl-siloxane and reactive polymer poly-methyl-hydrogen siloxane. On curing, the latter is hydrolysed and condensed. The curing mechanism of the poly-methyl-siloxane is a base-catalysed hydrolysis of ≡Si−H to ≡Si−OH followed by silanol condensation. Water has been found to be a reactant, and the metal compounds of tin and zinc functions as catalyst. Even in apparently water-free systems, sufficient moisture is present in the textile fibres to permit the necessary hydrolysis reaction to take place. Silanol groups formed in the initial rapid hydrolysis stage react with excess silane groups or condense with themselves, giving rise to water as follows:

$$\equiv SiH \rightarrow \equiv SiOH$$
 (7.1)

$$\equiv SiOH + \equiv SiOH \rightarrow \equiv Si - O - Si \equiv +H_2O$$
 (7.2)

As curing is done at a temperature as high as 150°C, water formed after complete curing will vaporize. However, if the curing is insufficient, the reaction continues slowly during storage. But due to low temperature during storage, the water molecules cannot vaporize and make the fabric moist.

#### 7.4.8 Silicone on Wool

Silicone-based softening agents are normally associated with enhancements in fabric softness. A silicone-based softener, Megasoft Jet (Ciba), was applied to woollen fabrics by a pad-dry (3 min at 150°C) method. The surface energy of the softeners is lower than the wool fibre. Since they have a high tendency to spread on the fibre during drying, there is a strong tendency for softeners to form continuous layers between adjacent fibres. When silicone polymers are applied to the fabric, the formation of an elastic network on the surface of the fibre makes the fabric easier to bend and twist. Therefore the net effect of the softening agent on wool fibres is an initial decrease in fabric stiffness and improvement in the handle of the treated fabrics. The results of a subjective evaluation of the effects of fabric softness and harshness were consistent with the bending stiffness evaluation. The mechanism for the reduction in prickliness of

silicone-treated fabrics appears to be reduced fibre-fibre friction rather than a modification in the fibre modulus. The wool comfort metre (WCM) device was developed to objectively assess the comfort properties of knitted fabric. There is a good correlation with the results of a subjective assessment in the wearer trials of fabric prickle responses and this objective technique. Since the instrument has been designed to detect changes in wool fibre-bending stiffness, it is expected to be able to detect the changes in comfort on both softener- and plasmatreated fabrics.

While the WCM was able to assess the changes affected by silicone on treated fabrics, the differences between plasma-treated and untreated fabrics were not significant. It is likely that the modification in the fibre modulus is not the only mechanism involved. It is believed that glueing the protruding fibres together by a silicone polymer is the reason for the reduction in the WCM value of silicone-treated fabrics compared with untreated and plasma-treated fabrics, as fewer individual fibres can be detected by WCM.

#### 7.5 Mechanism of Action

The softening effect is mostly effective on the textile surface. In addition, small softener molecules may penetrate into the fibres and provide an internal plasticisation of the polymers by reducing the glass transition temperature. The physical arrangement of the softener molecules on the textile surfaces depends on their ionic nature and the relative hydrophobicity of the fibre surface. The electrostatic attraction is the driving force, but hydrophobic interactions also play a significant role. Cationic softener molecules orient themselves with positively charged ends towards negatively charged fibres (zeta potential) in the water, while hydrophobic carbon chains project outwards, providing excellent softening and lubricity. If the deposition is solely due to the electrostatic interaction, it should be stoichiometrically related to the number of functional groups of cellulose molecule. In the actual case, deposition does not level off until a large excess of cationic softener is applied. The model cannot explain why a cationic softener deposits onto a charge-fee synthetics or why non-ionic species may deliver softness. It can be assumed therefore that the driving force for the deposition of cationic softener molecules onto cellulose molecules is not the electrostatic attraction by the fibre, but it is the hydrophobic ejection from water. Due to their lack of affinity for water, cationic molecules leave an aqueous medium and deposit on the available surface. Once on the fibre surface, the cationic charge mainly helps dispersing rather than anchoring. Once on the fibre, the cationic molecules interact with cotton through van der Waals interactions and by electrostatic attraction if the fibre is negatively charged. The hydrophobic interaction stated

above is different from hydrophobic bonding. When hydrocarbons are dispersed in an aqueous phase, water molecules in their vicinity organise into a quasi-crystalline structure, improving hydrogen bonding capacity with a consequent reduction of entropy. The phenomenon is known as 'hydrophobic hydration'. When hydrocarbon chains are removed from the aqueous environment by associating or depositing onto fibre surfaces, these water molecules go back into the bulk. The entropy thus gained by the system provides the driving force of the adsorption process and consequently is favoured by a temperature rise.

In case of anionic softeners, the negatively charged ends are repelled by the negatively charged fibre surfaces, causing higher hydrophilicity and less softening than cationic softeners. The orientation of non-ionic softener molecules depends on the nature of fibre surface; that is, the hydrophilic surfaces attract hydrophilic portions while the hydrophobic surfaces attract hydrophobic portions of the softener molecules. Silicones are in a position to cross-link with cellulose, whereby the ether groups align themselves according to the primary hydroxyl groups of cellulose (Fig. 7.11).

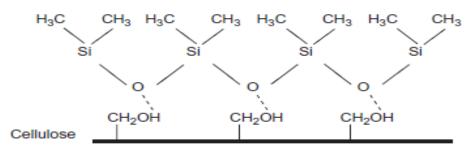


Fig. 7.11 Interaction of silicone polymer with cellulose.

Silicones deposit on the fibre surface in a more or less formed film, so that no gaps which can destroy the properties of the silicone film arise. The fabrics being impregnated need to be thoroughly pre-cleaned.

According to Bereck, the high molecular flexibility of the silicone chain is the reason for their low glass transition temperature (about -100°C) and their special softness. According to them, the methyl groups of the OSi(CH3)2. Structure shield the oxygen atoms from outside contact. Therefore the surface of the fibres finished with poly-dimethyl-siloxane is mostly nonpolar and hydrophobic. Strong hydrogen bonds exists between the amino groups of modified silicones and hydroxyl groups of cellulose or amino groups of wool, silk and polyamide fibres. The bonds act as an anchor for the silicone, which forms an evenly distributed film on the fibre surface, resulting in a good water repellency and a very soft hand. With optimum content of side groups, the poly-siloxane segments between the anchor sites are long enough to maintain

their high flexibility. This explains the high softness and lubricating effects of amino-silicones on polar fibres.

In the case of relatively nonpolar fibres like polyester, the hydrophobic segments of silicone interact strongly with the hydrophobic fibre surface. The positively charged amino side groups repel each other and enhance the flexibility of the silicone chain loops. This is the reason behind the especially soft hand of the amino-silicone treated on nonpolar fibres. Quaternary modified groups provide high alkaline and shear stability. The efficacy of a silicone product is primarily dependent on the distribution and orientation of the polymer on the fibre surface, which is determined by the nature and magnitude of polymer–fibre interaction.

In addition to the nature of functional group, the amine content, viscosity or chain length and particle size may influence softening and wettability. The optimum number of amino group is somewhat higher for polyester and significantly lower for polyester/cotton as compared to that for pure cotton. Due to high hydrophobic nature of polyester, the softening is much less dependent on the amino content than in case of cotton. With an increase in viscosity or chain length, the treated fabrics become increasing hydrophobic and may impart a better handle. Reactive silicones may give a smoother and much more hydrophobic finish compared to a methyl end group containing nonreactive varieties, depending on the chain length and curing conditions. The treated fabrics become increasingly softer and smoother as well as more hydrophobic during storage, apparently due to chain extension on the surface.

In polyester/cotton blends (65:35) most, if not all, of the amino-silicone is located on the cotton surface, and the polyester component is practically free of silicone. Fluorescent microscopy and Energy Dispersive X-ray analysis showed that amino-silicones diffuse into cotton, but not into polyester. As silicones greatly decrease fibre–fibre friction, they can also improve abrasion resistance to a great extent due to the increased mobility of the fibres. They can absorb and dissipate mechanical energy and they can bend easily unlike stiff fabrics with high fibre friction. For the same reason, they tend to increase pilling formation.

## 7.6 Additives

Nowadays, a modern softening agent should be multifunctional. Products only based on fatty acid amine condensates are being found less today in practise. Besides the aspect of the soft handle, multifunctional softeners today should have other finishing aims such as hydrophilicity, sew-ability, anti-static, surface smoothening, shearing stability, etc. Multifunctional softeners contain further additives besides the classical fatty acid amine

condensates. The properties can be expanded to many fields by choosing the right formulation. A softener for application on the jet dyeing machine must be drawn up in such a way that it can stand the high pump and nozzle-shearing forces. But more than that, such a product must also possess an extremely high emulsion stability. A suitable de-foaming system will guarantee a low foam formation during usage. The foam which is formed should be decomposed quickly, and additives which reduce the surface tension of the water can be helpful. The product must continue to have a good affinity for the textile, which is to be finished so that total bath exhaustion and a good effect can be achieved.

Removal of the natural substances in pre-treatment processes, as well as long dyeing period and/or easy-care finishing agents, together with severe drying/condensation conditions lead to sewing damages through fibres becoming brittle and a decrease in firmness, especially with knit goods. High-speed industrial sewing machines allow almost no time for the fibre to move, as it is already very restricted in its mobility and elasticity. In a split second, the mesh which was hit by the needle must expand from its actual diameter to the diameter of the needle. Changed consumer demands lead to new demands towards softener finishing. The market demanded the 'super-soft wave'. Therefore development went towards high-class, multifunctional softeners, which not only contain emulsified fatty acid condensates, but also different silicones and waxes with respective special additives. Such combinations or special products not only allow for distinctly better effects, but also the properties of the softeners can be tailor-made to meet the individual requirement profile. Nowadays, the optimisation of the soft handle is not the only target that one wants to meet by the usage of modern products. Besides softness, smoothness and volume special properties which increase the consumers' benefit play an increasingly important role.

Antistatic, humidity transport, water and soil repellency, odour impeding and antibacterial effects and skin care properties are only a few examples of the additional benefits which modern finishing agents can offer today. Nowadays, these special effects play an increasingly important role not only in clothing textiles, but also in technical textiles.

Textile softeners are usually marketed as aqueous dispersions at 10–50% active matters. In addition to active agents, the commercial softeners may contain emulsifiers, dispersants and defoamers to fulfil the technical needs. Conventional silicone oils are insoluble in water. Commercially, the emulsions are made with fatty alcohol ethoxylates emulsifiers with HLB values between 9 and 15. Due to lower costs and good dispersability, alkyl-phenol-ethoxylates

are also used in non-European countries, but the product may not meet Ökotex100 standard. Some of the additives used for smoothness are:

- (1) Fatty acid esters (C16–C18)
- (2) Paraffins (C16–C18, mp 35–75°C)
- (3) Partially oxidised poly-ethylenes (mp 100–120°C for low density and 120–140°C for high density types)
- (4) Amino-functional silicones

The other few additives for improvement of specific functional properties are:

- (1) Sew-ability, smoothness—polyethylene
- (2) Handle and foaming—silicone
- (3) Handle and volume—silicone, quats
- (4) Antistatic—quats, betaines

Further additives, such as viscosity adjustment, perfumes, preservatives, pH adjusters, etc., are added to broaden their usability. The softening treatments are often combined with easy care and antistatic treatments. As a rule, hydrophobic softeners provide an extra softness, whereas hydrophilic softeners bring about some fullness. In combination with fluorocarbons, most softeners reduce oil repellency. Some ionic, surface active and silicone-free softeners are not compatible with water repellents. As softeners are also excellent fibre lubricants, the finishes often give poor anti-pilling and slippage properties. Synergistic results are obtained when softeners are blended with the following active substances:

- (1) Functional PDMSs/lubricants
- (2) Modified polyurethanes
- (3) Modified poly-acrylates
- (4) Poly-ethylenes

Functional PDMSs or lubricants are blended where functionality of the softeners gives added lustre, extra body and antistatic property. Modified polyurethanes are added as a handle modifier. The fabric resilience is dramatically improved by the formation of an elastic and flexible film. The reactivity of these products is reduced by blocking groups in order to ensure

adequate storage stability. The blocked groups split off at a high temperature in the presence of a special catalyst. The reactive iso-cyanate groups thus released cause the formation of a very fine film on the fibre surface. Modified acrylates reduce the tendency towards pilling, thereby serve as nonslip finishes, as well as binding agents. High-density poly-ethylenes are blended with silicone softeners to improve the sew-ability abrasion property and the tear strength of the fabric. To prevent softeners from breaking due to shearing forces (especially silicone softeners) the following points are to be observed:

- (1) Apply in a weakly acid pH-range (5–6).
- (2) Select the liquor temperature in such a way that the turbidity point of the emulsifier system is not exceeded. However, for synthetic fibres, the bath exhaustion is often achieved through a controlled breakage of the emulsion.
- (3) Only use products which are designated by the producer as stable to shearing powers

#### 7.7 Estimation of Active Matter Content

For various reasons, it is necessary to estimate the active matter content of the softeners. The active matter in cationic softeners may be determined by methylene blue method. A solution (e.g. 10 g/L) of the softener is prepared with distilled water and pH is adjusted to 8 with sodium carbonate. 10 mL of the solution is taken in a 250 mL-stoppered reagent bottle and 25 mL of 0.003% methylene blue added, followed by 0.2 mL sulphuric acid (conc.), 5 g anhydrous sodium sulphate and 15 mL of chloroform. After gentle shaking, the bottle is allowed to stand for sometimes, when the mixture is divided into two layers. The upper layer, which is coloured blue, is then titrated against 0.1% solution of an anionic detergent powder (about 40% strength). At the end point, both the layers have the same intensity of colour. The active content in nonionic softeners (based on EO or polyethylene glycol condensates) may be determined by the phosphor-molybdic acid method or ammonium cobalto-thiocyanate method. In the later method, 1–2 g of the sample is weighed and dissolved in water and made up to 250 mL of this solution, 50 mL is transferred to a separating funnel and treated with 20 mL cobalto-thiocyanate reagent (prepared by dissolving 30 g cobalt nitrate and 200 g ammonium thiocyanate in 1 L water) after neutralising with dilute HCl (0.1 N). The coloured complex thus formed is then extracted with 20 mL dichloromethane, and the intensity of colour is measured at 600 nm in a spectrophoto-meter. The amount of non-ionic matter is then calculated from a previously drawn calibration curve using a standard non-ionic product. The active matter in polyethylene emulsion softeners can be determined by breaking the emulsion and extracting polyethylene

with ether. In this method, a 5 g sample is taken in a 100 mL beaker, heated to 60°C and 15 mL of the 10% alum solution is added, followed by 25 mL water. The contents are boiled until the emulsion breaks completely. It is subsequently cooled, extracted with ether in a separating funnel, and the amount of polyethylene is determined after evaporating ether and drying.

The active matter in silicone emulsion may be determined by the oil content method using common salt, 1:4 di-oxane or by the silica content method. In the oil content method using common salt, 20 mL of silicone emulsion is taken in a clean, dry 100 mL-stoppered measuring cylinder and shaken with 20 mL of 30% common salt for about 2 min. The cylinder is then placed in a boiling water bath for about 30 min after loosening the lid. Due to the salting-out effect, three different layers will emerge; that is, 30% common salt layer (slightly hazy) at the bottom, an emulsifier layer (thick whitish residue) above the water layer and a clear silicone oil forming the topmost layer. The volume of the silicone oil is read and from the density of silicone oil (0.97–0.98 g/mL), the silicone content (by weight) is calculated. In the 1:4 di-oxane method, the determination is carried out at cold by taking a 20 mL silicone emulsion and 20 mL 1:4 di-oxane in a 100 mL cylinder, as discussed above, and kept aside for 1–2 h for a complete separation of silicone oil. The silicone content is calculated from the volume of oil as above.

The determination of the silica content gives the exact purity of the product as compared to the oil content method. The silica content can be determined by the wet washing procedure (Gaines, 2015). In this method, 0.5–1.0% of the silicone emulsion is taken in a 500 mL beaker and treated with 30 mL of concentrated sulphuric acid and 30 mL of concentrated hydrochloric acid (added in portions) on a hot plate inside a fuming cabinet to completely destroy the organic matters in silicone. The acid is evaporated to dryness and the precipitated silica is dispersed in 100–150 mL of water containing 1–2 mL conc. hydrochloric acid and boiled for 10 min, filtered hot using Whatman No. 40 filter paper, washed until free from acid, dried and finally washed in a muffle furnace at 800°C for about 30 min to obtain silica content. 6.8 Measurement of softness. The fabric hand is the estimated quality of a fabric, evaluated as a reaction to the sense of touch, which is integrated in our brain as a total value. Among various components of the sensory perception are the smoothness, compressibility and elasticity of the textile sample. Like colour perception, handle is a subjective feeling, and there have been many efforts to find methods of an objective evaluation. The American Association of Textile Chemists and Colourists has published guidelines for subjective hand evaluation (AATCC Evaluation

Procedure 5, 1990). In the AATCC evaluation method, the following instructions are to be followed:

- (1) The evaluator is to be assisted by a facilitator, who is to provide instructions about the specific elements of hand to be evaluated, the rating scale to be used, the number of samples and specimens rated or compared, the order of presentation, the duration of the rating session, etc.
- (2) The evaluator may or may not see the sample during evaluation.
- (3) The facilitator is to place a specimen on a smooth, non-metallic surface with a correct alignment, and the surface is to be evaluated as uppermost.
- (4) If the thermal element (i.e. warm/cool) is to be evaluated, the evaluator first assesses this by contacting the fabric surface with his or her fingertips.
- (5) While still on the flat surface, the specimen is to be held down with one hand and stroked or touched with the other hand.
- (6) The evaluator is to then touch the specimen by lightly pressing it with the fingers and palm of the hand.
- (7) The evaluator is then to pick up the specimen and rub it between the thumb and fingertips.
- (8) The evaluator is then to squeeze the specimen gently between the thumb, fingers and palm by making a fist.
- (9) If the ease of stretch is to be judged, the specimen is to be held so that there is at least 9.0 cm (3.5 in.) and not more than 25 cm (10 in.) of fabric between the hands. With the elbow close to the body, hands are to be pulled apart, noting the ease of extending the specimen. The specimen is to be stretched lengthwise, width wise and diagonally.
- (10) If resiliency or recovery from squeezing is to be judged, the evaluator must look into the specimen. The specimen is to be clenched tightly in a fist and then released quickly (less than 5 s).
- (11) The specimens can be compared in pairs or sets and judged for the direction and magnitude of the differences in the constituent elements of the hand to be assessed. The specimen may be rated against a reference standard or two extremes may be established for a property of interest, assigning arbitrary numerical values to the extremes. The specimen may be rated as 'most

(rough)', 'least (rough)' or 'moderately (rough)'. Ranking specimen may be difficult with increasing number of samples. The perception scale for the description of change in a constituent element of hand may be as follows: 1 = no, 2 = slight, 3 = moderate and 4 = extreme difference perceived.

- (12) The evaluation should be repeated by the same individual within 5 days.
- (13) As everybody has his or her own dominant hand, all assessments should be made with the same hand. The physical attributes for constituent elements of hand are as follows
- (1) Compression—hard, thin, thick, springy, fullness, bulky, firm, soft, lively, lofty, resilient
- (2) Bending—stiff, pliable, supple, crisp, limp, papery, lively, springy, boardy
- (3) Shearing—supple, clinging, tight, loose, firm, pliable, elastic, stretchy
- (4) Surface—coarse, rough, slippery, harsh, smooth, fuzzy, soft, scratchy, slick, waxy, nappy, oily, raspy, warm, cool

In quality control jobs, sometimes a single or a set of reference standards with different hand values is used. The set of standard is produced from the same fabric as the one being evaluated (test sample) by applying the same softener at incremental concentrations. Only the bending property (flex rigidity) can be evaluated by cantilever method.

Flexural rigidity, 
$$G = (1/8) \times W \times L^3$$
 (7.3)

where W is the fabric weight in g cm-2 and L is the average sagged length of sample in cm.

# 7.7.1 Handle-O-Meter

In Handle-O-Meter (ASTM, 2006), the stiffness of fabric is determined by measuring the force required to push a specimen (maximum 20 cm or 8 in.) into a slot of predetermined width (two flat plate supports separated by a fixed distance) made with a metal blade (attached to a motor driven beam) working at a predetermined capacity. The force is applied to the fabric swatch midway between the supports by means of a blade. The required force will depend on the flex rigidity of the sample and the frictional resistance on the corner of the slit. Thus both the surface smoothness and the compressibility are taken into account. The mean values for lengthwise and width wise for both sides of fabric are recorded. In the ring method, a simple method for the evaluation of softness, a piece of fabric is pulled through a circular hole, and the force needed to do so is continuously measured. The measured resistance force consists of

components of the flexural rigidity, the surface friction the shear rigidity and the compressibility. Two parameters, the maximum force measured and the work calculated, are used for evaluation of handle or softness.

#### 7.7.2 KTU-Griff-Tester

Hand is a specific property of textiles, mostly determined subjectively, but the number of investigations done in the last decade suggests that new objective methods for its evaluation are going to appear. Some data show that one of the more perspective mechanical test methods for knitted material, suitable for its hand evaluation, is pulling a disc-shaped specimen through a round hole. During the test, the H–P (path resistance) curve is obtained, and, depending on its parameters, it is proposed to evaluate the magnitude of textile hand. The data already obtained suggest a good means for optimising the parameters of the experimental equipment and testing conditions. A few years ago, the instrumental method KTU-Griff-Tester which could determine the textile hand parameters was introduced. The behaviour of a disc-shaped specimen of knitted fabric, which is pulled through a round hole, was studied by Strazdiené et al. The test was performed by a special pulling device (Fig. 7.12) mountable on a tensile testing device, which consists of two transparent perpendicular plates; a replaceable stand with a hole in the centre the radius of which, r, was 10, 15 or 20 mm, respectively, and a limiting plate with a hole of the same radius, made of acrylic glass. The distance d between the plates was adjustable in the range of 2–15 mm. A spherical punch (3) of radius p = 5 mm and with a needle-type handle was used to pull the specimen through the hole in the stand. Two cases of pulling a disc-shaped specimen (r = 56.5 mm) were investigated. A = free pulling C = restrained pulling simultaneously through the space between limiting plates and through the round hole. In the pulling process, the specimen started to wrinkle and assumed a complicated shape, the geometry of which depended upon the properties and type of fabric tested.

Theoretical investigations of the behaviour of knitted fabrics revealed that the pulling method reported could be considered not only as an instrumental basis for the measurement and prediction of textile hand, but also as a suitable method for the evaluation of anisotropy, drapability, and other specific properties of textile materials. It has been shown that the geometrical behaviour of a specimen can be described by referring to the model where the variation of the outer contour of a disc-shaped specimen is approximated by the sine-curve function. It is shown that pulling a disc-shaped specimen through a hole evidently reveals the non-homogeneity of textile-material properties in different directions. It is also simple in the

sense of its performance, provides new information about material behaviour and can be widely used in studies of the material science of soft materials.

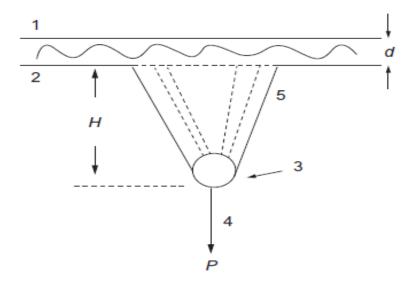


Fig. 7.12 KTU-Griff-Tester (schematic).

The device called KTU-Griff-Tester registers pulling curve (pulling deformation/ pulling force) H–P. The present pulling curve allows obtaining the following four pulling parameters:

- (1) The maximum pulling force (Pmax)
- (2) Tangent of the slope angle of the typical pulling curve H–P initial part (tgα)
- (3) Maximum specimen deformation (Hmax)
- (4) Pulling work (A) being proportional to the area lying below the typical H–P curve Kozeniauskiené et al. studied the efficiency of softening with eight liquid chemical softeners of the textiles manufactured from cotton, bamboo or viscose spun yams. Effectiveness of technological treatment has been analysed via the application of an experimental method based on the pulling of a disc-shaped specimen through the central nozzle of KTU-Griff-Tester device. Analysis of the obtained results has demonstrated that the washing of textiles leads to a decrease in the hand parameters thereof, such as the maximum pulling force (Pmax), tangent of the slope angle of the typical pulling curve H–P initial part (tgα), pulling work (A) and complex hand parameter (Q), as well as in mechanical thickness ratio (M) compared to the ones of the unwashed (control) textiles independent of the fibre content and manufacturing type thereof. Hand properties of the textiles under investigation have been shown to improve after chemical softening compared to the washed textiles, but they have never been comparable to

the ones of the untreated (control) textiles. Some data show that one of the more perspective mechanical test methods for knitted material, suitable for its hand evaluation, is pulling a discshaped specimen through a round hole. During the test, the H-P (path resistance) curve is obtained, and, depending on its parameters, it is proposed to evaluate the magnitude of textile hand. The data already obtained suggest a good means for optimising the parameters of the experimental equipment and testing conditions. Grinevičiūtė et al. conducted an objective and subjective hand evaluation of 25 textile fabrics (woven and knitted). Textile hand was assessed subjectively by a hand-picked judges panel, which in the way of a direct survey assessed the fabrics in respect to 10 properties (smoothness, softness, thickness, roughness, weight, hardness, elasticity, stiffness, warmth and pleasing to handle) suitable to characterise the fabric hand. The textile hand was evaluated objectively using the KTU-Griff-Tester on the basis of five determined parameters. A comparative analysis was made between the results obtained by the judges' survey and the parameters determined experimentally by using statistical methods. The level of the judges' agreement according to each fabric property was evaluated on the basis of Kendall's concordance coefficient. A correlation between the assessment of the separate judge and the assessment of the total judges' group was determined on the basis of Spearman's rank correlation coefficient. Experimental data were estimated similarly as in the case of subjectively obtained results. The obtained dependencies between the subjectively and objectively determined parameters, suitable for the textile hand characterisation, were not linear. They therefore concluded that the correlation between the subjective and objective methods is tenuous and coincidental.

## 7.7.3 Kawabata System

The manual assessment of softness of textile materials is highly subjective and differs from person to person. The physical test methods for the characterisation of fabric softness were reviewed by Ellis and Garnsworthy. Most of the methods were based on tensile, shear and bending properties, as well as compressibility and surface smoothness. Kawabata and Niwa (1975) led to the construction of a sophisticated measurement device, Kawabata Evaluation System-Fabrics (KES-F), which has been extensively studied in research and industry during last two decades. It gives detailed information on 'handle-related' fabric performance by the evaluation of 16 physical parameters. It consists of several different measuring instrument such as tensile and shear properties (KES-F1), bending properties (KES-F2), compress-ability (KES-F3), surface (KES-F4) and thermal (KES-F7). The measured parameters and the area weight are normalised and correlated to the subjective handle scores. From the correlation, for

every hand evaluation a transformation equation is developed, resulting in a primary hand value between 0 and 10. From these primary hand values, a total hand value ranges from 0 (not acceptable) to 5 (excellent). For every fabric type, new correlation equations have to be developed. Niwa and Seto postulated that one KES-F parameter shear hysteresis is particularly suitable for a quick quantification of softness. The silicon polymers are not biodegradable, but they are easily adsorbed on the sludge. Softeners emulsions can, therefore, be eliminated from wastewater at levels up to 80–95%. Softener emulsions tested in biological wastewater treatment do not show any sign of hampering the bacteria. The nitrogen intake caused by amino-silicones is 10 times less than with fatty amines. Silicones are one of the most innovative classes of textile finishing auxiliaries. Due to outstanding performance, tailor-ability and low environmental impact, silicones are expected to have a bright future.

# 7.8 Effect on Sewability

Hurt and Tyler established that the finishing processes applied to the fabrics should modify their frictional properties and thus have an influence on needle penetration and sewing damage (Hurt and Tyler, 1975, 1976). First, the needle penetration forces are critically affected by the use of a lubricant or softener. Second, and most important, fabrics exhibiting higher penetration forces were also the fabrics exhibiting sewing damage. Lomov presented a mathematical model for the needle penetration force in woven fabrics. Mallet and Du used finite element modelling techniques to predict penetration forces in fabrics (Mallet and Du, 1999). The main conclusion that can be drawn from the cost/benefit of the different softener formulation analysis is that the combination of silicone and polyethylene-based softeners used boasted the most interesting cost/performance behaviour. In fact, it produced the lowest penetration forces of all products at a very low price point. None of the remaining cases produced such a good result. This is a most interesting result, and the underlying reasons for it should be further studied. Another important conclusion is that the effect of different softeners on sew-ability varies significantly, with high cost not necessarily reflecting better effectiveness. The use of specific instruments for the quantitative assessment of the associated parameters is thus of high interest to fabric manufacturers.

### 7.9 Effect on Pilling

Pilling is defined as the entangling of fibres on a fabric surface during wearing, washing, dry cleaning or testing to form fibre balls or pills that stand on the surface of the fabric. Pills are of such density that light will not pass through them, and they cast a shadow. Pilling is a dynamic process comprised of two phenomena:

- (1) Fuzzing—the protruding of fibres from the fabric surface
- (2) Pill formation—the persistence of formed neps at the same surface

Pilling of fabrics has always been a great concern, as it spoils fabric surface appearance, initiates fabric attrition and diminishes its serviceability. The problem of pilling became even more serious with the emergence of synthetic fibres, such as polyester, especially if those fibres are present in the form of a blend, with some fibre of lower tensile strength. The fabric type also has an important bearing on its pilling performance. A knitted fabric tends to produce more pills compared with a woven fabric because of its loose structure, which allows easier fibre migration. A tightly woven fabric structure with a greater number of ends per inch and picks per inch has a lower pilling tendency than a loosely woven fabric. An analysis of the conditions giving rise to pilling on polyester/cotton fabrics shows that its formation is always promoted by a number of factors. Fibre characteristics like length and fineness of the component fibres influence the pilling tendency of fabrics to a significant extent. The yarn factors that promote pilling are:

- (1) Low twist
- (2) Hairy and bulky yarns
- (3) Single yarns
- (4) High proportion of polyester components

Wherever possible, weaves with maximum interlacings could be used to an advantage. Long floats expose longer lengths of fibres to the abradant and increase the pilling tendency. Singeing, heat setting and cropping reduce the pilling tendency to a significant extent and are the most effective ways of controlling pills. The type of weave is also an important factor for determining the pilling tendency of woven fabrics. It has been found that twill weaves tend to pill more compared to plain weaves. The choice of softeners is of paramount importance, as the pilling performance of a fabric is one of the major requirements of customers, along with the fabric handle. In this study, it was concluded that certain softeners, which decrease fibre-to-fibre friction by internal lubrication, such as non-ionic organo-modified silicone microemulsions and amino-functional poly-siloxanes, result in a decrease in fabric pilling performance. Furthermore, as far as polyester/viscose-blended fabrics are concerned, sanforising treatment also results in an increase in pilling propensity.

#### 7.10 Future Trends

Softeners are the most important finishing agent. However, it is a real challenge to assess and to reproduce softness, as it is a combination of several psychophysical sensations. The softness requirement is versatile, hence there are a large number of softeners having various ionic natures, namely, anionic, cationic, amphoteric and non-ionic that are in vogue. The demand is ever-increasing, but it is restrained by eco-friendliness and biodegradability. Various derivatives of silicones with versatile properties play an important role in the softening processes; amino-silicones are the most popular as they provide an elegant handle to textiles. However, the finished fabric tends to yellow when exposed to high temperature. The thermoyellowing of amino-silicones strongly depends on the structure of the amino side chains, the amino content and curing temperature and time. The problem gained importance in the mid1980s when the processors enhanced the curing temperature during a crease-resistant finish to minimise residual formaldehyde. To minimise or to avoid yellowing, a number of alternate structures have been evaluated.

# **Chapter 8 Water Repellent Finishes**

#### 8.1 Introduction

Finishes that repel water, oil and dry dirt are important in all parts of the textile market – for clothing, home and technical textiles. Water repellency is achieved using different product groups, but oil repellency is attained only with fluorocarbon polymers. They are modified to have a wide range of properties to fit the different demands of the users and the intended purpose. This is one of the most interesting new developments of chemical finishing.

The oldest repellent finish is to repel water. The purpose of this finish is self-evident. Drops of water should not spread on the surface of the textile and should not wet the fabric. The drops should stay on the surface and easily drip off. Similarly, oil repellent finishes should prevent oily fluids from wetting treated textiles. In a similar manner, soil-repellent finishes should protect textiles from both dry and wet soils. In all cases, the air permeability of the finished fabric should not be significantly reduced.4 Waterproofing treatments will not be covered in depth. A waterproof textile should withstand the hydrostatic pressure exerted by a column of water from at least a 1 m height before the first drops of water penetrate through the fabric. In practice this is mostly achieved with coatings which have the disadvantages of stiff handle, lack of air and vapour permeability and consequently poor wear comfort. In addition to the desired repellency effects, other undesirable fabric properties are often found with repellent finishes. These include problems with static electricity, poor soil removal in aqueous laundering, stiffer fabric hand, greying (soil re-deposition) during aqueous laundering and increased flammability. Some fabric properties that are often improved by repellent finishes include better durable press properties, more rapid drying and ironing, and increased resistance to acids, bases and other chemicals. Table 8.1 shows typical textile applications for repellent fabrics and their requirements.

# 8.2 Mechanisms of Repellency

Repellent finishes achieve their properties by reducing the free energy at fibre surfaces. If the adhesive interactions between a fibre and a drop of liquid placed on the fibre are greater than the internal cohesive interactions within the liquid, the drop will spread. If the adhesive interactions between the fibre and the liquid are less than the internal cohesive interactions within the liquid, the drop will not spread. Surfaces that exhibit low interactions with liquids are referred to as low energy surfaces. Their critical surface energy or surface tension  $\gamma C$  must be lower than the surface tension of the liquid  $\gamma L$  (the internal cohesive interaction) that is

repelled.  $\gamma$ L of water, at 73 mN m-1, is two to three times greater than  $\gamma$ L of oils (20–35 mN m-1). Therefore, oil repellency finishes with fluorocarbons ( $\gamma$ C = 10–20 mN m-1) always achieve water repellency, but fluorine-free products, for example silicones ( $\gamma$ C = 24–30 mN m-1) will not repel oil.7 Low energy surfaces also provide a measure of dry soil repellency by preventing soil particles from strongly adhering to fibre surfaces. This low interaction allows the soil particles to be easily dislodged and removed by mechanical action. There are different ways that low energy surfaces can be applied to textiles. The first way is mechanical incorporation of the water-repellent products in or on the fibre and fabric surface, in the fibre pores and in the spacing between the fibres and the yarns. Examples of these are paraffin emulsions. Another approach is the chemical reaction of the repellent material with the fibre surface. Examples of these are fatty acid resins. Yet another method is the formation of a repellent film on the fibre surface. Examples of these are silicone and fluorocarbon products. The final approach is to use special fabric constructions like stretched polytetrafluoroethylene films (Goretex), films of hydrophilic polyester (Sympatex) and micro-porous coatings (hydrophilic modified polyurethanes).

Table 8.1 Typical textiles and their requirements for repellency finishes according to Lämmermann.<sup>10</sup>

Kind of textile	OR	WR	DS	SR	CF	AS	Н	Р
Sport wear, leisure wear	+	+++	0	+	+	+	+++	++
Uniforms, workwear	+++	+++	++	+++	+	+	++	+++
Upholstery and automotive fabrics		++	+++	++	+++	+++	+	+
Awnings, sunblinds, curtain fabrics	+	+++	+++	0	0	0	0	+
Table and bed linen	+++	++	++	+++	+	0	+	+++
Carpets	++	++	+++	0	++	++	0	+

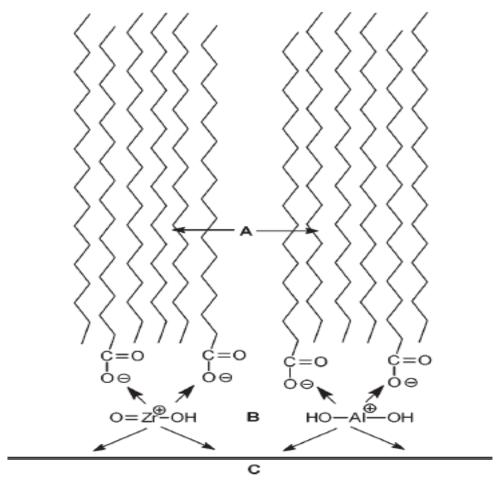
Oil repellency = OR, water repellency = WR, dry soil = DS, soil release = SR, crocking fastness = CF, antistatic = AS, handle = H, permanence = P.

# 8.3 Repellent Chemistry

# 8.3.1 Paraffin Repellents

These were one of the earliest water repellents used, but do not repel oil. Typically the products are emulsions that contain aluminium or zirconium salts of fatty acids (usually stearic acid). These materials increase the finish's adhesion to polar fibre surfaces by forming polar—non-polar junctions as shown in Fig.8.1. The paraffinic portion of the repellent mixture is attracted to the hydrophobic regions, while the polar ends of the fatty acid are attracted to the metal salts at the fibre surface. These finishes can be applied by both exhaustion and padding. They are compatible with most kinds of finishes but they increase flammability. Although they are

available at relatively low cost and generate uniform waterproof effects, the lack of durability to laundering and dry cleaning and their low air and vapour permeability limits the use of paraffin-based repellents.



8.1 Fatty acid metal salts. A, hydrophobic interactions; B, polar interactions; C, fibre surface.

# 8.3.2 Stearic Acid-Melamine Repellents

Compounds formed by reacting stearic acid and formaldehyde with melamine constitute another class of water-repellent materials. An example is shown in Fig.8.2. The hydrophobic character of the stearic acid groups provide the water repellency, while the remaining N-methylol groups can react with cellulose or with each other (crosslinking) to generate permanent effects. Advantages of the stearic acid—melamine repellents include increased durability to laundering and a full hand imparted to treated fabrics. Some products of this type can be effectively applied by exhaustion procedures. Their use as extenders for fluorocarbon repellents is now increasingly replaced by boosters7 as described in Section 8.3.4.Disadvantages of stearic acid—melamine repellents include problems similar to durable

press finishes (a tendency to exhibit finish mark-off, decreased fabric tear strength and abrasion resistance, changes in shade of dyed fabric, and release of formaldehyde).

## 8.2 Stearic acid-melamine derivative.

# 8.3.3 Silicone Water Repellents

Poly-dimethyl-siloxane products that are useful as water repellents can form a hydrophobic layer around fibres (Fig. 8.3). The unique structure of the poly-dimethyl-siloxanes provides the ability to form hydrogen bonds with fibres as well as display a hydrophobic outer surface. In order to gain some measure of durability, silicones designed as water-repellent treatments usually consist of three components, a silanol, a silane and a catalyst such as tin octoate (Fig. 8.4). The catalyst enables not only moderate condensation conditions but also promotes the orientation of the silicone film on the fibre surface. The outward oriented methyl groups generate the water repellency. During the drying step after pad application, the silanol and silane components can react (Fig. 8.5) to form a three-dimensional cross-linked sheath around the fibre. This reaction is often completed after storage of about one day, then providing full repellency. The Si–H groups of the silane are the reactive links in the silicone chain, generating crosslinks or being oxidised by air or hydrolysed by water to hydroxyl groups. These hydroxyl groups may cause further crosslinking, but if too many of them stay unreacted, their hydrophilicity will decrease the repellency.

Advantages of silicone water repellents include a high degree of water repellency at relatively low (0.5–1 % owf) on weight of fabric concentrations, very soft fabric hand, improved sewability and shape retention, and improved appearance and feel of pile fabrics. Some modified silicone repellents can be exhaust applied (to pressure-sensitive fabrics). The disadvantages of silicone repellents include increased pilling and seam slippage, reduced repellency if excessive amounts are applied (for example silicone double layer with polar outside, Fig. 8.6), only moderate durability to laundering (through hydrolysis of siloxane and rupture of the film by

strong cellulose fibre swelling) and dry cleaning (adsorption of surfactants), and no oil and soil repellency. The silicone finish may enhance the attraction of hydrophobic dirt. In addition, the waste water, especially the residual baths, from these finish application processes are toxic to fish.

# 8.3.4 Fluorocarbon-Based Repellents

Fluorocarbons (FC) provide fibre surfaces with the lowest surface energies of all the repellent finishes in use. Both oil and water repellency can be achieved. FC repellents are synthesised by incorporating per-fluoro alkyl groups into acrylic or urethane monomers that can then be polymerised to form fabric finishes (Fig. 8.7). Originally, the per-fluoro alkyl groups were produced by electrochemical fluorination, but today they are produced by telomerisation (Fig. 8.8). The final polymer, when applied to a fibre, should form a structure that presents a dense CF3 outer surface for maximum repellency. A typical structure is shown in Fig. 8.9. The length of the per-fluorinated side chains should be about 8–10 carbons. The small spacer group, mostly ethylene, can be modified to improve emulsification and solubility of the polymer.10 Co-monomers (X, Y, for example stearyl- or lauryl-methacrylate, butyl-acrylate, methylol- or epoxy-functional acrylates and block copolymers from  $\alpha$ ,  $\omega$ -dihydroxy-dimethyl-polysiloxane) affect fabric hand, film formation and durability. In this way and by adding appropriate emulsifiers, FC products can be widely modified for many special performance profiles (see Table 8.2). A more detailed overview of this interesting area of textile chemical engineering is given by Grottenmüller and Holme.

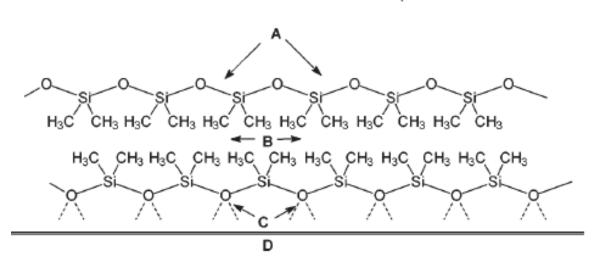
Table 8.2 Effects of blocked isocyanates as FC boosters

Effect	Caused by					
Higher repellency	Better film formation and orientation of the perfluorinated side chains					
Higher permanence to washing, dry cleaning and rubbing	Crosslinking with functional groups on the fibre surface and by incorporation in the network of the reaction products of multifunctional isocyanates					
Lower curing temperature	Catalytic effects, activation of the crosslinking and crystallisation					
LAD (laundry-air-dry), lower temperature of the repellency regeneration after washing	Better and easier orientation of the perfluorinated side chains					
Less VOC (volatile organic compounds)	Higher fluorocarbon efficiency (less FC polymer for equal repellency)					
Softer hand	Less FC polymer caused by higher flourocarbon efficiency					

8.3 Polydimethylsiloxane on a fibre surface. A, hydrophobic surface; B, hydrogen bonds to polar surface; C, fibre surface.

8.4 Components of a silicone water repellent.

8.5 Silanol-silane reaction. A is removal of H<sub>2</sub>; B is Si-O-Si crosslinked polymer; tin octoate is the catalyst.



8.6 Silicone double layer on the fibre. A, polar surface; B, hydrophobic attraction of the methyl groups; C, hydrogen bonds to polar fibre surface; D, fibre surface.

Most FC products are padded, dried and cured. Heat treatment causes an orientation of the perfluoro side chains to almost crystalline structures. This is crucial for optimal repellency. Washing and dry cleaning disturb this orientation and reduce finish performance. The orientation must be regenerated by a new heat treatment (ironing, pressing or tumble drying). But with some new FC products, drying in air is sufficient (laundry–air–dry or LAD products). Tailored FCs and blocked isocyanates, the so-called boosters, are used for this effect. Depending on the kind of blocking group, the isocyanate is activated at different temperatures and then reacts with the functional groups of the FC, the fibre or with itself (crosslinking). This fixation on the fibre surface provides durability to washing, dry cleaning and rubbing as a second important effect. Boosters also cause better film formation and thereby higher repellency effects. Useful effects of boosters are listed in Table 8.2. However, high amounts of boosters adversely affect fabric hand. General advantages of fluorocarbon-repellent finishes include low active add-ons (< 1 % owf) and more rapid drying of treated fabrics. Special FCs allow improved soil release during household laundering or stain resistance on nylon, which is especially useful for carpets. Disadvantages of fluorocarbon repellents include high cost, grey need for special treatment of waste water from application processes and the fact that they usually cannot be applied effectively by exhaustion (but there are some new FC products that are exceptions to this rule). Low-curing FCs are another new development. They get their repellency without heat, only after drying at room temperature. This is of interest for the

impregnation of garments, upholstery and carpets. An inevitable disadvantage is their low durability because of the lack of fixation by crosslinking. A novel FC development is inspired by nature and therefore called bionic finishes. Fluorocarbon polymers are applied together with dendrimers, causing self-organisation where the fluorocarbon chains are enriched on the surface and cocrystallise with the dendrimers. Dendrimers are highly branched oligomers with non-polar chains forming a star-brush structure. They force the polar parts of the FC polymers to form the surface structure mentioned at the beginning of this section. The resulting polar and non-polar sandwich arrangements are highly ordered, causing equal or better repellency effects with lower amounts of fluorocarbon compared to dendrimer-free FC finishes. Other advantages include low condensation temperature (80-130 °C), high abrasion resistance, good wash permanence and soft hand. A hypothetical structure of a dendrimer is shown in Fig. 8.10. An overview on the importance of FC finishes in the three market segments of clothing, household and technical textiles is presented by Otto. An important group of water-repellent textiles are micro-fibre fabrics finished with fluorocarbon polymers. Emerising creates further effects that are of interest for leisure wear. Problems and their solutions that arise from this finish combination are discussed by Nassl et al. A group of FC finished articles with special importance are ballistic fabrics, providing protection against bullets, splinters and cutting. They consist of several layers of para-aramide wovens, thoroughly finished with fluorocarbon polymers. Without this water repellency they would lose their protective action when wetted (gliding effect of water.)

$$R_{F}CH_{2}CH_{2}OH + HOCC = CH_{2} \xrightarrow{-H_{2}O} R_{F}CH_{2}CH_{2}OCC = CH_{2}$$

$$R_{F} = C_{m}F_{2m+1}$$

$$R = H, CH_{3}$$

$$R_{F} = C_{m}F_{2m+1}$$

$$C_{H_{2}} = C_{H_{2}} C_{H_{2}} C_{H_{2}}$$

$$C_{H_{2}} = C_{H_{2}} C_{H_{2}} C_{H_{2}} C_{H_{2}} C_{H_{2}}$$

$$C_{H_{2}} = C_{H_{2}} C_{H_{2}} C_{H_{2}} C_{H_{2}} C_{H_{2}}$$

$$C_{H_{2}} = C_{H_{2}} C_{H_$$

8.7 Preparation of perfluoro containing acrylic polymers.

$$n F_2 C = C F_2 \xrightarrow{\text{UV}} F_3 C (C F_2 C F_2)_n I \xrightarrow{\text{H}_2 C = C O C C H_3} F_3 C (C F_2 C F_2)_n C H_2 C H O C C H_3$$

$$n = 2 - 5$$

$$F_3 C (C F_2 C F_2)_n C H_2 C H O C C H_3 \xrightarrow{\text{I}_1 \text{II}} F_3 C (C F_2 C F_2)_n C H_2 C H_2 C H_2 C H_3$$

$$F_3 C (C F_2 C F_2)_n C H_2 C H O C C H_3 \xrightarrow{\text{I}_1 \text{II}} F_3 C (C F_2 C F_2)_n C H_2 C H_2 C H_2 C H_3 C H_3$$

8.8 Telomerisation process for preparing fluoroalkyls.

8.9 Fluorocarbon repellent on fibre surface. m = 8-10. X and Y are co-monomers, mainly stearylacrylates. R = H or  $CH_3$  (polyacrylic or polymethacrylic acid esters). A is the fibre surface.

$$R = -C - C_{17}H_{35}$$
 or  $-(CH_2)_nNHC - C_{17}H_{35}$ 

# 8.10 Hypothetical structure of a dendrimer synthesised from three distearyl-amines or -amides and a trifunctional isocyanate X(N=C=O)<sub>3</sub>.

# 8.4 Evaluation of Textiles Treated With Repellent Finishes

Rapid and simple tests for water repellency are the water drop test (method standardised by TEGEWA) and the spray test. If there is insufficient differentiation, even with a higher distance between sample and nozzle, the water resistance with the hydrostatic pressure test should be checked. Some of the most important quantitative test methods are given in Table 8.3. For fabrics that require durable repellency performance, the test methods in Table 8.3 can be applied to fabrics that have been laundered or dry cleaned by standard methods (as AATCC TM 124 and TM 86) in order to determine the durability of the repellency properties. Three groups of corresponding test methods may be distinguished, static drop tests, spray tests and hydrostatic pressure tests.

## 8.5 Troubleshooting For Repellent Finishes and Particularities

Conditions for high repellency of finished fabrics include a close-packed textile structure with small inter-yarn spaces (therefore better woven than knitted) with fine yarns (preferably microfibres or microfilaments) and a uniform distribution of the repellent finish. This last condition is enhanced by the removal of protruding surface fibres by singeing or shearing. Any residual sizing agents or surfactants present on the fabric can defeat the repellency properties. All fabrics destined for repellent finishes should be well prepared prior to finishing.19 Non-rewetting, thermally degradable or volatile surfactants such as isopropanol should be used in the repellent finish bath to maximise the final properties. Repellent finishes are usually compatible with easy-care and durable press finishes and many softeners. However, most silicone products interfere with the oil repellency of fluorocarbon finishes and should generally

be avoided in an oil repellent formulation. This is a remarkable contrast to the incorporation of silicone segments in the backbone chain of fluoro-polymers, which generates a special soft handle.

The combination of FC and antistatic finishes, achieved with selected products is important for synthetic micro-fibre textiles. Other common finish combinations include hand builders, flame retardant and antimicrobial agents, which generate valuable and useful multifunctional finishes. FC finishes alone do not sufficiently prevent coffee, tea and other foodstuffs from 'dyeing' nylon and wool carpets with coloured spots, especially when these liquids are not quickly removed. Stain blocking for these carpets is achieved by the combination of FC products with syntans. The latter are also used for wet fastness improvement of acid dyed nylon. Stain repellency is promoted by hydrophobic finishes, mostly fluorocarbons, but with decreasing costs also bysilicones, fatty acid-modified melamine products and by paraffin waxes fixed with zirconium salts.

Naturally, water repellency impedes the access of the washing liquor during laundering. Therefore, so-called dual-action fluorocarbon block copolymers were developed, which combine repellency in the dry state and soil-release effects in an aqueous environment (see Chapter 7 on soil-release finishes). Dual active fluorocarbons enable a better removal of oily stains and dirt in domestic washing or laundering. With conventional FC products, the wash water is hindered from wetting and penetrating the fabric. Dual action fluorocarbons are called hybrid fluoro-chemicals because they are block copolymers containing hydrophobic (like the usual FCs) and highly hydrophilic segments. In air the per-fluorinated side chains are, as usual, outwardly oriented and develop high repellency, but in water the double face surface structure flips and the hydrophilic segments turn outwards to promote the wash effect as an oily soil release finish. During heat drying or ironing the sandwich-like surface flips again to generate water, oil and soil repellency. Traditional commercial FC products consist of 15-30 % fluorocarbon polymer, 1-3 % emulsifier, 8-25 % organic solvents and water. The exhaust air of the drying and curing processes of fluorocarbon finishes therefore often contains high amounts of volatile organic compounds (VOC), like glycols and other organic solvents and a lower content of residual monomers. Cellulosic fibres retain most of the glycols. The exhaust air problem is greater with FC finishes on synthetic fibres.

Table 8.3 Test methods for fabrics with repellent finishes

Test method	Procedure summary
AATCC TM 22 – Water repellency: spray test	Treated fabric is stretched taut, held at a 45° angle and sprayed with 250 ml of water from above. The resulting wetting pattern, if any, is rated using photographic standards. This is a simple, rapid method suitable for the plant floor.
AATCC TM 42 – Water resistance: impact penetration test	Similar to AATCC TM 22, but a weighed piece of blotter paper is placed under the fabric. The weight gain of the paper after 500 ml of water has been sprayed on the fabric is recorded.
AATCC TM 35 – Water resistance: rain test	The treated fabric, backed by a weighed piece of blotter paper, is sprayed with water under constant hydrostatic pressure for 5 min. The weight gain of the paper after the test is recorded. This test requires a special apparatus.
ISO 9865 (DIN 53 888) Bundesmann rain-shower test	Four samples of treated fabric are subjected to simulated rain for 10 min. The fabrics (placed on inclined cups and sealed at the edges) are in constant motion and the side of the fabric not exposed to the rain is subjected to a rubbing action. The repellency of the fabric is determined by the appearance of the wetted side, the amount of water absorbed by the fabric and the amount of water passing through the fabric. This test requires an elaborate special apparatus.
AATCC TM 127 and ISO 811 or EN 20 811 – Water resistance: hydrostatic pressure test (former DIN 53886, Schopper- or Schmerber test)	One surface of the treated fabric is subjected to a constantly increasing hydrostatic pressure until three points of leakage appear on the opposite surface. The pressure at the third point of leakage is recorded in centimetres or metres on a water gauge. A static pressure variation of this test determines the time until the named leakage occurs at a given pressure. This test requires a
AATCC TM 118 – Oil repellency: hydrocarbon resistance test, equivalent to ISO 14 419	properties of eight standard fluids, consisting of a selected series of eight hydrocarbons with decreasing surface tensions, are placed on the treated fabric and observed for wetting, wicking and contact angle. The highest numbered fluid that does not wet the fabric is recorded as the oil repellency rating with grade numbers from 0 to 8. This method is a simple, rapid test suitable for the plant floor but it is a static test involving no mechanical stress that is often common in real life.
DuPont water repellency test 3M Dry soil resistance test	Similar to AATCC TM 118, but with 8 water-propan- 2-ol mixtures with increasing alcohol content. Samples of treated fabric are shaken in a sealed container with a standard dry soil. Loose soil is removed with compressed air and the appearance of the fabrics is compared to photographic standards.

VOCs can be reduced by more than 90 % by the addition of corresponding boosters, providing equal effects with lower quantities of finish products. Long-life by-products of the fluorocarbon synthesis by electrochemical fluorination are the perfluoro-octane-sulfonic acid and their corresponding salts (perfluoro-octyl sulfonate, PFOS). These environmentally critical products (with concerns about persistence, bioaccumulation and potential toxicity) are avoided by synthesis via telomerisation. Silicones and fluorocarbon polymers cannot easily be stripped off, especially when they are cross-linked. As it is the nature of repellent finishes to reduce adhesion, there are problems with back coating and laminating of fabrics finished by padding fluoro-chemicals. One solution is the one side application of the repellents by nip-padding, spray, foam or squeegee techniques.

# **Chapter 9 Soil Release Finishes**

### 9.1 Introduction

The easy-care attributes of garments made from durable press finished cotton and synthetic fibres led to their ready acceptance by consumers in the 1960s. However, it quickly became apparent that these garments were more difficult to clean than garments made from natural fibres. A great deal of research in the textile industry was focused on the problem of soil release and many products were introduced to the market to overcome this performance deficiency. Soil-release finishes on textiles facilitate the removal of soils during laundering under common household conditions. The main factors affecting soil release are listed in Table 9.1. Fabrics treated with soil-release finishes are particularly suited to active wear and leisure wear, markets that are continuing to grow in importance. Industrial uniforms and napery are other market areas where soil release is a desirable product feature. Recent work has shown that soil release can even be incorporated into yarn finishes.

Table 9.1 Main factors affecting soil release

Factor	Explanation
Nature of the soil	Oily soil or particulate soil, hydrophobic or hydrophilic, liquid or solid
Kind of fibres	Type of fibre, hydrophilic or hydrophobic, smooth or porous fibre surface
Nature of textile	Textile construction; yarn (staple or filament), fabric (knit, woven or nonwoven)
Effects of preparation	Residual waxes, warp sizes, or other hydrophobic materials
Effects of dyeing and printing	Difference in binder films, residual hydrophobic dyeing auxiliaries
Effects of other finishes	Compatible with antistatic finishes, easy-care finishes and other finishes not harmed by a hydrophilic surface.  Not compatible with conventional repellent finishes and other finishes where hydrophilicity is detrimental to finish performance
Washing conditions	Detergents, hydrodynamic flow in the washing machine

Other fabric properties that are enhanced by soil-release finishes include protection from soil re-deposition during laundering and absorbency or transport of liquid water. Fabrics with

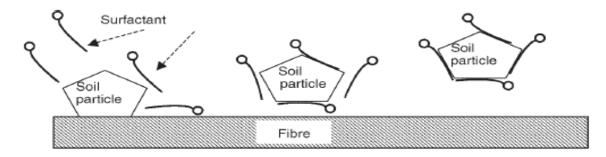
increased absorbency provide garments that feel more comfortable under hot, humid conditions, thus leading the name 'comfort finish' that has often been applied to fabrics treated with soil-release agents.

### 9.2 Mechanisms of Soil Release

Removal of soils from fabrics has been attributed to several mechanisms. These are:

- 1 Adsorption of detergent and absorption of water leading to:
- Rollup of oily soil
- Penetration of soil-fibre interface by wash liquid
- Solubilisation and emulsification of soils.
- 2 Mechanical work leading to:
- Hydrodynamic flow carrying away the removed soil
- Fibre flexing to force soil from between fibres
- Surface abrasion to remove soil physically
- Swelling of finish to reduce inter-fibre spacing.

Of these mechanisms, solubilisation and emulsification of soils are controlled by detergent composition, hydrodynamic flow is controlled by washing machine design and fibre flexing is controlled by fabric construction. The textile chemist can only influence the mechanisms that involve the fibre surface, i.e. rollup of oily soil, penetration of soil–fibre interface, surface abrasion and finish swelling. Finishes have been developed that provide soil release performance by taking advantage of all of these mechanisms.



9.1 Release of particulate soil.

Particulate soil is removed from fibres by a two-step process. First, a thin layer of wash liquid penetrates between the particle and the fibre surface, enabling surfactants to adsorb onto the particle surface (Fig. 9.1). Then, the particle becomes solvated and is transported away from the fibre and into the bulk of the wash liquid by mechanical action. Finishes that are hydrophilic (enhancing penetration of the fibre-soil interface) with low adhesion to soil under washing conditions should improve particulate soil release. Ablative or sacrificial finishes that leave the fibre surface during washing and take the soil particles along with them can also benefit particulate soil release. For the most part however, the removal of particulate soils is determined by detergent composition and mechanical action. Oily soils that are liquids at wash temperatures are released by a 'roll up' mechanism (Fig. 9.2), if the surface free energies of the fibre oil have relationship indicated and the by:

$$R = \gamma_{FO} - \gamma_{FW} + \gamma_{OW} \cos\Theta > 0$$
 [9.1]

where R is the resultant force of the interfacial energies between the fibre and the oil,  $\gamma FO$ ; the fibre and the wash liquid,  $\gamma FW$ ; and the oil and the wash liquid,  $\gamma OW.1$  The angle  $\Theta$  is the angle formed at the intersection of the fibre–oil–water interface (Fig.9.3). For R to be greater than 0, the contact angle  $\Theta$  must be  $180^{\circ}$  ( $\cos\Theta$  =–1). This leads to:

$$\gamma_{FO} - \gamma_{FW} > \gamma_{OW}$$
 [9.2]

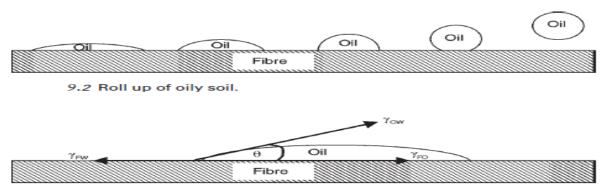
indicating that the difference between the two fibre liquid interactions must be greater than the interfacial tension of the oil with the wash liquid. Analysis of this inequality leads to the following conclusions: • A low fibre—wash liquid interfacial energy is desired, that is a hydrophilic finish is preferred for spontaneous oil roll up.

- A high fibre—oil interfacial energy is desired, that is the finish should also be oleo-phobic.
- A low interfacial tension between the oil and the wash liquid will favour oily soil release.

Additional finish characteristics that should facilitate oily soil release include the ability of the finish to swell during the washing process and flexibility under mechanical action. As in particulate soil release, sacrificial finishes are expected to benefit oily soil release.

The effects of fabric and yarn construction on soil release must be considered. Tightly woven fabrics will be more difficult to clean than loosely knit fabrics and yarns made from spun fibres will be more difficult to clean than yarns made from textured filament fibres because the tighter structures will have less ability to flex from mechanical action during laundering.

The colour of the treated and stained fabric cannot be ignored. Very dark fabrics and very light fabrics can appear to have less soil simply from optical effects. Table 9.2 gives an overview on the chemical mechanisms of soil-release finishing.



9.3 Fibre-oil-water interface.

Table 9.2 Chemical mechanisms of soil-release finishes

Chemical mechanism	Examples of structure types involved
Increasing hydrophilicity, swelling of fibre and finish	PVA, CMC, caprolactam oligomers, ethoxylated products, sulfonates, polyacrylic acid or adipic acid copolymers
Some hydrophobicity, (HLB value about 15)	Hybrid fluorocarbons (dual action block copolymers), or polyacrylic acid esters or polyethylene terephthalate block copolymers
Electrostatic repulsion	Anionic polymers such as polyacrylates and CMC in alkaline washing liquors or sulfonates repulse partly negatively charged soil particles or micelles, including dispersed soil particles
Protective coating with ablative or sacrificial finishes	PVA, CMC, starch
Alkali treatment of polyester Plasma treatment	New carboxylic and hydroxyl groups on the fibre surface by hydrolysis of ester structures  New carboxylic and hydroxyl groups on the fibre surface by carbon radical formation and oxidation

HLB is the hydrophilic-lipophilic balance; PVA, polyvinyl alcohol; CMC, carboxymethyl cellulose.

## 9.3 Soil-Release Chemistry

Finishes providing soil release can be classified in numerous ways, by method of application, by fibre type used with them, by chemical structure, electrical charge and so on. Here, chemical structure will be used.

# 9.3.1 Carboxy-Based Finishes

Some of the earliest carboxy-based finishes were developed to be used in conjunction with durable press finishes on cotton and cotton blend fabrics. The first patent appeared in 1969. The

composition of these finishes is based on acrylic and methacrylic acid and ester copolymers (Fig. 9.4). An ester to acid ratio of ~70:30 is typical. This ratio seems to provide the proper blend of hydrophilicity and oleo-phobicity (hydrophilic-lipophilic balance, HLB) required for a soil-release finish. The HLB scale8 is often used for the pre-selection of surfactants and ranges from about 0 (very hydrophobic) to nearly 20 (very hydrophilic). For good soil release performance, HLB values of about 15 are favoured. But this is only a rough orientation, because the important copolymer block arrangements are not expressed by these HLB values. Greater hydrophilicity would strongly reduce durability to laundering. These products are usually pad applied in combination with DMDHEU cross linkers and provide soil release by a finish swelling mechanism (Fig. 9.5). The ease of incorporating different acrylic monomers into copolymers has led to a wide variety of available finishes. Often a monomer such as N-methylol acrylamide that can react easily with DMDHEU during the finish curing step is added for increased laundering durability. For these finishes to be effective, about 2.5 % solids add-on of the soil release polymer is necessary. Other carboxy polymers that have been used as soilrelease finishes include styrene-maleic anhydride copolymers and sodium carboxy-methyl cellulose (Na-CMC).

$$\begin{array}{c|c} R \\ CH_2 - C \\ C = O \\ OH - X \end{array} \begin{array}{c} CH_2 - CH \\ C = O \\ OR' - y \end{array}$$

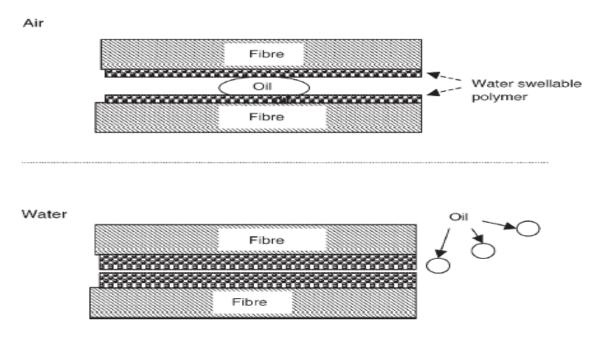
$$R = H, CH_3$$

$$R' = CH_3, CH_2CH_3$$

9.4 Acrylic copolymer soil-release finishes.

## 9.3.2 Hydroxy-Based Finishes

One of the earliest soil-release materials was starch, which functioned as a sacrificial treatment. Other starch- and cellulose-based products that have been used as soil-release agents include methyl cellulose, ethyl cellulose, hydroxy-propyl starch, hydroxy-ethyl cellulose, hydroxy-propyl-methyl cellulose and hydrolysed cellulose acetates. With some exceptions, these finishes lack the laundering durability desired in a finish expected to last the life of a garment and must be applied in combination with a binder or crosslinking agent.



9.5 Mechanism of soil release with acrylic copolymers.

### 9.3.3 Ethoxy-based finishes

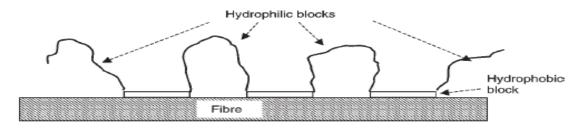
A great variety of materials containing oxyethylene groups have been offered as soil-release finishes. These products may contain actual polyethylene blocks or ethylene oxide reaction products with acids, alcohols, amines, phenols, etc. Binding agents may or may not be required, depending on the durability of the finish. One important group of soil-release agents for polyester fibres is based on condensation copolymers of terephthalic acid with ethylene glycol and polyethylene glycol (Fig. 9.6). The structure of this polyester–ether copolymer contains blocks of polyethylene terephthalate and poly-oxyethylene terephthalate that provide a structure that has regions of hydrophilicity interspersed with hydrophobic regions that have a strong attraction for the polyester surface (Fig. 9.7). These products can provide extremely durable soil-release properties for polyester fabrics by either exhaust or pad applications with about 0.5 % solids add-on. It is possible to exhaust apply these products during the dyeing process.

A modification of the condensation copolymer compounds involves incorporating anionic character into the polymer chain by use of sulfonated monomers. The necessary hydrophilicity is provided by the sulfonated blocks (Fig. 9.8). Like the other polyester condensation polymers, these anionic products can be applied by exhaust or padding. The exhaustion efficiency can be significantly improved by adding small amounts of magnesium chloride to the application bath. High soil-release performance, excellent softness and combinability with fluorocarbon finishes may be achieved by special silicone/poly-alkylene oxide copolymers. The silicone segments

contain hydrophobic dimethyl-siloxane structures and hydrophilic silicone modifications with ethoxylated or amino group-containing side groups. The different hydrophilic–lipophilic balance (HLB) of the poly-alkylene blocks is adjusted by the ratio of hydrophobic (polypropylene oxide) and hydrophilic (polyethylene oxide) components.

$$\begin{bmatrix} O & O & O \\ C & O & C \\ C$$

9.6 Polyester-ether condensation copolymer.



9.7 Copolymer with hydrophilic and hydrophobic blocks on hydro-phobic fibre.

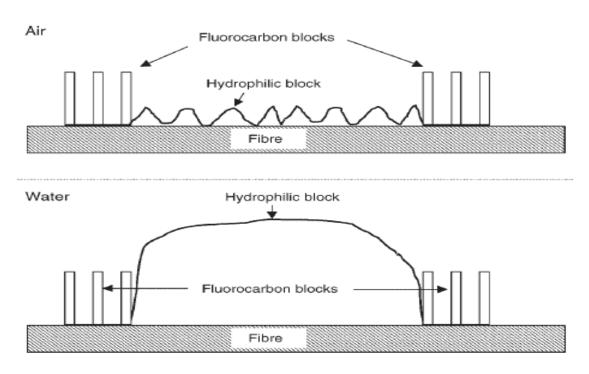
9.8 Modified polyester condensation copolymer.

### 9.3.4 Fluorine-Based Finishes

Attempting to use a fluorine-containing compound to impart hydrophilicity to a fibre surface may at first appear to be a hopeless task owing to the extreme hydrophobicity of most fluorocarbons. However, with the proper choice of copolymer blocks, this seemingly impossible feat can be accomplished. An example of the chemical structure of a successful fluorine-based soil-release compound is shown in Fig. 9.9 and classified as a hybrid fluorocarbon of the block copolymer type. These unique polymers have the unusual property of being hydrophobic and oleo-phobic in air and hydrophilic and oil-releasing during the laundering process. This 'dual action' mechanism is illustrated in Fig. 9.10. The hydrophilic blocks are shielded by the fluorocarbon segments when dry, presenting a repellent surface. After immersion in the wash bath, the hydrophilic blocks can swell and actually reverse the interfacial characteristics of the surface, yielding the hydrophilic surface necessary for oily soil release. Typically, these modified fluoro-polymers are pad applied to fabrics in combination

with durable press crosslinking agents to increase the durability of the finish. The higher cost of the fluoro-chemical soil release agents compared to the acrylic copolymers is somewhat compensated by the low add-on (~0.5 % solids add-on) required for soil-release performance. Mixtures of both polymer types provide a common compromise between efficiency and costs.

## 9.9 Fluorine-based soil-release compound.



9.10 'Dual action' fluorocarbon in air and water.

## 9.3.5 Non-Polymer Soil-Release Treatments

Alkali and plasma treatments of polyester generate a more hydrophilic fibre surface by forming new carboxyl and hydroxyl groups. Under alkaline washing conditions the carboxyl structures become anionic carboxylate groups, giving rise to high electrostatic repulsion of the negatively charged soil particles both in pure form and those enclosed in anionic surfactant micelles.

### 9.4 Evaluation of soil release

The ideal evaluation method for a soil-release finish would be to stain the fabric with the same material that will soil consumers' clothes and then to wash the fabric with the same detergents in the same washing machines at the same water temperatures that consumers will use. This is not practical in real life so compromises must be made. The American Association of Textile Chemists and Colourists (AATCC) has developed standardised procedures for evaluating soil-release finishes that provide a strong indication of the actual finish performance in the real world. When used by fabric manufacturers and their customers, these procedures are helpful guides in product development and resolution of finish quality issues. However, reliance on these tests alone may be misleading. Good finish development procedures should always include some testing with the actual soils and detergents likely to be encountered by the consumer. Evaluation of soil-release effects after washing is mostly visually done by comparison with photographic standards, but also by reflectance measurements and other instrumental techniques, including microscopy. Reflectance data using the Kubelka–Munk equation correlate fairly with the oily soil content but not with residual particulate soil (which is probably partly buried within the fabric and shielded from the light path).

### 9.4.1 Oily Soil-Release Testing

AATCC Test Method 13016 specifies all the parameters that strongly influence soil release of oily soils. A measured amount of corn oil is placed on the fabric to be tested and pressed into the fabric with a specified force. The soiled fabric is washed with a standardised detergent under specific conditions of water temperature and time in a specified washing machine. After tumble drying and equilibration, the stained fabric is compared to photographic standards and rated accordingly. For durability testing, the fabrics are washed prior to staining in accordance with AATCC Test Method 124.17

### 9.4.2 Soil Reposition

AATCC Test Method 15118 can be used to estimate the degree of soil redeposition likely to occur during laundering. The fabrics to be tested are exposed to a soiling medium (two dry soils and fabric swatches pre-treated with a standard oily soil) during a laundering simulation with a standard detergent. The change in reflectance of the fabric before and after the testing is an indication of the redeposition potential of the fabric.

## 9.4.3 Moisture Transport

The procedure given in AATCC Test Method 7919 can also be used to determine the degree of absorbency of fabrics finished with soil-release agents. Typically, near instantaneous wetting of the fabric with water (< 1 s) is expected from non-fluorine- containing soil-release finishes.

# 9.5 Troubleshooting for Soil-Release Finishes

The performance of a soil-release finish depends upon its ability to provide a hydrophilic surface during the laundering process. Therefore any material deposited on the fibre surface that would reduce this necessary hydrophilicity should be avoided. Softeners, lubricants and other products that modify surface properties should be carefully investigated in laboratory trials before being used with fabrics treated with soil-release finishes. The relative amounts of the binders and cross linkers applied in conjunction with acrylic copolymer soil-release compounds must be carefully determined. Too little crosslinking and the soil-release finish is not durable to multiple launderings; too much crosslinking and the finish cannot swell as much as is needed for adequate soil release.

# **Chapter 10 Ultraviolet Protection Finishes**

### 10.1 Introduction

This chapter tackles the prevention from harmful effects of solar ultraviolet (UV) radiation on human skin. These effects were recognised in the early 1990s. Long-term exposure to UV light can result in acceleration of skin ageing, photo-dermatosis (acne), phototoxic reactions to drugs, erythema (skin reddening), sunburn, increased risk of melanoma (skin cancer), eye damage (opacification of the cornea) and DNA damage.1 Numerous publications have appeared concerning the use of textiles to protect the wearer from these harmful effects; a selection are quoted. Solar radiation striking the earth's surface is composed of light waves with wavelengths ranging from the infrared to the UV. Table 10.1 gives the wavelengths, relative intensities and average photon energies of this radiation. Although the intensity of UV radiation is much less than visible or infrared radiation, the energy per photon is significantly higher. The very high energy of the UV-C photons is mostly absorbed by ozone in the higher regions of the atmosphere decreasing their relative intensity on the earth surface to almost zero. But the energies of UV-A and UV-B photons that reach the earth surface exceed the carboncarbon single bond energy of 335 kJ mol-1, which is why UV radiation can be used to initiate chemical reactions. The actual damage to human skin from UV radiation is a function of the wavelength of the incident radiation, with the most damage done by radiation less than 300 nm. If this erythemal effect is multiplied by the intensity of the incident solar light, as a function of wavelength, the wavelengths of maximum danger to skin are 305–310 nm. Therefore, to be useful in protecting the wearer from solar UV radiation, textiles must demonstrate effectiveness in the 300–320 nm range.

Table 10.1 Characteristics of solar radiation striking the earth's surface

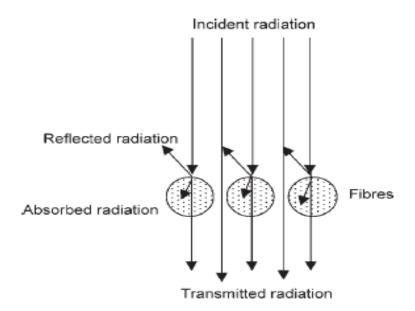
Classification	Wavelength (nm)	Relative intensity (%)	Average photon energy (kJ mol <sup>-1</sup> )
UV-B radiation UV-A <sub>1</sub> radiation UV-A <sub>2</sub> radiation Visible radiation Infrared radiation	280-320	0.5	400
	320-360	2.4	350
	360-400	3.2	315
	400-800	51.8	200
	800-3000	42.1	63

To quantify the protective effect of textiles, the solar protection factor (SPF) is determined. The SPF is the ratio of the potential erythemal effect to the actual erythemal effect transmitted through the fabric by the radiation and can be calculated from spectroscopic measurements. The larger the SPF, the more protective the fabric is to UV radiation. In Europe and Australia,

the SPF is referred to as the ultraviolet protection factor (UPF). The SPF is also used with so-called 'sun blocking' skin creams, giving a relative measure of how much longer a person can be exposed to sunlight before skin damage occurs.4 Typically, a fabric with an SPF of > 40 is considered to provide excellent protection against UV radiation (according to AS/NZS 4399: Sun protective clothing – Evaluation and classification, Standards Australia, Sydney). It is possible to realise about 80 % of the theoretical maximum of SPF 200. Since the most probable time for long-term solar exposure is in the summer, the most likely candidates for UV protective finishes are lightweight woven and knitted fabrics intended for producing shirts, blouses, T-shirts, swimwear, beachwear, sportswear, and the like. Industrial fabrics designed for awnings, canopies, tents and blinds may also benefit from a UV-protective treatment.

### 10.2 Mechanism of UV Protection

When radiation strikes a fibre surface, it can be reflected, absorbed, transmitted through the fibre or pass between fibres (Fig. 10.1). The relative amounts of radiation reflected, absorbed or transmitted depend on many factors, including the fibre type, the fibre surface smoothness, the fabric cover factor (the fraction of the surface area of the fabric covered by yarns) and the presence or absence of fibre de-lustrants, dyes and UV absorbers.



10.1 Radiation in contact with a textile surface.

The effect of fibre type on the SPF of undyed fabrics of similar construction is demonstrated in Table 10.2. Cotton and silk fibres offer little protection to UV radiation since the radiation can pass through without being markedly absorbed. Wool and polyester, on the other hand, have significant higher SPFs since these fibres will absorb UV radiation. Nylon falls in between

these extremes. One factor influencing nylon and polyester absorbance is the presence of the de-lustrant TiO2, a material that strongly absorbs UV radiation. If the fibres absorb all of the incident radiation, then the only source of transmitted rays is from the spacing between the yarns. By definition, the theoretical maximum SPF is the reciprocal of 1 minus the cover factor.

Table 10.2 Solar protection factors (SPF) of undyed fabrics<sup>1</sup>

Fabric description	Approximate SPF
Cotton tricot	4
Wool tricot	45
Silk twill	7
Polyester tricot	26
Nylon/elastomer 80/20 tricot	12

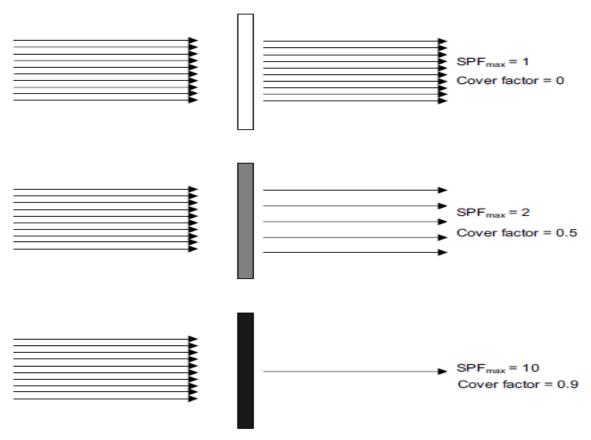
$$SPF_{max} = \frac{1}{1 - cover factor}$$
 [10.1]

Figure 10.2 illustrates the relationship between the maximum SPF and the cover factor. Using a SPF value of 50 as the goal, a fabric with a cover factor of 0.98 and composed of fibres that absorb all of the non-reflected UV radiation will provide its wearer with excellent protection against solar UV radiation. Of course, tight micro-fibre fabrics provide a better UV protection than fabrics made from normal sized fibres with the same specific weight and type of construction. Many dyes absorb UV radiation as well as visible light. A cotton fabric dyed to a deep shade can achieve SPF values of 50 or higher just from the presence of the dye. Since fashion and comfort often dictate the use of lightly coloured fabrics for summer apparel, the need arose for UV absorbing materials that could be applied to fibres to provide the desired SPF values in light shades. Dyestuff and auxiliary manufacturers have responded by developing a variety of materials suitable for use as UV protection finishes.

# 10.3 Chemistry of UV Protection Finishes

The requirements for a material to be effective as a UV protection finish include efficient absorption of UV radiation at 300–320 nm, quick transformation of the high UV energy into the vibration energy in the absorber molecules and then into heat energy in the surroundings without photo-degradation. Further requirements are convenient application to textile fibres and lack of added colour for the treated fibre. Some typical chemical structures useful for UV protection are shown in Fig.10.3 and Fig. 10.4. The reversible chemical reaction, induced by UV absorption of hydroxy-phenyl structures of UV absorbers, is shown. By careful choice of substituents, molecules can be formed that have the required absorbance of UV radiation, lack

of added colour and the necessary affinity to fibres and fastness. In most cases, the UV absorber is applied with the dyes during the dyeing process. Several possible application methods are described by Haerri and Haenz.



10.2 Interaction of radiation with fabrics of varying cover factors.

## 10.4 Evaluation of UV Protection Finishes

Several organisations around the world have developed or have proposed performance standards for UV protection fabrics. These organisations and their standards are summarised in Table 10.3. Although there are multiple standards for UV protective fabrics, there are significant differences between the various organisations. The particular standard for the intended market area should be consulted during fabric development. Before the development of instrumental methods, SPF values of fabrics were determined by irradiating human subjects and measuring the critical amount of radiation necessary to cause skin reddening at a particular wavelength with and without wearing the fabrics. Fortunately, several methods (Table 10.4) are now available that do not result in a sunburned participant. These methods all determine the transmittance of UV radiation through fabrics and calculate the SPF value using standard charts for the solar spectrum and the erythemal effect. UV Standard 801 considers in addition the effects of usage of the finished textiles that normally reduce the UV protection.

Phenyl salicylates

Benzophenones

$$N$$
 $N$ 
 $N$ 
 $N$ 
 $N$ 
 $N$ 
 $N$ 

Benzotriazoles

Cyanoacrylates

R<sub>1</sub> = alkyl, alkoxy, halide, sulfoalkoxy

n = 0, 1, 2

 $X = H, SO_3^-$ 

R<sub>2</sub> = hydroxy, alkoxy, alkylthio, substituted alkyl, substituted phenyl, o-hydroxyphenyl

R<sub>3</sub> = R<sub>2</sub> (identical or different)

Phenyltriazines

# 10.3 Structures of UV absorbers for synthetic fibres.

$$X_1$$
 HO  $X_2$ 

Benzotriazole derivatives

R = alkyl, alkoxy, sulfonate

X<sub>1</sub> = H, sulfonate, halide, sulfonated arylalkyl

$$\begin{array}{c|c} R_2 & O & O \\ II & II \\ \hline (R_1O)_n & ASO_3M \end{array}$$

Oxalic acid dianilide derivatives

R<sub>1</sub> = substituted or unsubstituted alkylbenzyl

R<sub>2</sub> = H, halide, alkyl, phenylalkyl

R<sub>3</sub> = R<sub>2</sub> (identical or different)

A = direct bond or alkylene linkage

M = H or alkali metal

10.4 Structures of UV absorbers for natural fibres.

Table 10.3 Standards for UV protection finished fabrics

Organisation	Standard	Title
Standards Australia/ New Zealand (AS/NZS)	AS/NZS 4399	Sun protective clothing – evaluation and classification
Comité European de Normalisation	Proposed CEN	Classification and marking of UV protective apparel
British Standards Institution	BS 7949	Children's clothing; requirements for protection against erythemally weighted solar UV radiation
American Society for Testing and Materials	Proposed ASTM	Standard guide for labeling of UV-protective textiles
Commission Internationale de l'Eclairage	CIE TC 6-29	Proposed UV protective index for clothing
International Test Association for Applied UV Protection	UV standard 801	UV standard 801

Table 10.4 Test methods for UV protective fabrics

Test method	Title
AATCC TM 183	Transmittance or blocking of erythemally weighted UV radiation through fabrics
BS 7914	Method of test for penetration of erythemally weighted solar UV radiation through clothing fabrics
CEN/TC 248/WG 14	Apparel fabrics; solar UV protective properties, method of test

# 10.5 Troubleshooting For UV Protection Finishes and Combinability

UV absorbers have the same need for wash fastness and light fastness as dyestuffs. Laundering trials should be carried out with all new formulations to confirm that the claimed UV protection is actually active during the life of the garment. One concern is specific to the use of UV absorbers in combination with optical brightening agents (OBA). These brightening agents function by absorbing UV radiation and re-emitting visible light. If a UV absorber is also present in the fibre, the brightening effect from the OBA can be greatly diminished or even absent. Proper choice of an appropriate OBA can minimise this problem. In most other cases combination with other finishes does not reduce the UV protection. A two-step application is necessary if the pH values of the UV protection finish bath and that of the other finishes are very different. The UV protection finish should be applied first. Problems may arise from limited bath uptake after a repellent finish or after calendaring.

# **Chapter 11 Antimicrobial Finishes**

### 11.1 Introduction

Two different aspects of antimicrobial protection provided by chemical finishes can be distinguished. The first is the protection of the textile user against pathogenic or odour causing microorganisms (hygiene finishes). The second aspect is the protection of the textile itself from damage caused by mould, mildew or rot producing microorganisms. Both aspects will be discussed in this chapter. The growth of microorganisms on textiles can lead to functional, hygienic and aesthetic difficulties (for example staining). The most trouble-causing organisms are fungi and bacteria. Under very moist conditions, algae can also grow on textiles but are troublesome only because they act as nutrient sources for fungi and bacteria. Fungi cause multiple problems to textiles including discoloration, coloured stains, and fibre damage. Bacteria are not as damaging to fibres, but can produce some fibre damage, unpleasant odours and a slick, slimy feel. Often, fungi and bacteria are both present on the fabric in a symbiotic relationship. Substances added to fibres, such as lubricants, anti-stats, natural-based auxiliaries (for example size, thickener and hand modifiers) and dirt provide a food source for microorganisms. Synthetic fibres are not totally immune to microorganisms, for example polyurethane fibres and coatings can be damaged. Of course, because of evolution, natural fibres are more easily attacked. Wool is more likely to suffer bacterial attack than cotton, and cotton is more likely than wool to be attacked by fungi.

Antimicrobial finishes are particularly important for industrial fabrics that are exposed to weather. Fabrics used for awnings, screens, tents, tarpaulins, ropes, and the like, need protection from rotting and mildew. Home furnishings such as carpeting, shower curtains, mattress ticking and upholstery also frequently receive antimicrobial finishes. Fabrics and protective clothing used in areas where there might be danger of infection from pathogens can benefit from antimicrobial finishing. These include hospitals, nursing homes, schools, hotels, and crowded public areas. Textiles in museums are often treated with antimicrobial finishes for preservation reasons. Sized fabrics that are to be stored or shipped under conditions of high temperature (~ 40 °C or 100 °F) and humidity require an antimicrobial finish to retard or prevent microbial growth fuelled by the presence of warp size. Textiles left wet between processing steps for an extended time often also need an antimicrobial treatment.

The use of antimicrobial finishes to prevent unpleasant odours on intimate apparel, underwear, socks and athletic wear is an important market need. The odours are produced by the bacterial

decomposition of sweat and other body fluids, and controlling bacterial growth by hygiene finishes reduces or eliminates the problem.

## 11.2 Properties of An Effective Antimicrobial Finish

The growth rate of microbes can be astoundingly rapid. The bacteria population, for example, will double every 20 to 30 min under ideal conditions (36–40 °C or 77–98 °F, pH 5–9). At this rate, one single bacteria cell can increase to 1 048 576 cells in just 7 hours. Therefore, antimicrobial finishes must be quick acting to be effective. In addition to being fast acting, a number of other important criteria can be listed for antimicrobial finishes. The antimicrobial must kill or stop the growth of microbes and must maintain this property through multiple cleaning cycles or outdoor exposure. The antimicrobial must be safe for the manufacturer to apply and the consumer to wear. The finish must meet strict government regulations and have a minimal environmental impact. The antimicrobial finish must be easily applied at the textile mill, should be compatible with other finishing agents, have little if any adverse effects on other fabric properties including wear comfort, and should be of low cost.

### 11.3 Mechanisms of Antimicrobial Finishes

Despite the long list of requirements, a variety of chemical finishes have been used to produce textiles with demonstrable antimicrobial properties. These products can be divided into two types based on the mode of attack on microbes. One type consists of chemicals that can be considered to operate by a controlled-release mechanism. The antimicrobial is slowly released from a reservoir either on the fabric surface or in the interior of the fibre. This 'leaching' type of antimicrobial can be very effective against microbes on the fibre surface or in the surrounding environment. However, eventually the reservoir will be depleted and the finish will no longer be effective. In addition, the antimicrobial that is released to the environment may interfere with other desirable microbes, such as those present in waste treatment facilities. The second type of antimicrobial finish consists of molecules that are chemically bound to fibre surfaces. These products can control only those microbes that are present on the fibre surface, not in the surrounding environment. 'Bound' antimicrobials, because of their attachment to the fibre, can potentially be abraded away or become deactivated and lose long term durability.

Antimicrobial finishes that control the growth and spread of microbes are more properly called bio-stats, i.e. bacterio-stats, fungi-stats. Products that actually kill microbes are biocides, i.e. bacterio-cides, fungicides. This distinction is important when dealing with governmental regulations, since biocides are strongly controlled. Textiles with biostatic properties, however,

are subject to fewer regulations. The actual mechanisms by which antimicrobial finishes control microbial growth are extremely varied, ranging from preventing cell reproduction, blocking of enzymes, reaction with the cell membrane (for example with silver ions) to the destruction of the cell walls and poisoning the cell from within. An understanding of these mechanisms, although important for microbiologists, is not really a requirement for the textile chemist who applies and evaluates the effectiveness of antimicrobial finishes.

# 11.4 Chemistry of Antimicrobial Finishes

### 11.4.1 Antimicrobials for controlled release

Many antimicrobial products that were formerly used with textiles are now strictly regulated because of their toxicity and potential for environmental damage. Products such as copper naphthenate, copper-8-quinolinate, and numerous organo mercury compounds fall into this category. Other materials that still have limited use in specialised areas include tributyl tin oxide (deleted in many countries, Fig. 11.1a), dichloro-phene (Fig. 11.1b) and 3-iodopropynylbutylcarbamate (Fig. 11.1c). These products typically show a very broad spectrum of activity against bacteria and fungi, but suffer from application and durability problems.

Some more useful products of this same general type include benzimidazol derivatives, salicylanilides and alkylol-amide salts of undecylenic acid (particularly effective against fungi). Application of these materials with resin pre-condensates can improve durability to laundering, but also deactivation by reaction with the resin may occur. A widely used biocide and preservation product is formaldehyde. Solutions of formaldehyde in water, called formalin, were used for disinfection and conservation, for example, of biological samples for display. Bound formaldehyde is released in small amounts from common easy-care and durable press finishes. Therefore these finishes include – at least until they are washed – a small antimicrobial side effect. This can also be true for some quaternary compounds, for example wet fastness improvers and softeners. But for more effective requirements specific antimicrobial finishes are necessary. One of the most widely used antimicrobial products today is 2, 4, 4'-trichloro-2'-hydroxydiphenyl ether, known more commonly as 'triclosan' (Fig. 11.1d). Triclosan finds extensive use in mouthwashes, toothpastes, liquid hand soaps, deodorant products, and the like. Although it is effective against most bacteria, it has poor antifungal properties. Triclosan is also important as a textile finish, but since its water solubility is very low, aqueous application requires use of dispersing agents and binders.

(a) 
$$(CH_3CH_2CH_2CH_2)_3Sn-O-Sn(CH_2CH_2CH_2CH_3)_3$$
  
Tributyl tin oxide (TBTO)

2,2'-Dihydroxy-5,5'-dichlorodiphenyl methane

(c) 
$$H_3CCH_2CH_2CH_2-N-C-OCH_2-C=I$$

3-lodopropynylbutyl carbamate

### 11.1 Controlled-release antimicrobials.

Quaternary ammonium salts have been found to be effective antibacterial agents in cleaning products and for disinfecting swimming pools and hot tubs. However, their high degree of water solubility limits their use as textile finishes. Research into controlled-release antimicrobials continues with organo-silver compounds and silver zeolites, which are promising candidates for textile finishes. Silver ions, for example, incorporated in glass ceramic, have a very low toxicity profile and excellent heat stability. These principles are also used for fibre modification, an alternative to the antimicrobial finishes with high permanence. In recent years a variety of antimicrobial modified fibres have been developed, including polyester, nylon, polypropylene and acrylic types. An example of these fibre modifications is the incorporation of 0.5–2 % of organic nitro compounds (for example based on 5-nitrofurfural) before primary wet or dry spinning. Regenerated cellulosics can be modified with carboxylic or sulfonic acid groups, followed by immersing in a solution of cationic antimicrobials which are then fixed to the cellulose by salt bonds. A novel approach to the controlled release of antimicrobials is microencapsulation. These capsules are incorporated either in the fibre during primary spinning or in coatings on the fabric surface.

### 11.4.2 Bound Antimicrobials

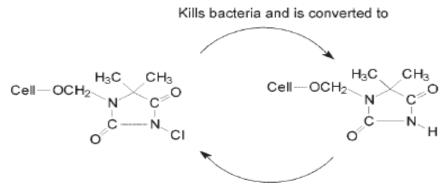
Several antimicrobial finishes that function at fibre surfaces have been commercialised. One popular product is based on octadecyl-amino-dimethyl-trimethoxy-silyl-propyl-ammonium chloride (Fig. 11.2a). This material can be applied by either exhaust or continuous methods. After application, a curing step is required to form a siloxane polymer coating on the fibre surface. This coating immobilises the antimicrobial part of the molecule (the quaternary nitrogen) and provides the necessary durability to laundering. Another bound finish has been developed with PHMB, poly-hexa-methylene biguanide (Fig. 11.2b). PHMB can also be either pad or exhaust applied. This chemical has the proper molecular structure to bind tightly to fibre surfaces, yet still be an effective antimicrobial. The antimicrobial effect of cationically charged materials is thought to involve interaction of the cationic molecule with anionic phospholipids in the microbe's cell walls.

$$CH_3 \quad CI^-$$
 (a) 
$$(CH_3O)_3Si-CH_2CH_2CH_2-N^{+-}C_{18}H_{37}$$
 
$$CH_3$$
 Octadecylaminodimethyltrimethoxysilylpropylammonium chloride 
$$NH \quad NH_2^+ \quad CI^-$$
 
$$-CH_2CH_2CH_2NHCNHCNHCH_2CH_2CH_2-12-16$$
 
$$Polyhexamethylene biguanide$$

11.2 Bound antimicrobials.

This interaction is believed to increase the permeability of the cell walls to the point of cell death. A new and novel approach to bound antimicrobials was recently introduced. Cotton reacted with methylol-5, 5-dimethyldyantoin is then treated with hypochlorite to form chloramines in the fibre (Fig. 11.3). These chloramine sites have antibacterial activity and can function as renewable antimicrobial agents by continued treatment with hypochlorite through household bleaching and washing after reacting with bacteria (Fig. 11.4). Problems with using higher concentrations of chloramines include yellowing with heat (for example ironing) and cellulose fibre damage especially significant strength loss, caused by oxy- and hydrocellulose (generated by hypo-chlorous acid). Another novel approach is the application of chitosan. This modified biopolymer is manufactured from inexpensive natural waste.

### 11.3 Formation of chloramines on cellulose.



Treatment with OCI converts back to

### 11.4 Renewable bound antimicrobial.

Chitin from crustacean shells (e.g. from crabs) is converted to chitosan by alkaline treatment. Chitin is an analogue of cellulose with N-acetyl groups instead of hydroxy groups in position (Fig. 11.5). Alkali splits most of them (75–95 %), generating free amino groups that provide fungi-static and bactero-static effects. This mild antimicrobial activity may be amplified by methylation of the amino groups to quaternary trimethyl-ammonium structures. Chitosan can be applied by microencapsulation or by reactive bonding to cellulose and by crosslinking of chitosan. The advantages of the antimicrobial finish with chitosan include high absorbency properties, moisture control, promotion of wound healing, non-allergenic, non-toxic and biodegradable properties.

Cellulose if R = 
$$-OH$$

Chitin if R =  $-NH-C-CH_3$ 

Chitosan if R =  $-NH-C-CH_3$ 

Chitosan if R =  $-NH-C-CH_3$  and  $-NH_2$ 

Formation of chitosan  $-NH-C-CH_3$ 

Formation of quaternary  $-NH-C-CH_3$ 
 $-OH-C-CH_3$ 
 $-OH-C-C-CH_3$ 
 $-OH-$ 

#### 11.5 Chitosan from chitin.

### 11.5 Evaluation of Antimicrobial Finishes

The AATCC Technical Manual has a number of test methods that are useful for evaluating antimicrobial finishes on textiles. These tests are summarised in Table 11.1. Two types of antimicrobial tests are dominant, the agar-based zone of inhibition tests and the bacteria counting tests. The relatively new ISO/DIS 20645 and the corresponding EN ISO 20645 are based on the agar diffusion test and ISO 11721 is a burial test (part 1 for the determination of an antimicrobial finish and part 2 for the determination of the long-term resistance). A detailed survey and a comparison of the main test methods for evaluating antimicrobial finishes is given by Dring and summarised in Table 11.2. The main difficulties of these tests are mostly poor reproducibility of the test results and often insufficient correlation between laboratory results and actual conditions in the field. Careful attention to detail and trained laboratory personnel are essential for accurate and repeatable results from these methods.

Table 11.1 Antimicrobial test methods used with textiles

AATCC test method	Comments
Antibacterial activity of textile materials: parallel streak method; test method 147 (agar plate test)	Rapid qualitative method for determining anti- bacterial activity of treated textile materials against both Gram-positive and Gram-negative bacteria. Treated material is placed in nutrient agar that is streaked with test bacteria. Bacterial growth is determined visually after incubation. Antibacterial activity is demonstrated by zones of inhibition on and around the textile.
Antibacterial finishes on textile materials, assessment of: test method 100	Quantitative method for determining the degree of antimicrobial activity of treated textiles. The amount of bacterial growth in inoculated and incubated textiles is determined through serial dilutions and subsequent inoculations of sterile agar. Grampositive and Gram-negative bacteria are used.
Antifungal activity, assessment on textile materials: mildew and rot resistance of textiles; test method 30	Four methods for determining the antifungal properties of treated textiles. One method involves testing fabric properties after burial in soil that contains fungi. In a second method, cellulose fabric is exposed to <i>Chaetomium globosum</i> in an agar plate and the subsequent growth visually determined. The third method exposes textiles to <i>Aspergillus niger</i> in an agar plate and visually determines any fungal growth. The fourth method uses a humidity jar to expose textiles to mixture of fungi spores. Any growth on the textile is visually determined.
Antimicrobial activity assessment of carpets; test method 174	Methods are given for the qualitative and quantitative determination of antibacterial activity and the qualitative evaluation of antifungal properties of carpet samples using procedures and materials similar to those in the above test methods.

A more rapid test method, developed by the British Textile Technology Group in the late 1980s, is based on adenosine triphosphate (ATP) luminescence. The growth of microorganisms is assessed by firefly bioluminescent detection and ATP analysis.

# 11.6 Troubleshooting for Antimicrobial Finishes

Difficulties arise in several areas with antimicrobial finishes. The use of binders and resins with controlled-release finishes can lead to stiff hand and fabric strength loss. Colour changes after finishing can also occur. Some consumers may develop dermatitis from prolonged skin contact with antimicrobial finishes. Therefore antimicrobial finishes are excluded from the Öko-Tex Standard 100 label. If residual finish is improperly disposed of at the finishing plant, the desirable and necessary microbes in the waste treatment facility can be destroyed, causing serious ecological problems. Another area of concern is whether the long term use of antimicrobial products in our society will lead to the eventual development of resistant microbes with perhaps deadly consequences. A general problem of antimicrobial finishes is their selective effect. Some are efficient against fungi, others against Gram-positive or Gramnegative bacteria. Therefore commercial biocide formulations are often composed of a mixture

of several substances with different activities. Another general problem is to find the balance between high biocide activity and the requirements of safe handling, including non-toxicity to humans at usual concentrations and environmental demands. Consumers and their organisations have become more aware of toxicological and environmental problems, such as skin irritation, sensitising and allergic potential, biodegradability and bioaccumulation. In response to these concerns, legislation increasingly restricts commerce, handling, use and disposal of dangerous and potentially dangerous substances.

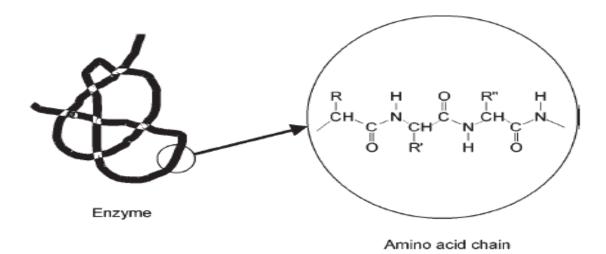
Table 11.2 Comparison of main antimicrobial test methods according to Dring<sup>3</sup>

	Agar plate test	Soil burial test	Saturated atmosphere test	Count test
Procedure	Similar to AATCC TM 147 (Table 15.1) and ISO 20645. Agar may contain a nutrient or be nutrient-free to test if samples are a viable carbon source.	Samples are buried up to 28 days, longer for coatings and plastics.	Samples are sprayed with a mixed spore suspension (fungi) and held over water. Organism must obtain its carbon from material under test.	Inoculated samples are left in contact for 0–24 hour. Organisms are ther extracted and counted.
Evaluation	Visual, growth of contact and halo	Loss of tensile strength or weight. Control samples must rot in about 7 days.	Visual assessment (including a microscope)	Number of colony forming units (cfu)
Advantages	Quick test for bacteria and fungi, variability of test conditions	The most severe test	Especially suitable for testing fungi or yeasts	Relatively quick, distinguishes between biocide and biostat
Disadvantages	Size of halo indicates high diffusion or efficiency of the biocide	Long term and expensive	Long time of incubation	Only for bacteria. Time consuming and expensive
Accuracy (at average sample number	Good (4 samples)	± 10 % (10 samples)	± 10 %	± 30–40 % (6 samples)

# **Chapter 12 Bio-Finishing**

### 12.1 Introduction

Bio-finishing, also called bio-polishing, is a finishing process applied to cellulosic textiles that produces permanent effects by the use of enzymes. Bio-finishing removes protruding fibres and slubs from fabrics, significantly reduces pilling, softens fabric hand and provides a smooth fabric appearance, especially for knitwear and as a pre-treatments for printing. Second rate articles can obtain the high value eye appeal of first rate ones. In denim processing, biofinishing can reduce or eliminate abrasive stones and the aggressive chlorine chemistry, achieving the desired 'worn' looks. Bio-finishing is not only useful for cotton but also for regenerated cellulose fabrics, especially for lyocell and micro-fibre articles. By incorporating enzymes into detergents to remove protruding surface fibres, improved colour retention is achieved after multiple launderings. The disadvantages of bio-polishing are the formation of fibre dust, which has to be removed thoroughly, the reproducibility of the effect (which is dependent upon many parameters) and in the worst case, loss of tear strength. Enzymes are high molecular weight proteins produced by living organisms to catalyse the chemical reactions essential for the organism's survival. They have complex three-dimensional structures composed of long chains of amino acids (Fig. 12.1) with molecular weights ranging from 10 000 to about 150 000 and occasionally to more than 1 000 000.



#### 12.1 Enzyme structure.

These naturally occurring molecules provide a high degree of catalytic specificity unmatched by man-made catalysts. The enzyme and substrate form a 'lock and key' complex that requires the enzyme to have a specific molecular alignment in order to act as a catalyst. The lock and

key theory of Emil Fischer was broadened by Koshland Jr to the induced-fit theory of the enzyme-substrate-complex. Chemical reactions catalysed by enzymes can typically be carried out – as is most usual in nature – under mild aqueous conditions without the need for high temperatures, extreme pH values or chemical solvents. What a dream for every chemist!

Table 12.1 Enzyme treatments of textiles

Type of enzyme	Textile use and effects
Cellulases	Bio-finishing, bio-polishing, anti-pilling, softness, smoothness, lustre improvement and stone-washed effects on denim
Amylases	Standard procedure for the removal of starch warp size
Proteases	In household washing agents better removal of protein containing soil or stains. Anti-felting of wool, accompanied by high loss of weight, tear strength and of the typical handle, <sup>2,3</sup> degumming of silk with the problem of silk fibroin damage
Lipases	In detergents for the hydrolysis of lipids
Pectinases	Hydrolysis of pectins, for example in cotton preparation <sup>4</sup> and retting of flax and hemp
Catalases	Catalyse the decomposition of hydrogen peroxide, important before reactive dying or printing of peroxide bleached fabrics and yarn
Peroxidases	Used as an enzymatic rinse process after reactive dyeing, oxidative splitting of hydrolysed reactive dyes on the fibre and in the liquor, providing better wet fastness, decolourised waste water and potentially toxic decomposition compounds (aromatic nitro-compounds)
Ligninases	Removal of burrs and other plant compounds from raw wool
Collagenases	Removal of residual skin parts in wool
Esterases	In development: polyester finish, removal of oligomers
Nitrilases	In development: polyacrylonitrile preparation for better coloration

Enzymes find commercial use in detergents, leather processing, baking, brewing, cheese manufacture, fruit juice processing, dairy production, animal feed, wine making and textiles. A wide variety of different enzymes finds use in textile areas as shown in Table 12.1. This chapter will focus on cellulases, which are enzymes that catalyse the hydrolysis of the cellulose polymer. The names of the examples of textile-relevant enzymes follow the nomenclature of Duclaux from 1898, characterising an enzyme by the end-syllable 'ase', added to the name of the substrate that is split, synthesised or otherwise catalysed. As with all catalysts, enzymes reduce the activation energy of a specific reaction. The discovery of large quantities of new enzyme systems afforded a more differentiated nomenclature, realised in 1964 by the International Union of Pure and Applied Chemistry (IUPAC) and the International Union for Biochemistry (IUB). In the new enzyme classification (EC) the first number refers to one of

the six main groups and the following numbers to subgroups, for example EC 3.4.5.6, where 3stands for hydrolases. Bio-finishing can be simply defined as a biological way of giving a wet treatment to the textiles. It includes enzymatic de-sizing, bio-scouring, bio-bleaching, bio-washing, bio-polishing, bio-polymer finishing and aromatherapy as well as speciality finishes like wrinkle-free effect, antimicrobial finish, deep sleep finishing, etc. by using some or the other biological means which have been classified into three types, namely:

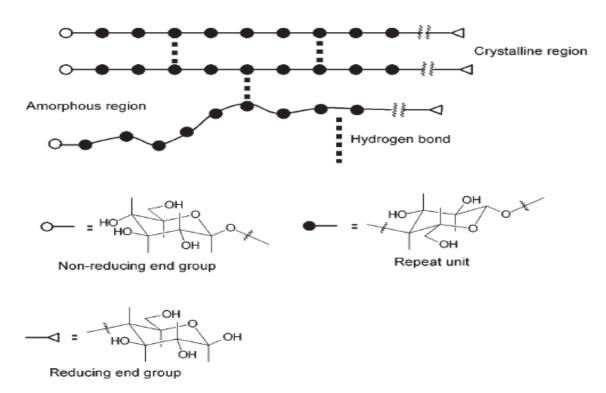
- 1. Enzymes: The main bio-finishing methods are enzymatic bio-finishing responsible for enhancing a number of fabric properties, including its appearance and feel.
- 2. Biopolymers: Apart for the enzymatic method of bio-finishing, some naturally available biopolymers are used like chitosan and  $\beta$ -cyclo-dextrin, which are responsible for antimicrobial finishing treatments.
- 3. Aromatherapies: Some aromatherapies like neem, rose, lavender, jasmine, aloe vera and many others are used for bio-finishing fabrics to inculcate properties like antimicrobial, fragrance, deep sleeping finish, anti-inflammatory, etc.

# 12.2 Action of Cellulase Enzymes On Cellulose

Enzymes that hydrolyse cellulose are found in nature in both Tricho-derma and Humicola fungi. The  $\beta$  (1–4) linkages between adjacent repeat units in the cellulose polymer chain are the sites that are vulnerable to catalytic hydrolysis by cellulases (Fig. 12.2). These enzymes are thus able to provide a food source for the organisms by producing glucose from cellulose. Industrial production of cellulases involves large scale growth of fungal populations, followed by extraction of the enzyme. The extracted enzymes contain multiple components that work together to yield glucose from a cellulose polymer chain. At least four components have been identified as being important in providing efficient glucose production. Endo-glucanases hydrolyse cellulose at random locations along the polymer chain. Beta-glucanases hydrolyse cellulose polymers from the non-reducing end producing glucose and leaving a polymer chain with one less repeat unit.

Cellulose polymer chain

### 12.2 Celluase hydrolysis site.



12.3 Schematic representation of cellulose fibre internal structure.

Cellobio-hydrolases produce cellobiose (the glucose dimer) from polymer chains, and cellobiases convert cellobiose into glucose. These components and their points of attack on cellulose polymer chains are shown schematically in Fig. 12.3 and Fig.12.4. Numerous studies of the mechanism of cellulase interactions with cellulose have been reported. The generally accepted mechanism is adsorption of the endo-glucanase, beta-glucanase or cellobio-hydrolase components onto the fibre surface followed by complex formation with the cellulose polymer chain and water. After hydrolysis of the  $\beta$  (1–4) bond, the enzyme desorbs and is available for further adsorption and reaction. Endo-glucanase effectively opens up more of the fibre structure to attack by beta-glucanase and cellobio-hydrolase. These two components in turn produce

water-soluble fragments from the exposed area. This synergistic action allows for rapid reduction in fibre strength.

Enzymes are proteins. Like other proteins, enzymes consist of long chains of amino acids, held together by peptide bonds. They are present in all living cells. They control the metabolic process and convert nutrients into energy and fresh cell material. The enzyme also takes part in the breakdown of food material into simpler compounds. Some of the best known enzymes are those found in the digestive tract where pepsin, trypsin and peptidases breakdown proteins into amino acids, lipases split fats into glycerol and fatty acids and amylases breakdown starch into simple sugars. Enzymes are biocatalysts. Unlike food proteins such as egg albumin, gelatine or soya protein, they help to catalyse reactions. Without being consumed in the process, enzymes can speed up chemical processes that would otherwise run very slowly, if at all. To date more than 3000 different enzymes have been isolated and classified. However, only a limited number of enzymes are commercially available and an even smaller number are used in large quantities. More than 75% of industrial enzymes are hydrolytic in action. Proteindegrading enzymes constitute about 40% of all enzyme sales. Detergents (37%), textiles (12%), starch (11%), baking (8%) and animal feed (6%) are the main industries that use about 75% of industrially produced enzymes. Major companies involved in the preparation and marketing of enzymes are Novozymes (formerly Novo Nordisk), Genencor, DSM, Röhm and Haas and Clariant (formerly Sandoz), among others. The enzymes differ from each other due to the following features:

- Differences in amino acids present
- Difference in the sequence of amino acids in the structure
- The presence or absence of metal ions
- The structure conformation as a whole

The principal properties of enzymes are

1. Specific in action: Contrary to inorganic catalysts such as acids, bases, metals and metal oxides, enzymes are very specific in action. Every enzyme can breakdown or synthesise one particular compound. Various species of a particular enzyme may have limited actions in the compound with which they react. Most proteases, for instance, can break down several types of protein, but in each protein molecule, only certain bonds will be cleaned depending on the which species of enzyme is used.

- 2. High efficiency: The enzyme catalase, which is found abundantly in the liver and red blood cells, is so efficient that in 1 min, one enzyme molecule can catalyse the breakdown of five million molecules of hydrogen peroxide to water and oxygen.
- 3. Natural source of origin: Enzymes are present in all biological systems. They come from natural systems, and when they are degraded the amino acid of which they are made of can be readily absorbed back into nature.

Fruits, cereals, milk, fats, meat, cotton, leather and wood are some typical candidates for enzymatic conversion in the industry. Both the usable products and the waste of most enzymatic reactions are nontoxic and readily broken down. Bio-finishing, also called bio-polishing, is a finishing process applied to cellulose textiles that produces permanent effects from the use of enzymes. Bio-finishing removes protruding fibre and slubs from fabrics, significantly reduces pilling, softens fabric hand and provides a smooth fabric appearance, especially for knitwear and as a pre-treatment for printing. In denim processing, bio-finishing can reduce or eliminate abrasive stone and aggressive chlorine treatments. Bio-finishing is not only useful for cotton but also for regenerated cellulose fabrics, especially for lyocell and micro-fibre articles. The enzymes may be incorporated into detergents to remove protruding surface fibres. This improved colour retention is achieved after multiple launderings. The disadvantages of bio-finishing are the formation of fibre dust, which has to be removed thoroughly, the reproduce-ability of the effect and in the worst case, loss of tear strength.

Enzymes are high molecular weight proteins produced by living organisms to catalyse the chemical reaction essential for the organism's survival. They have complex three-dimensional structures composed of long chains of amino acids with molecular weights ranging from 10,000 to about 150,000 and occasionally to more the 10,000,000. These naturally occurring molecules provide a high degree of catalytic specificity unmatched by man-made catalysts.

The advantages of using enzymes are as follows:

- Enzymes are biodegradable and ecofriendly.
- The enzyme remains intact at the end of reaction by acting as catalyst.
- There is remarkable chemical precision due to far greater reaction specificity, even in the absence of functional group protection. Enzyme engineering can further improve stability and specific activity.

- The processes are easy to control because the enzyme activity depends upon operating conditions.
- Applicable under milder conditions of temperature (below 100°C), pressure (atmospheric) and pH (around neutral). However, high-temperature stable enzymes are also available nowadays for faster reactions.
- Lower energy input leading to lower fuel costs and lower emissions of greenhouse gases to the environment.
- Enzymatic reactions are several orders of magnitude faster.
- By-products are rarely generated.
- Fewer wastes and minimum disposal problems.
- Safe to handle and noncorrosive.
- Reduced pollution loads due to easy biodegradability of enzymes.
- Safely dischargeable after use, sometimes after inactivation by changing pH or temperature.
- Reduction in global warming, saving in acidification and nutrient enrichment.

Some of the problems and challenges of enzyme application are:

- Enzyme recovery and reuse.
- Scale up in industrial environment.
- Higher process time.
- May not always be economically feasible.

Precautions to be strictly followed are

- Live steam should not be used.
- Chemicals should be added in pre-diluted form.
- Compatible ionic surfactants should be used.
- Non-ionic wetting agents with appropriate cloud points are preferred.
- The temperature, pH and heavy metal contamination should be closely monitored.

• The enzymes should be carefully stored under prescribed conditions.

Most enzymes are much larger than the substrates they act on, and only a small portion of the enzyme (around 2–4 amino acids) is directly involved in catalysis. The region called the 'active site' contains these catalytic residues. The substrate is bound at the active site, and then the reaction proceeds. Enzymes can also contain sites that bind cofactors, which are needed for catalysis. As are all proteins, enzymes are long linear chains of amino acids that fold to produce a three-dimensional product. Each unique amino acid sequence produces a specific structure, which has unique properties. Individual protein chains may sometimes group together to form a protein complex. Most enzymes can be denatured (i.e., unfolded and inactivated) by heating or chemical denaturants, which disrupt the three-dimensional structure of the protein. Depending on the enzyme, denaturation may be reversible or irreversible. Enzymes are very specific in action. One Nobel laureate organic chemist suggested the simplistic 'lock and key' model (Fig.12.4). Both the enzyme and the substrate possess specific complementary geometric shapes that fit exactly into one another. The high substrate specificity of enzymes is due to the individual architecture of the active site where only certain molecules can 'stereo-fit in', i.e., fitting one body (key) into another body (lock) by exact matching of three dimensional space in between. The enzymes have a true activity centre in the form of fissures, holes, pockets, cavities or hollows. The active site is a perfect fit for a specific substrate, and once the substrate binds to the enzyme no further modification is necessary. The enzyme catalysis operates first to form an enzyme-substrate complex. At the active site on the enzyme, the hydrolysis of the substrate is accelerated. The decomposition products of the substrate thus formed are usually unstable in the active site due to steric hindrances that force them to be released and return the enzyme to its initial unbound state so that further substrate is reabsorbed on the active site of the enzyme. The process continues until the enzyme is deactivated by conditions in the processing bath. Deactivation may occur by competitive or non-competitive chemicals called 'bogies' in the processing bath. The competitive bogies compete with the substrate for the enzyme, whereas the non-competitive bogies are adsorbed on the enzyme, causing the shape of the enzyme to change; both bogies prevent enzymatic catalysis from occurring. All enzymatic systems function best within a narrow range of pH and temperature. If they are too far outside the range, the enzyme becomes deactivated by changes in the threedimensional structure. As the enzyme twists and coils into a shape that prevents sorption between enzyme and substrate, catalysis no longer occurs. However, although this model explains enzyme specificity, it fails to explain the stabilisation of the transition state that enzymes achieve. Koshland suggested a modification to the lock and key model called the 'induced fit model': the substrate induces a change in the shape of the active site to the correct fit. Because enzymes have rather flexible structures, the active site is moulded into the precise positions that enable the enzyme to perform its catalytic function. Most of the enzymes are inactivated or destroyed by temperatures over 75°C. Once destroyed, they cannot be revived or reactivated. Currently thermo-stable enzymes (such as a-Bacillus subtilis, a-Bacillus licheniformis, etc.) are available that can withstand temperatures up to 90°C. Certain enzymes require some specific bivalent metallic ions (e.g., Ca2+, Mg2+, Fe2+, Mn2+, etc.) as activators which probably act by stabilizing the enzyme–substrate complex or sensitizing the substrate to the attack of enzymes. Certain chemicals such as alkalis, antiseptics, and acid-liberating agents tend to inhibit enzyme activity.

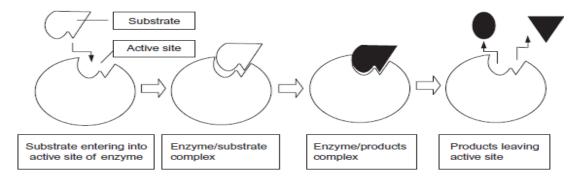
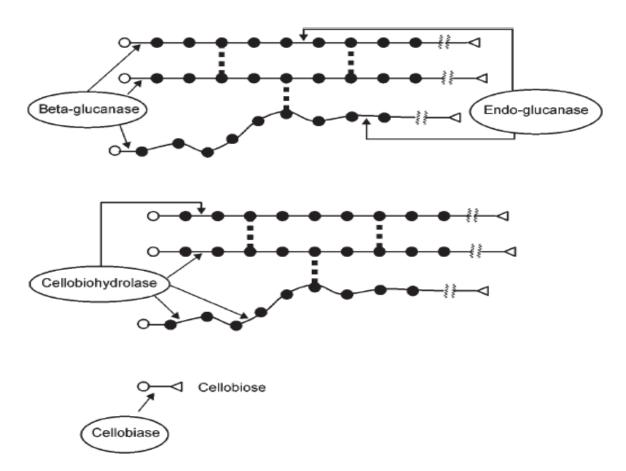


Fig. 12.4 Lock and key model of enzyme action.

### 12.3 Chemistry of Enzyme Finishing

More than with other chemical reactions, the enzyme catalysed hydrolysis of cellulose is strongly influenced by factors such as pH, temperature, time and agitation. The optimal pH for a particular cellulase depends upon its origin. Tricho-derma-based products (sometimes called 'acid cellulases') work best at pH 4.5–6, whereas cellulases from Humicola (often called 'neutral cellulases') are more effective at pH 6–6.5.6 The reaction temperature is also critical since at low temperatures, the reaction rate is slower than desired, but very high temperatures can deactivate the enzyme by providing enough energy to alter its molecular alignments and thereby destroy its catalytic ability. Since enzymes are true catalysts and are not consumed during the chemical reaction, the hydrolysis reaction will continue until either the reaction conditions change or the cellulose is physically removed from the reaction mixture. Mechanical agitation is important in order for the hydrolysis reaction to proceed efficiently. Recent work has demonstrated that the kinetics of the reaction are controlled by mass transfer effects. The adsorption–desorption mechanism of enzyme action depends on agitation to remove hydrolysis

by-products and expose new fibre areas to attack. Because the enzyme's catalytic action is not reduced during the reaction, an effective method of ending the hydrolysis must be employed to prevent excessive fibre loss. Since the molecule's physical alignments are crucial to its catalytic ability, procedures that alter the cellulase molecules



12.4 Action of cellulase components on cellulose.

internal structure can be used to deactivate the catalysis and halt the hydrolysis. High temperatures (> 70 °C or 160 °F for at least 20 min or short drying at 120 °C or 248 °F), high pH (>10) and high electrolyte content as well as enzyme poisons can serve to terminate the reaction by distorting the enzyme's molecular shape. Recent developments in enzyme manufacturing have led to commercial products that contain a preponderance of one cellulase component.6 These 'mono-component' enzymes are produced from modified Humicola strains and are primarily endo-glucanases active at pH 7–7.5 and are referred to as 'alkaline cellulases'.

#### 12.4 Evaluation of Bio-Finishing

The removal of cellulosic fibres to improve pilling performance, soften hand, and create an abraded appearance requires not only enzymatic hydrolysis but also mechanical action to carry away reaction by-products physically. The final results from the hydrolysis with respect to

appearance and hand are affected just as much by the mechanical components of the process as by the chemical components. Therefore, laboratory evaluations of bio-finished goods can only produce relative rather than absolute results. Enzyme suppliers determine the activity of their products by measuring the extent of the catalysed reaction under carefully controlled conditions. A standard test exists for amylases (AATCC Test Method 103), but the evaluation of cellulases is more complex and can vary from supplier to supplier. One common method is to measure the degradation of carboxy-methyl-cellulose solutions. Another is to determine the weight or strength loss of standard cotton fabrics under laboratory conditions where there is a correlation problem, because the mechanical conditions of the technical application are different to the laboratory ones. For example, the hydrolysis degree, HD, is determined by HD = (m0 - m)/m0 where m0 and m are the weight of the test material before and after bio-finishing.

#### 12.5 Denim Biowash

Indigo dye has poor substantivity for cellulose, hence it mostly remains at the surface of the fibre after dyeing and as such is called ring dyed. Such ring-dyed materials are subjected to treatment with stone or enzymes, which remove dyes randomly from the abraded portions of the fabric exposing white surfaces. This popular style is utilised in faded jeans. Microscopy reveals that for indigo dyeing, the extent of the penetration of the colourant into the cross-section of the cotton yarn depends on the pH of the bath. When the pH of the dye bath is decreased from 13 to 11, the yarn progressively becomes ring dyed. With increase in ring dyeing, more will be the colour yield and easier will be the wash-down process. The highest colour yield was observed within the pH range of 10.8–11.2.

Denim garments are made from warp-face cotton fabric, in which warp yarns are dyed with indigo dyes. These garments are subjected to a wash treatment to give them a worn look. In the traditional stonewashing process, the blue denim is faded by the abrasive action of pumice stones. Cellulases were first introduced in the 1980s, and nowadays more than 80% of denim finishers use cellulases or a combination of stones and cellulases to create the worn look on denim. Cellulases work by loosening the indigo dye on the denim in a process known as biostoning. This treatment can be applied to knit and woven cellulosic fabrics, such as cotton, viscose, linen and their respective blends. An enzymatic stonewash process requires equipment with sufficient shear forces and mixing, such as a drum washer.

The conventional denim wash process consists of treatment with pumice stones. About 1 kg stone is required for 1 kg of denim fabric. A large amount of pumice sludge is produced in this process. For example, a denim finisher processing 100,000 garments a week with stones typically generates 18 tons of sludge. This may block drains so it needs to be filtered out of wastewater. An environmental assessment was performed on jeans (OECD, 1998); the biostoning process produces sustainable textile wet processing very little sludge and is proved to be more environmentally friendly than the traditional stoning process using pumice. Often irreversible inactivation of the enzyme at the end of the application is beneficial, because ongoing enzymatic action may lead to undesired effects, such as extended tensile loss and over performance in the case of cellulase application. Inactivation of the enzyme can be easily realised by shifting the pH (above 10 for 5–10 min) and temperature (above 80°C) to extreme values for a relatively short period. More recently, some authors showed that laccase (with and without using a mediator) is an effective agent for the stonewashing effects of denim fabric.

# 12.6 Troubleshooting for Bio-Finishing

As mechanical agitation is important to effect the bio-finishing, only selected processes and machines can be used, for example tubular fabric preferably cut to open width and treated in open width washers. In the rope form the loosened fibre particles are filtered out by the fabric and cannot easily be removed. The pad-batch process, jig or package dyeing machines are not effective in bio-finishing. Not all cellulase enzymes give identical results, even with similar fabrics in similar equipment. Cellulases derived from Tricho-derma typically are the most aggressive in their action, whereas mono-component endo-glucanases often require the most mechanical action to achieve the desired effects. Slow deactivation of the cellulases during transport and storage can adversely affect the reproducibility of the resulting effects. If cotton is not washed carefully before bio-finishing, secondary fibre compounds as residual biocides can deactivate the cellulases. The same is true for natural or synthetic tannic acids, and resist or fastness improving agents for wool or nylon in cellulose fibre blends. Deactivation of cellulases after the desired effects have been achieved is very important. If the enzyme is not completely removed from the fabric, or is not effectively deactivated, the hydrolysis reaction will continue, although at a slower rate. As very large molecules, cellulases cannot diffuse into the crystalline parts of the cellulose fibres. They react on the fibre surface, so fibre damage takes time. But eventually enough hydrolysis will have taken place to weaken the affected fabrics or garments, leading to customer complaints and returns. Undesirable deactivation may be caused by high temperature and time, for example, caused by transport and storage and also

by enzyme poisons such as certain surfactants (especially cationic ones), formaldehyde-containing products or heavy metal ions. An activation effect on cellulases was reported by Nicolai and co-workers. Alkaline pre-treatment, low concentrations of selected non-ionic surfactants, poly-carboxylic acids and polyvinyl pyrrolidone can enhance the bio-finishing of cellulosics. The use of pH buffers during the hydrolysis reaction is strongly recommended, especially when abrading denim fabrics. Cellulase enzymes have very narrow pH ranges of effectiveness and denim fabrics can have significant quantities of residual alkali from the indigo dyeing process. Buffers are required to maintain the appropriate reaction conditions for maximum enzyme effectiveness. Because the effect of processing auxiliaries on cellulase catalysis is difficult to predict, it is important to evaluate any changes in processing formulas carefully by conducting small scale trials before making significant changes in production procedures.

### 12.7 Shrinkproofing for Wool

Conventional anti-felt finishing of wool is done by oxidation followed by resin treatment. The treatment causes environmental hazards by formation of adsorbable organic halides (AOX) due to the use of sodium hypochlorite, di-iso-cyanurate and polyamide epichloro-hydrin resin. It also creates a fish toxicity due to the cationic nature of the resin. Various finishing effects on wool can be obtained by the use of enzymes in more environmentally friendly ways than traditional chemical processes. However, the complexity of wool fibre makes it difficult to find enzymes that are able to modify some of the properties of wool efficiently, while not excessively damaging its structure. Due to the presence of scales on the surface of the wool fibres, frictional resistances on the two directions along the fibre axes are different. This causes fibres to entangle and shrink during agitation of the fibre mass. A process conventionally used or wool shrink proofing is chlorination. This process degrades the exocuticle of the wool, forming cysteic acid residues and protein losses. This process has been replaced by proteinase enzyme treatment due to their high specificity and much lower environmental impact. However, proteinase treatment leads to protein degradation, resulting in a deterioration of fibre strength and limited shrink resistance. Several reports show that increasing enzyme size by chemical cross-linking with glutar-aldehyde or by the attachment of synthetic polymers such as polyethylene glycol can reduce enzyme penetration and the consequent reduction of strength and weight loss. Some of these processes have been tested on an industrial process scale. Some researchers describe methods to improve the shrink resistance of wool by pre-treating it with a gentler oxidizing agent, such as H2O2, and then with protease enzyme. Others refer to

processes to achieve shrink resistance by treating wool with a protease followed by a heat treatment. By bleaching wool with hydrogen peroxide in the presence of iminodi-succinic acid sodium salt (IDAS), a new environmentally friendly chelating agent followed by treatment with lipoprotein lipase enzyme at 50°C and pH 7 for 1 h, it is possible to obtain machinewashable, pilling-resistant wool without a severe loss in the fibre strength. Some properties of wool, such as shrink proofing, anti-pilling and dye-ability (towards anionic and reactive dyes) are improved. The degree of whiteness and wettability of the pre-oxidised wool fabrics was enhanced. The treatment with a 1% enzyme (subtilisin serine alkaline protease) and 1.4% sodium sulphite applied for 30 min gives complete shrinkage control with strength retention and 3.71% weight loss. The screening for new protease-producing microorganisms with a high specificity for cuticles is being investigated as an alternative for the existing proteases. Papain is the best-known cysteine protease; it was isolated in 1879 from the fruits of Carica papaya. The optimal activity of papain occurs at pH 5.8–7.0 and at temperature 50–57°C, when casein is used as the substrate. A patent application about the use of laccase from T. versicolor plus a mediator to increase the shrink resistance of wool has been published. Also, Lantto et al. found that wool fibres can be activated with laccase if a suitable mediator is present. Therefore the use of laccase for anti-shrink treatment of wool seems very promising.

The effect of enzyme treatment (savinase, resinase, xylanase and pectinase) on the physical, chemical and structural properties of wool and specialty hair fibres was evaluated (Das and It was observed that xylanase and pectinase treatments had as good a cleaning efficiency as conventional soap scouring. Furthermore, at the concentrations used, neither of these two enzymes caused any physical damage to the fibres, as confirmed by the tenacity and diameter values and SEM pictures. The effectiveness of resinase as a scouring agent was, however, not very satisfactory. Because specialty hair fibres possess very few impurities compared with sheep wool, the milder treatment conditions would be very appropriate for the treatment of these fibres. The presence of any one of the three proteo-lytic enzymes in the dye bath increases the amount of dye absorbed in all the dyeing processes studied. The action of the enzymes on increasing the dye absorption becomes more evident when the dyeing temperature is lower, and this action is greatest when the temperature gets closer to that of the maximum activity of the enzyme.

### 12.8 Surface Modification of Synthetic Fibres

Synthetic fibres share common disadvantages, such as high hydrophobicity and crystallinity, that affect not only wearing comfort (making these fibres less suitable to be in contact with

human skin), but also the processing of fibres by impeding the application of finishing compounds and colouring agents. Classical methodologies to improve fibre hydro-philicity, such as alkaline or acid hydrolysis, lead to the deterioration of fibre properties, such as irreversible yellowing and loss of resistance. Recent studies show that surface modification and hydrolysis of polyester and polyamide with enzymes are environmentally benign methods. The major advantages of polymer modification with enzymes as compared to chemical methods are milder reaction conditions and highly specific non-destructive transformations, targeted to less fibre damage. Synthetic materials are generally considered resistant to biodegradation. However, fairly recently it has been established that enzymes are also able to act on synthetic materials. The advantage of enzymes over conventional techniques is that the favourable bulk properties of polyesters (PET) are not affected because the enzymes are too big to penetrate into the bulk phase of the material. Various research groups assessed the potential of laccases, lipases, poly-esterases (serine esterase) and cutinases in the oxidation or hydrolysis of polyester materials A poly-esterase was reported by Yoon et al. for the surface modification of PET and poly-trimethylene-terephthalate (PTT). The authors reported that the formation of terephthalic acid (a hydrolysis product) could be monitored at 240 nm. The enzymatic treatment resulted in significant de-pilling, efficient de-sizing, increased hydrophilicity and reactivity with cationic dyes and improved oily stain release. Recently, Nechwatal et al. tested several commercial lipases/esterases for their ability to hydrolyse oligomers formed during the manufacture of PET, which may otherwise create problems after dyeing by deposition on machines and fibres. Cutinase, lipases and poly-esterases increase hydrophilicity by actual hydrolysis of PET, whereas laccases oxidise the polyester surface. Cutinases (EC 3.1.1.74) are serine hydrolases that can accept a wide range of substrates such as poly (ethylene terephthalate) and polyamide. Crystallinity greatly affects the capability of cutinase to hydrolyse the ester bonds. Cutinase displays relatively high activity towards amorphous polyester and little activity on highly crystalline substrates. The structure and properties of cutinases are well described by Carvalho et al. In contrast to lipases, cutinases do not require interfacial activation; the active site is accessible because it does not have a lid; the oxyanion hole is preformed but considerably flexible in solution. Cutinases seem to have a large potential in the enzymatic surface modification of polyester. The optimum pH and temperature for cutinase from Fusarium solani pisi are 8-8.5 and 25°C; above 35°C the activity decreases rapidly. Novoenzymes improved temperature stability to as high as 65-80°C. Cutinase or lipase treatment does not result in pitting corrosion, as in the case of alkaline treatment; surface treatment is more or less

homogeneous. However, cutinase strongly adsorbs to the polyester surface. The adsorbed enzyme can be removed by using proteases or using a thorough washing and extraction method. Due to poor hydrophilicity, textile materials made from polyamide 6, 6 are also uncomfortable to wear. It leads to static cling and stain retention during laundering. The fibre is unsuitable for specific finishing treatments, such as the coupling of flame-retardant or covalent immobilisation of proteins. Coupling agents are substances that are used in small quantities to treat a surface so that bonding occurs between it and other surfaces. Immobilisation is the binding interactions of target biomolecules in liquid samples with specific chemical groups or macromolecules that are immobilised on a solid material. Bio-catalytic processes have been developed to modify polyamide surfaces enhancing hydrophilicity. Enzymes that are able to hydrolyse polyamide surfaces are proteases, amidases and cutinase. The hydrolysis of polyamide is based on the breakage of the amide linkages of the polymer surface, resulting in amino and carboxylic groups yielding. In addition to hydrolytic enzymes, oxidases from lingolytic fungi have been shown to depolymerise polyamides. Nylon-degrading peroxidases attack methylene groups adjacent to the nitrogen atoms, and so the reaction then proceeds in an autooxidative manner. A study by Parvinzadeh confirms the structural changes of nylon 6 fibres using Subtilisins protease by measuring the dyeability, hydrophilicity, chemical changes and fastness properties. The enhancement of the hydrophilicity of synthetic polymers such as polyester and polyamide is a key requirement for many applications ranging from electronics to functional and technical textiles. The new functionalised fibres can have a totally new range of applications, such as filter media, smart, technical and high-performance materials. Future challenges are in the area of improved activity and better temperature stability. Despite some achievement the potential benefits of enzymatic modification of synthetic fibres are far from being fully explored. For acrylic and cellulose acetate fibres, enzymes can be used to accomplish the formation of reactive and/or hydrophilic groups at the surface by hydrolysis of their pendant groups without affecting, in theory, the integrity of the main chain of the polymer. The pendant group in poly-acrylonitrile (PAN) is the nitrile group. The ester group of the polysaccharide substituent is a potential supplier to hydrolyse the cellulose acetate fibres in a controlled manner, creating hydroxyl groups at the surface. The modification of PAN and cellulose acetate with enzymes results in two types of products: soluble compounds and new chemical groups attached to insoluble fibre substrate. For PAN fibres, only nitrilase and amidase generate a soluble product, ammonia; nitrile hydratases generate amide groups as new side chains of the PAN main chain. For cellulose acetate, the hydrolysis of its side chains

releases acetic acid to the reaction media, and the hydroxyl group is located on the polymer backbone. The surface of PAN was modified by nitrile hydratase and amidase enzymes obtained from different sources (Rhodococcus rhodochrous and Agrobacterium tumefaciens). After enzymatic treatment the fabric became more hydrophilic and the adsorption of dye was enhanced. Although there is no industrial application yet, the research results demonstrate that the enzymatic treatment of PAN would give advantages in the quality of treated fibres, as well as in energy conservation and pollution control. Despite different substrates, origins and the amount of enzyme used, it is possible to specifically modify the nitriles of PAN into amides or carboxylic groups at moderate temperature and pH conditions with distinct chemical properties. Several aspects, including staining properties and hydrophilicity, are clearly improved for PAN fibres. Owing to its excellent mechanical properties, stability and low cost, the modified PAN is also of interest for filters in reverse osmosis, gas separation, protein immobilisation, ion exchange, ultrafiltration and dialysis.

# 12.9 Use of Biopolymers

There are some seldom-used cyclic oligosaccharides as well, but because of their cost, they are not applicable to industrial applications. Cyclodextrins (CDs) are cyclic oligomers of  $\alpha$ -d-gluco-pyranose that can be produced with the transformation of starch by certain bacteria, such as Bacillus macerans. CDs can act as hosts and form inclusion compounds with various small molecules. Such complexes can be formed in solutions in a solid state as well as when CDs are linked to various surfaces, where they can act as permanent or temporary hosts for small molecules that provide certain desirable attributes. This characteristic makes CD a promising reagent in textile finishing. The practically important, industrially produced CDs are the  $\alpha$ ,  $\beta$  and  $\gamma$  CDs. In the textile field, CDs can have many applications such as:

- (1) Absorption of unpleasant odours.
- (2) Formation of complex and release of fragrances, 'skincare-active' and bioactive substances.

Further, various textile materials treated with CDs could be used as selective filters for the adsorption of small pollutants from wastewater. After the discovery of CDs, scientists considered them poisonous substances, and their ability for complexes formation was only considered a scientific curiosity. Later on, research on CDs proved that they are not only nontoxic, but they can be helpful for protecting flavours, vitamins and natural colours (Astray et al., 2009). CDs already play a significant role in the textile industry and can be used in the

dyeing, surface modification, encapsulation, washing and preparation of polymers and in fibre spinning. In the textile field, CDs may have many applications such as:

- Absorb unpleasant odours.
- They can form complex and release fragrances or 'skin care-active' substances like vitamins, caffeine and menthol as well as bioactive substances such as biocides and insecticides.
- Textile materials treated with CDs could be used as selective filters for the adsorption of small pollutants from wastewaters—'preparation of textile nano-sponges'.

One of the new concepts for the modification of textile substrates is based on the permanent fixation of supra-molecular compounds on the material's surface, which thus imparts new functionality to the fabric. CDs are among the more promising supra-molecular moieties applied to textiles. The covalent bonding of CDs onto textile fibres was first patented in 1980 by Szejtli and colleagues and reported to bond CD via the cross-linking reagent epichlor-hydrin onto alkali-swollen cellulose fibres. They found out that CD covalently bonded to cellulose retained the ability to form complexes. When cellulose was treated with a drug, it complexed with CD. The complexed drug releases upon contact with the skin. Cellulose textile substrate containing covalently bound  $\beta$ -CD was treated with solution of iodine, potassium iodide and methanol as a solvent to prepare a medical bandage.

Szejtli published a very extensive review about CDs in the textile industry. In his review, he divided the application of CDs in textile sectors in the following areas:

- (1) Binding of CDs to fibre surfaces.
- (2) In textile dyeing.
- (3) In textile finishing.
- (4) In detergents.
- (5) Miscellaneous applications of CDs in textile industry and textile care.

The attachment of CD molecules on textile substrates provides hosting cavities that can include a large variety of guest molecules for specific functionality. There are two possible approaches to bond CDs onto textile fibres, such as chemical bonding of modified CDs on the fibre surfaces or to use bifunctional reagents to link CDs covalently on fibre surfaces. A very effective bonding of CDs on cellulose fibres can be achieved by a high performance resin finish or with

non-formaldehyde reagents such as poly-carboxylic acids, which can covalently esterify hydroxyl groups of cellulose and CDs and therefore the grafting of CDs on cellulose can occur. An insect repellent is a substance applied to skin, clothing or other surfaces which discourages insects from landing or climbing on that surface. Synthetic repellents (such as para-dichlorobenzene) tend to be more effective than 'natural' repellents, but on the other hand, they are usually toxic. Cedar oil is often used as a natural insect repellent or for its aromatic properties, especially in aromatherapy. Essential oil repellents tend to be short-lived in their effectiveness due to their volatile nature. To prevent the essential oils from evaporating form the textile materials, we can encapsulate them in  $\beta$ -CD.

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# **Glossary of Textile Finishing**

ABSORBENCY -Wettability, hydrophilising.

ABSORPTION- The ability of a material to take another substance into itself.

ABSORPTIVE- An absorptive substance is one which takes up liquid.

ACETIC ACID- 80 %-CH3COOH-Medium strength, volatile acid; used to adjust the pH. AFFINITY-Refers to a force with which a dye or an auxiliary binds to the substrate

AGGLOMERATE-Accumulation, conglomeration of molecules, ions, dyes, soil particles. AIR-FREE STEAMER-In steaming processes with reductive chemicals, the steamer must be free from air. As a rule of thumb, a quantity of up to 0.3 % air content in the steamer does not impair the production results.

ALKALI SPOTS- Spots which are mostly brightened in the middle becoming gradually stronger in colour toward the edges and then appear shapeless in the dyed fabric. The places on the fabric that are affected are mostly harder and are dyed a deeper shade.

ALUMINIUM SULPHATE- Al2 (SO4)3 · 18H2O- Aqueous solutions react in a strongly acidic manner to coagulating alginate thickeners.

AMMONIA SOLUTION- NH4OH Weak, volatile base. Used to adjust the pH.

AMMONIUM SULPHATE- (NH4)2SO4- Acid donor; aqueous solutions have about pH 5. Upon boiling, ammonia escapes and sulphuric acid is released causing the pH to sink further. ANTI-MIGRANT- Auxiliary used to influence, inhibit or block undesired dye migration, whether physically (e.g. drying) or chemically caused by the products used.

BARRINESS- Also called barré, barriness or horizontal stripes in knitwear/woven fabrics of viscose, polyamide or polyester.

BATH EXHAUSTION- Concentration difference in the dye bath before and after dyeing as determined by colorimetric measurement, given in % of the original liquor concentration. The bath exhaustion is dependent on the dye build-up in each case.

BLEACHING- Chemical treatment of textiles with appropriate bleaching agents to brighten or remove colour tones of natural fibres due to growth or of man-made fibres due to how they were manufactured.

BOOSTER- Device to apply a blank vat for the continuous method. The booster is located in the steamer.

BRONZING- A defect which shows up as a metallic-glittering appearance and is associated with poor rubbing fastness properties. Causes: inadequate pre-treatment (oil residues, etc.), dye precipitations, dye oversaturation on the fibre, and premature oxidation in sulphur and vat dyeings.

BUILD-UP- Colour yield of an individual dye in the fibre, related to the amount of dyes applied.

CATALYTIC FADING- Mutual negative influence on the light fastness of dye combinations. CAUSTIC SODA (LYE)- Sodium hydroxide (NaOH).

CAVITATION- Cavitation is the formation and dissolution of cavities in liquids due to changes of pressure.

CENTRIFUGE- (Extractor, hydro-extractor, whizzer); used to remove water (pre-drying) from textile materials.

COAGULATION- Also flocculation of colloidally loose particles by agglomeration, flocking, lumping and sinking to the bottom as an insoluble gel. Occurs by heating or by adding electrolytes or opposite- charged colloids.

COMMON SALT- Sodium chloride (NaCl).

COMPLEX FORMER- (Also called chelating or complexing agents).

Products which through functional groups are capable of complexing metal ions and thus improving their solubility in a certain milieu.

CONDENSATE- Condensation of evaporated water from steam pipes and tanks, to be regarded as soft water and is usually conveyed back to the steam boiler in the production plant. CONDITIONING- I. Adaptation to defined conditions, such as a standard climate of 20 °C  $\pm$  2 °C and 65 %  $\pm$  2 % relative humidity.

II. Term used for determining the moisture content of textile grey goods in conditioning machines.

COUNTER-FLOW- Washing liquors or drying air flow against the direction of the moving fabric.

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COUNTER-FLOW- Washing liquors or drying air flow against the direction of the moving fabric.

CREASE PREVENTING/INHIBITING AGENT -Auxiliary for preventing running creases, especially when fabric is treated in rope form.

CREASE RECOVERY ANGLE -Serves as magnitude for measuring the effect of crease resistant finishes.

CREASE RESISTANCE- Measure of the resistance of textiles to creasing

CREASE-RESISTANT FINISH- Resin finishing of cellulose to reduce creasing to a minimum. CREASING- The sensitivity of fabrics to constant bending, deflection, flexing, pressing or crushing in a crosswise direction to the warp and weft threads.

CROCKMETER- American standard device for testing the rubbing fastness of dyed textiles. DIAMMONIUM PHOSPHATE- (NH4)2HPO4-Aqueous solutions are slightly alkaline. Upon heating, ammonia escapes and the pH value sinks. Acid donor (catalyst) for crosslinking pigment binders/fixatives. Ensures optimum fastness properties.

DICHROMATIC- Two-coloured.

DIFFUSION- Progressive movement of molecules of a substance by natural self-motion. A dissolved substance migrates from places with higher concentration to places with lower concentration until a concentration equilibrium has occurred within the system. This process is accelerated by raising the temperature.

DIN -Deutsche Industrie Norm (German Industrial Standard).

DIOXIN- Collective term for 75 chemically related substances which differ widely in their toxicity. Close chemical relationship with dibenzo-furans (group of 135 substances). Both have a skeletal structure of carbon, hydrogen, oxygen and chlorine atoms. Dioxins have a carcinogenic effect.

DISPERSING AGENT-Dispersing agents act as distributing agents in aqueous treatment baths. They counteract the separation of poorly soluble substances and are mostly surfaceactive compounds.

ELECTROLYTE- Substances that break down into ions upon dissolving in water. This makes the solvent conductive for an electrical current. The most important electrolyte groups are acids, bases and salts. Electrolytes are often used to reduce (cut) dyes and textile auxiliaries. Electrolytes increase the substantivity of the dye to the fibre.

ENDOTHERMIC- Heat-absorbing, opposite of Exothermic.

FLASH AGER- Energy and space-saving rapid steamer consisting of a liquor application device, an IR zone for precipitate-free heating of the goods, a steamer and a steam super heater with the steam directed on the counter flow principle, a water lock to prevent air from entering, and a ceiling heater against the formation of droplets. Steaming times are 5-30 s, fabric drawing- in length 4-10 m, steam consumption 100-200 kg/h. especially recommended for reactive and vat dyes, causticizing of regenerated cellulose fibres and the continuous alkalisation of PES.

FORMALDEHYDE 20 % -CH2O- Slightly water-soluble, pungent-smelling gas. Has a reductive and strongly antiseptic effect. Preserving agent.

FORMIC ACID- HCOOH- Strong, volatile acid. Used to adjust the pH.

GLAUBER SALT- Sodium sulphate (Na2SO4); electrolyte.

GLUCOSE- In the form of a fine white powder, crystals, or a thick colourless syrup, with the formula C6H12O6; strongly hygroscopic; readily soluble in water; exhibits a neutral reaction. A differentiation is made between a and b; b-glucose molecules are the basis for cellulose. Glucose is used in vat dyeing to protect against the over-reduction of blue dyes (of the RS; BNsp. type) above 80 °C.

GLYCERIN- C3H5 (OH) 3- Solvent for dyes, hygroscopics.

GREEN DEGRADATION- Combination dyeings with yellow, green and blue dyes when exposed to light and weathering, can behave more unfavourably than expected based on the light fastness of the individual dyes of the combination.

HANK, ROPE

- a) Hank = yarn form
- b) Rope = piece goods form (fabric gathered in the warp or stitch wale direction, in the form of free fabric transport in pre-treatment and dyeing processes).

HORIZONTAL PAD DYER- Pad dyer in which the goods are padded in the nip between the rollers without a trough.

HOT FLUE- Hot air drying machine for fabric or for intermediate drying. The passage of the goods is mostly either vertically up and down in the hot air chamber or slanted back and forth with double-sided ventilation nozzles from the top and the bottom.

HYDROCELLULOSE- Water absorption or the rupture of the oxygen bridge between the glucose structural elements causes mineral acid-damaged cellulose. Properties: weakened strength, strong reduction power, changed colour affinity.

HYGROSCOPICS- Water-attracting products added to finishes, printing pastes, and sizes to level out air humidity and fibre moisture as well as influence the weight and handle of the goods.

IMMERSION LENGTH- Impregnation time for uniform wetting of the goods between dipping and squeezing off.

IMMERSION SQUEEZING-In order to intensify the liquor exchange in wet-on-wet processes or the wetting in dry on-wet processing at high production speeds, additional immersion squeezers are used.

IMPREGNATION-Lat.: Drenching, soaking; the treatment of textiles with solutions, dispersions, emulsions. For all continuous and semi-continuous processes. Impregnation is understood as the passage of a fabric through an impregnation solution, followed by uniform squeezing over the entire length and width of the goods.

INFRARED (IR) - IR radiation which is not observed by the human eye in the long wave region. The wavelengths lie between 780 - 1,000,000 nm. Only a small portion of the 780 - 3000 nm of the vibration spectrum is of practical interest. The transition range is between 700 - 860 nm. All bodies give off IR radiations, and the warmer the body, the more intense the radiation and the shorter the wavelength will be. IR radiation is also termed thermal radiation.

IR DRYING-Drying technology preferentially uses the invisible IR radiation of the electromagnetic wave radiation spectrum. The wave range which is most important from the technical drying standpoint lies between 700 - 3000 nm, where particles are set in more rapid vibrations which warms the goods.

JIGGER-Short-liquor dyeing machine where the fabric, wound around a lap roller, is rewound on a rewinding roller and, during the rewinding process, is led through a treatment bath. LIQUOR THROUGHPUT- Relation of liquor/amount of goods/time.

LOOSE STOCK- Fine, light, loose assembly of smooth or crimped individual fibres (naturally grown, cut or broken up). Mostly in a staple length of 30 - 150 mm.

LP- Abbreviation of Liquor pick-up, given as a percentage of the increase in weight after the padding process related to the dry weight.

MAGNESIA HARDNESS- (MgH), unit per degree of German Hardness of magnesium oxide (MgO) 7.15 mg/l water.

MAGNESIUM CHLORIDE-MgCl2 Strongly hygroscopic; used as a catalyst in resin finishing.

MARSEILLE SOAP- Soap made of unsaturated fatty acid with a low cloud point. For this reason, it is suitable for washing at low temperatures  $(20 - 30 \, ^{\circ}\text{C})$ .

MERCERISATION IN THE GREY- Mercerisation without previous kier boiling, boiling off, or any other wet treatment (dry mercerisation).

MERCERISING- Treatment of cotton yarns, woven fabrics or knitwear with cold, strong caustic soda solution under tension. The process is based on the observation that under the influence of alkaline solution, cotton swells/shrinks, increases in density and strength and shows an increased dye affinity.

METAMERISM- A pair of colour patterns can show the same tristimulus value under a given illuminant, i.e. the same colour appearance even though the spectral curves are different.

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METAMERISM- A pair of colour patterns can show the same tristimulus value under a given illuminant, i.e. the same colour appearance even though the spectral curves are different. MIGRATION-The capacity of a substance in or on the substrate to migrate from places of higher concentration to places of lower concentration. Migration examples: levelling-out during the dyeing process, uneven drying in padding processes, on contact of dyeings or prints with substrates which have an affinity to the dyed substrate (e.g. disperse dyeing in a PVC coating).

MIGRATION PREVENTER- Substance added to the padding liquor to prevent migration of the dyes on drying

(MIGRASOL SAP). MOIRÉ EFFECTS- Caused by the interference of two linear systems. Carried out mostly with lightly ribbed fabric with imprinted (lustrous) pattern effects in taffeta, brocade or twill. Besides these desired moiré effects there is also undesired moiré which is considered a defect.

MOISTURE ABSORPTION- Approximate mean moisture values in % at 65 % relative humidity and 20 °C.

MULTIFIBRE- Mixed fibre stripes mainly consisting of CV, PAC, PES, PA, CO, CTA or WO, PAC, PES, PA, CO, CA. Used for wet fastness tests to prove staining behaviour.

NEPS- Cotton defect in the form of felted-together fibre knots, as growth neps (husk residues, immature and dead cotton, wetness results) or processing neps (torn-apart and twisted together, mainly mature fibres which develop on the carding machine).

NEUTRALISATION- Mutual saturation of acid and base to neutral reaction pH = 7 (levelling of H and OH ion concentration). The result is a neutral salt. This is however not the essential feature, which is the H and OH ion levelling "to water".

NIP PAD DYER- Horizontal 2-bowl pad dyer, where the fabric enters from above and the padding liquor is held between two squeezing rollers (in the nip).

NONIONIC- Forming no ions

NONIONIC SURFACE-ACTIVE SUBSTANCES- Non-ionic surfactants, surface-active substances which do not form ions in an aqueous solution.

ÖKO (ECO) LABEL- An eco-label with substantiated, verifiable criteria conveys motivation for ecological optimisation of textile goods in the industry, security to the trade, and trust in the purchased textile to the consumer.

OVER-DRYING- Excessive drying of a fibre material so that even the capillary water component is removed. Heat and over-drying damage all textiles to a greater or lesser extent. This can result in thermal splitting of polymer chains.

OVER-REDUCTION- An undesired property of certain vat dyes with the use of an excessively high dyeing temperature, too high an amount of sodium dithionite, too long dyeing time, or a combination of these factors. Especially sensitive are blue dyes of the BEZATHREN RS type. Over reduction leads to loss of strength, duller shades and in some cases to lower chlorine fastness. Over-reduction is irreversible.

OXIDATION- Oxidation is a chemical reaction. The substance to be oxidised releases electrons to the oxidation agent. Opposite of: Reduction.

OXIDATION AGENT- Cause oxidation of the reacting agent by means of electron uptake. Use of oxidation agents: during oxidation bleaching, as oxidative starch hydrolysing products, for oxidation/ after-soaping of fast dyes (vat, sulphur, naphthol).

OXYCELLULOSE- Oxidation-damaged cellulose (degraded to a greater or lesser extent though oxidative acids, bleaching agents); properties similar to hydrocellulose and photocellulose; proof: oxy-cellulose alone with Berlin blue, Fehling's solution, methylene blue and vatting test – like hydrocellulose.

PAD DYER- The purpose of the pad dyer is to remove the excess liquor uniformly taken up by the fabric from the dyeing trough along the entire width. A differentiation is made between a trough with squeezing rollers and a trough-less high performance (nip) pad dyer.

PAD JIGGER PROCESS- Piece goods are padded with dye solutions and developed on the jigger. This method has been developed above all for dyeing cotton with vat dyes. It ensures good dye penetration. Since a portion of the dye is converted into the blank vat, the discontinuous vatting in the full bath of the jigger can result in tailing. This defect can be prevented by adding padding liquor to the bath at the beginning and end of the first passage.

PADDING- Impregnation of piece goods on the padder with dyes or chemicals.

PARTICLE SIZE- Refers to dye particles; important especially with water-insoluble dyes.

PASSAGE- Also called end or run; one complete revolution of the goods through the jigger. PENETRATION- Penetration of a fibre structure by the applied liquor, which must wet all the surfaces as much as possible, so that the levelness of the product application is ensured before the fixation treatment.

PERFORATION- Systematic perforation of materials to allow substances to pass through them. Thus dyeing bobbins or dye beams are perforated so that the dye liquor can enter into a liquor exchange with the goods to be dyed.

PEROXIDE- (Superoxide); compounds of the hydrogen peroxide, sodium peroxide and magnesium peroxide type; oxygen-releasing principle; oxidation agent (hydrogen peroxide is often used in peroxide bleaching, which has advantages over other bleaching variants from the technical application and ecological standpoint); the activation of the peroxide bleach with hydrogen peroxide is carried out with alkali. Sodium hypochlorite (NaOCl) or sodium chlorite (NaClO2) exhibit serious ecological disadvantages in terms of exhaust air pollution by developing toxic gases or waste water pollution

(AOX). PERSPIROMETER- Device for testing wet general use fastness properties (perspiration and water fastness). The test specimen is placed between glass or acrylic resin plates and then placed in the device and treated under defined pressure at the prescribed temperature for the prescribed time.

PHOSPHORIC ACID H3PO4- Medium-strength acid; used as a catalyst in resin finishing. PICK-UP Liquor pick-up (LP) in %.

PIGMENT- Practically insoluble, solid, finely dispersed colorant which in fine dispersion produces a coloured appearance; insoluble dyes are called pigments.

POTASSIUM CARBONATE K2CO3- Aqueous solutions are strongly alkaline. Strongly hygroscopic. Used to adjust the pH.

PRE-DRYING- By a mild drying treatment, migration in the main drier is prevented. Predrying in the IR zone to 25 - 30 % residual moisture.

PRE-TREATMENT- All processes for improving the wetting and absorbency, dye uptake capacity, the cleanness of the textile material, increasing the degree of whiteness and for improving fabric winding, i.e. the relaxing and structuring properties. At the same time, tension and material irregularities from previous weaving and knitting processes must be levelled out.

REDOX POTENTIAL- (r H) "Redox" is the abbreviation of Reduction Oxidation. The redox potential is a measure for rating reduction and oxidation processes in solutions where the electric potential is measured in volts/millivolts.

REDUCTION- Reduction is a chemical reaction. The compound which is reduced accepts electrons from the reducing agent. Opposite of: oxidation.

REDUCTION AGENT- Causes by donation of electrons a reduction of the reaction partner. Reduction agents must reduce both the dye in vat dyeings and also remove the oxygen present in the dye bath and subsequently released during the dyeing.

RESIDUAL MOISTURE CONTENT- The residual moisture content is an important factor in continuous dyeing. After the fabric leaves the pad dyer, the padding liquor is evenly distributed over the goods.

RESIN FINISHING- Imparts additional properties depending on the requirement profile. Finish to increase the general use properties. Thanks to resin finishing, especially with textiles made of natural cellulose, there is a greater or lesser reduction of the abrasion, tensile strength and tear propagation fastness.

RETARDING- Slowing, braking, decelerating; e.g. of the dye uptake rate, which with all dyes involved is carried out in the sense of a levelling process. This however can have a selective effect on certain dyes which can then interfere with the levelling of such dye combinations. Excessively high application amounts of levelling agents can impede the dye in the dye bath. ROLLER HARDNESS- With rubber or plastic rollers for finishing machines, among other things the hardness/ softness is designated according to different evaluation scales.

SEMI-CONTINUOUS PROCESSES- A finishing process that combines a continuous processing step (e.g. padding) and a discontinuous one (e.g. development on the jigger).

SEMI-PIGMENTATION PROCESS- Principle: The dyeing liquor consists of finely dispersed dye, alkali and hydro sulphite, is prepared at a cold temperature, and is then heated to vatting temperature.

SEQUESTERING AGENT- Designation of chemical compounds which take multivalent metal ions out of water and bind them into an anionic (chelate complex, e.g. polyphosphates, ethylene-diamine-tetra-acetic acid [EDTA]). For ecological reasons, inorganic polyphosphates are now rarely used. Certain types, thanks to their dispersing effect, increase the anti-redeposition power of surfactant liquors thus counteracting possible greying and hardening of the textiles due to the re-deposition of soil.

SODIUM CARBONATE- Na2CO3 Aqueous solutions are strongly alkaline; used to adjust the pH.

SODIUM DITHIONITE- (Sodium hydrosulphite, sodium hypo-disulphite); Na2S2O4; white, anhydrous powder; only completely dry and cold storage-stable; well water-soluble; solution readily decomposes to sodium sulphite and sodium sulphate; strongly reductive; alkaline solution strongly attracts oxygen; used as reduction agent in vat dyeing; also known as hydrosulphite.

SODIUM HYDROXIDE NaOH- (caustic soda, soda lye, caustic soda solution) Hygroscopic; draws CO2 from the air (forming sodium carbonate); water-soluble; strong formation of heat on dissolving (exothermal).

SODIUM NITRITE NaNO2- (Nitrite, nitrous sodium) weakly hygroscopic; readily water-soluble; used to prevent over-reduction with vat dyes between 60 - 80 °C.

SODIUM PERSULPHATE- Na2S2O8 Oxidation agent.

SODIUM SILICATE- Mixture of Na2SiO3 and Na2Si2O5. Stabilizer in peroxide bleaching.

SODIUM SULPHATE- Na2SO4 Glauber salt.

SODIUM TETRABORATE- Na2B4O7 · 10H2O (Borax) Aqueous solutions are alkaline. Coagulation of guar ethers thickeners.

SOIL-RELEASE FINISH- Application of certain finishing processes to achieve better washing-off of soil and spots. The chemical basis of well-known soil-release products are:

• Silicon compounds • Carboxy-methyl-celluloses • Ethoxylated compounds • Poly-glycol esters of terephthalic acid • Acrylic acid polymers • Fluoro-chemicals

SOLUTION- A true solution is the uniform distribution of one substance in another (through diffusion) or, according to Pauling, a phase consisting of several particles which cannot be converted into each other easily. The main component of a solution is the solvent, whereas the other components are designated dissolved substances.

SPECKS- Poorly dissolved or inadequately dispersed dye, but precipitations based on inadequate product compatibility can also form deposits on the goods called specks.

STAINING- Soiling of white adjacent fibres in fastness tests or of adjacent white or coloured fibres during wet finishing processes.

STANDARD CLIMATE- In DIN EN ISO 139, the standard climate for textile testing has been defined as 65  $\% \pm 2$  % relative humidity and 20 °C  $\pm 2$  °C room temperature.

SUBSTANTIVITY- The expression of the absorbing (exhausting) power of a dye or auxiliary, i.e. its ability to be applied to a textile as a liquid medium. However, the expression for the force with which a dye/auxiliary is held by the substrate is designated as the affinity.

SUPERHEATED STEAM- This is 100 % steam whose temperature is above that of saturated steam. This steam is not saturated.

SWIMMING ROLLER- (S-roller); construction of a roller which floats on a cushion of compressed oil.

TENSILE STRENGTH- (Strength, tenacity, breaking strength, limit strength, tear strength); resistance of a material to tensile stress; the tensile strength is a fineness-related tensile force. In the SI system, the tensile strength corresponds to the measured tensile strength in Newtons divided by the fibre fineness in Tex.

TEX SYSTEM- For designation of the linear mass (see fineness designation) of textile fibres, intermediate products, yarns and twisted yarns. 1 Tex is the fineness at which a fibre/yarn of 1 km length weighs 1 g (1 Tex = 1 g/km).

THERMOMIGRATION- Desorption as the migration of dyes, especially disperse dyes on PES, under the influence of dry heat in the presence of surfactants. In thermal after-treatments of dyed polyester fibres (drying, post-fixation, finishing), the disperse dye can migrate to the surface. Thermo-migrated disperse dyes are no longer fixed and therefore reduce the fastness level.

THERMOSOLING- Treatment which, by means of hot air, superheated steam, contact heat or radiation, causes the dye to diffuse into the interior of the fibre. With thermosoling, it is important that the fabric is exposed to a constant temperature over the entire width as temperature fluctuations can lead to shade differences.

TROUGH- Dye liquor tank in the pad dyer, liquor tanks in sizing and finishing machines. UREA NH2CONH2 Can improve the solubility of dyes. Prevents the clogging/drying of the screen in the pigment printing system and reduces the formaldehyde content on the goods (measured according to Law 112).

WASHING- In washing processes, unwanted substances are washed off from the substrate by dissolving or dispersing them in water. A wash process is a combination of the four following physical-chemical factors:

• Chemistry • Mechanics • Temperature • Time

WASHING OFF- The removal of softening agents, lubricant preparations, sizes, chemicals, textile auxiliaries, unfixed dyes and finishing agents. The washing-off process greatly influences water consumption, costs, waste water problems and fastness levels.

WATER HARDNESS- Water hardness is the amount of calcium and magnesium ions (hard water salts) in water. These are capable of forming undesired bonds with textile auxiliaries and dyes. By 1°dH (1 degree of German hardness) is understood that 10 mg calcium oxide are present in 1 litre of water. This stoichiometry serves as the conversion base for other hard water salts. Hard water can be determined with the aid of colour indicators.

WATER LOCK- Device at the outlet of pre-treatment and dyeing steamers to ensure an airfree steam atmosphere. The outlet support often extends into the liquor of the first aftertreatment bath. WET FASTNESS- A collective term used mostly for general use fastness to water, perspiration, washing and wet ironing of prints and dyeings which, with higher requirements, also include alkali, over-dyeing, sea water and wet light fastness properties.

WETTING AGENTS- Hydrophobic fibre surfaces and entrapped air prevent the rapid wetting of fibres/textiles with water when the textile goods enter aqueous treatment baths. Wetting agents as mediators between incompatible phases are chemically structured so that they contain hydrophobic and hydrophilic components in the same molecule.

WETTING POWER- Degree of capacity for wetting, based on the capillary activity of wetting agents.

XENON HIGH-PRESSURE LAMP- (Inert gas high-pressure discharge lamp, ultraviolet lamp); its spectral radiation density distribution largely corresponds to that of a cloudy sky with a high UV component. Used for Light fastness test ISO 105-B02.