

5.1 Introduction

A very important concept in the textile operations is that fibre, as the fundamental component of textiles, should be protected as much as possible during processing. Starting before spinning and progressing through fabric formation, fibres are exposed to many harsh processing environments including physical (temperature, friction, tension and compression), chemical (pH, inorganic and organic compounds) and biological (bacteria and enzyme) variables. Fibre finishing is usually the first step in textile processes where chemicals are used to give fibres protective and functional properties. For instance, sizing agents are added to fibre assembly-yarn to facilitate the weaving operation, lubricating agents are needed to smooth the yarn spinning process and different additives are used to improve the spinning, yarn formation and fabric formation.

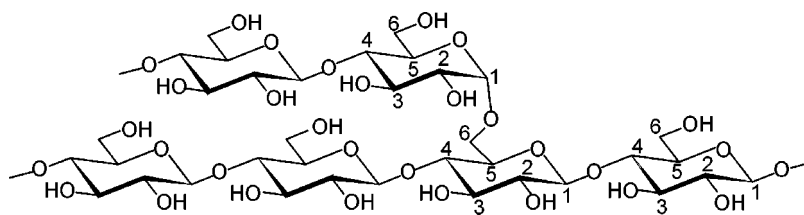
5.2 Sizing agents

As mentioned briefly above, sizing agents are added to the yarns that are used in the weaving operation. More specifically, sizing agents are added only to warp yarns, not to filling yarns. This is because the warp yarns are under high tension and constant friction during the weaving operation. Without sizing, the warp yarn cannot be used for weaving; it would be broken easily and quickly, causing the weaving machine to stop and result in very low and even no productivity for the weaving operation. The sizing agents can give warp yarns extra strength to withstand the high tension and a smooth surface to reduce friction.

5.2.1 Natural sizing agents

Starch and its derivatives

Starch is a natural polymer from the group called polysaccharides which has multi-anhydroglucose units. The chemical formula of starch is $(C_6H_{10}O_5)_n$. Starch has chemically two moieties, an amylose part which consists of anhydroglucopyranose



5.1 Chemical structure of amylopectin.

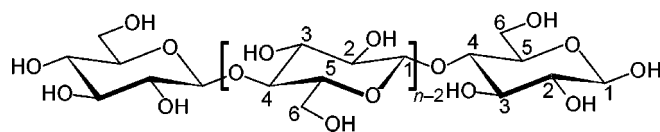
units joined by α -D 1,4 glycosidic bonds and an amylopectin part which consists of anhydroglucopyranose units linked by α -D 1, 6 glycosidic bonds as shown in Fig. 5.1. Amylose has a linear chain and amylopectin has a branched chain.

Starch can be decomposed by pyrolysis and acidic hydrolysis. British gums and dextrans are products derived from starch. They have a lower molecular weight and better water solubility. A qualitative test of starch and its derivative can be conducted using an aqueous solution containing $1.4 \text{ g l}^{-1} \text{ I}_2$ and $2.4 \text{ g l}^{-1} \text{ KI}$.¹ The existence of starch (specifically amylose) will turn the yellow-orange colour of the solution to a dark blue colour. When tested with I_2/KI solution, British gums and completely degraded dextrans give a reddish brown colour, partially degraded dextrans give a violet colour and white dextrin give a blue colour.²

Cellulose derivatives

Cellulose is another natural polymer belonging to the polysaccharides group. The chemical formula for cellulose is $(\text{C}_6\text{H}_{10}\text{O}_5)_n$. It has a chemical structure very similar to amylose. Instead of α -D 1,4 glycosidic bonds, cellulose has only β -D 1, 4 glycosidic bonds as shown in Fig. 5.2. Because of these β -D 1, 4 glycosidic bonds, the molecular chain of cellulose can extend quite linearly, making it a good fibre-forming polymer. In order to use cellulose for sizing purposes, it should be modified to shorten the molecular structure. Two of the most-used cellulose derivatives are carboxymethyl cellulose (CMC) and hydroxyethyl cellulose (HEC).

Carboxymethyl cellulose (CMC) is manufactured from alkali cellulose and sodium chloroacetate. The hydrogen atoms of hydroxyl groups on C2 and C6 are partially substituted with $-\text{CH}_2\text{COONa}$ or $-\text{CH}_2\text{COOH}$ depending on reaction conditions. The degree of substitution (DS) is usually between 0.2 and 1.5 (0.2 to 1.5 carboxymethyl groups ($-\text{CH}_2\text{COOH}$) per anhydroglucose unit). CMC with DS



5.2 Chemical structure of cellulose.

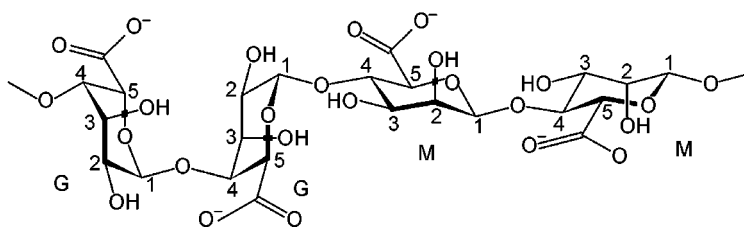
1.2 or below is water soluble. The final product always contains sodium salt. A foaming test can distinguish sodium CMC from other cellulose ethers, alginates and natural gums.³ Sodium CMC solution, after vigorous agitation, would not produce any foam layer. Uranyl nitrate can be used to detect the existence of CMC. A 4% uranyl nitrate is used to precipitate CMC between pH 3.5 and 4.⁴ A 0.5% methylene blue methanol solution may also be used to detect CMC on the fabric.⁵ After rinsing in distilled water and drying, the methylene blue treated sample may show a blue/purple colour which confirms the existence of CMC or acrylic sizes. A separate extraction of the fabric with toluene can exclude the acrylic sizes from the test.

When the hydrogen atom of the hydroxyl group on C6 of cellulose is partially substituted with a hydroxyethyl ($-\text{CH}_2\text{CH}_2\text{OH}$) group in a reaction with ethylene oxide under alkaline condition, hydroxyethyl cellulose (HEC) is produced. So far there are no known testing methods for HEC detection. However, if one wants to distinguish CMC from HEC, an ion tolerance test can be conducted. CMC is anionic and can be precipitated from an aqueous solution with a cationic surfactant. Since HEC is non-ionic, its aqueous solution is compatible with cationic surfactants. Based on the same ionic tolerance principle, a high salt concentration can precipitate CMC, not HEC.

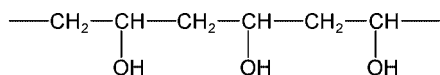
Alginates

Alginates are linear co-polymers of randomly arranged β -D 1, 4 mannuronic acid (M) and α -L 1, 4 guluronic acid (G) blocks as represented in Fig. 5.3. Its chemical structure is similar to that of cellulose except that it has a carboxylic group on the C5 position instead of a methylol group in the case of cellulose.

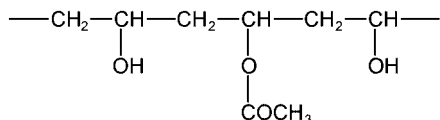
Alginates have good water solubility. Di- and higher valent metal ions, strong acids and bases can precipitate alginates out of its aqueous solutions. In order to distinguish alginates from other thickening agents, precipitation methods can be tried.³ A 2.5% CaCl_2 can cause a 0.5% sodium alginate solution to precipitate. Aqueous solutions of gum arabic, sodium carboxymethyl cellulose, carrageenan, gelatin, gum ghatti, karaya gum, carob bean gum, methyl cellulose and tragacanth gum would not be affected. Saturated $(\text{NH}_4)_2\text{SO}_4$ would not precipitate 0.5%



5.3 Chemical structure of alginate.



(a)



(b)

5.4 Chemical structures of PVA. (a) Fully hydrolysed and (b) partially hydrolysed PVA.

sodium alginate. But agar, sodium carboxymethyl cellulose, carrageenan, de-esterified pectin, gelatin, carob bean gum, methyl cellulose and starch would be affected. The existence of sodium alginate can be tested with acid $\text{Fe}_2(\text{SO}_4)_3$.³ When the sample has been in contact with a ferric sulphate solution for 5 min, a cherry-red colour appears and gradually changes to a deep purple colour. This confirms the presence of sodium alginate in the sample.

5.2.2 Synthetic sizing agents

Polyvinyl alcohol

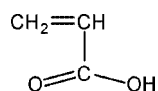
Polyvinyl alcohol (PVA) is the hydrolysis product of polyvinyl acetate. Depending on the hydrolysis conditions, there are fully hydrolysed PVA and partially hydrolysed PVA, as shown in Fig. 5.4a and b, respectively.

Fully hydrolysed PVA usually has a degree of hydrolysis (DH) of 98–99.8% and can dissolve in water only at $> 80^\circ\text{C}$. The solubility of partially hydrolysed PVA with a DH between 85 and 90% is dependent upon its molecular weight. Partially hydrolysed PVA with the higher molecular weight requires a high temperature to dissolve.

Specific detection of PVA on fabrics can be achieved using potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$).⁴ Two solutions are used. Solution A consists of 11.88 g $\text{K}_2\text{Cr}_2\text{O}_7$ and 25 ml concentrated H_2SO_4 in 50 ml distilled water. Solution B contains 30 g NaOH in 70 ml distilled water. After solutions A and B are applied to a white fabric sample sequentially, the brown colour developed indicates the existence of PVA. A yellow-green colour can be triggered by unsized goods, potato starch, styrene-maleic anhydride copolymer, alginates, guar, gelatin or CMC.

Acrylics

Acrylic is a generic term for a large group of homopolymers and copolymers



5.5 Chemical structure of acrylic acid.

derived from acrylic acid, shown in Fig. 5.5. Since the hydrogen atoms of the carboxylic group and the vinyl group can be substituted by many different chemical groups, a huge variety of polyacrylic acid and polyacrylates is currently available for many different applications. Most of them are used as an emulsion.

The analysis of acrylics is almost impossible without using sophisticated instruments. There are no known simple methods for the tests that need to be done in wet chemistry, which could be because (1) analysis with wet chemistry is too complicated; (2) analysis has to deal with too many different types of polymers; (3) analysis involves the use of many toxic organic solvents. If an analysis is needed, it is recommended that either Fourier transform infrared spectroscopy (FTIR) or gas chromatography (GC) be used to obtain results quickly and accurately. FTIR analysis of acrylics can show very distinctive absorption peaks between 1100 and 1150 cm^{-1} for the alkyl C–O–C stretching band and at 1750 cm^{-1} for the C=O stretching band. If the acrylic polymer system contains some vinyl monomers, a broad peak at 3020 cm^{-1} and a strong peak at 1660 cm^{-1} indicate the stretching bands of C–H and C=C in CH=CH structure, respectively.⁶ With GC analysis, acrylic acid, ethyl methacrylate, *n*-butyl acrylate, 2-ethylhexyl acrylate, isobutyl acrylate, methyl methacrylate, ethyl acrylate and methyl acrylate can all be detected and determined quantitatively.⁷ ASTM Test Method D3362⁸ is a standardised method to determine the purity analysis of acrylate. It is certain that more types of acrylates can be successfully analysed nowadays with modern instruments.

5.3 Lubricants and cohesive agents

Lubricants are added into fibre finishes to reduce the friction between fibres and the hard surface of the spinning equipment, whereas cohesive agents are used to increase the frictions between fibres. The overall purpose of using lubricants and cohesive agents is to run the spinning process faster, to reduce fibre damage as much as possible and to make the spinning process run at high speed for as long as possible, which means high quality, large volume and better productivity.

5.3.1 Waxes

Waxes are a group of organic compounds consisting mainly of heat-sensitive hydrocarbons which are insoluble in water but soluble in most organic solvents and, most of them, free from glyceride.⁹ Sources of waxes can be animal, vegetable, mineral, synthetic and petroleum. The waxes used for fibre finishing are

mostly petroleum-based. Paraffin wax is one of them. They are inexpensive and easily available. However, their performance is not as good as synthetic compounds. If used alone, waxes are very difficult to remove in the textile processes that follow their application and use, causing dyeing and finishing problems. Tests for waxes are mainly for their physical properties, such as melting point, flash point, colour, density, odour and so on. Readers interested in more specific tests are recommended to refer to ASTM D1168.¹⁰

One chemical test that is useful for textile wet processes may be the spot test. By using hydroxamic acid, waxes and fatty oils can be distinguished from their mineral counterparts.¹¹ In a small crucible, one drop of a saturated ethanol solution of hydroxamic acid is mixed with one drop of an ethanol solution of the sample and one drop of an ethanol solution of KOH. The mixture is heated to bubbling (evaporation) and then allowed to cool and is acidified with HCl. A drop of FeCl_3 is added to the acidified sample. A violet colour confirms the existence of waxes and fatty oils. Carboxylates may also give a positive result. If the saponification number of waxes needs to be known, ASTM D1387 can be used.¹² ASTM D1386 can be used to determine the acid number of waxes.¹³

5.3.2 Oils and greases

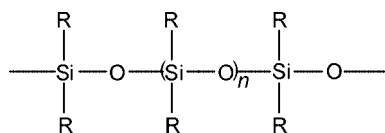
Oils and greases belong to the same type of organic compounds as waxes but have lower molecular weights. Oils have a lower viscosity and are used at lower temperature than greases.

Quantitative analysis of oils and greases can be conducted using methods regulated by IUPAC.¹⁴ Many ASTM methods can also be used to determine the specific chemical properties of oils and greases. For example, ASTM D1980 can be used for the analysis of the acid value;¹⁵ ASTM D1965 for the unsaponifiable matter;¹⁶ ASTM D1957 for the hydroxyl value;¹⁷ and ASTM D1541 for the total iodine value.¹⁸ For the purpose of a qualitative test, two simple but not specific methods can be tried with some interference from other chemicals present in the sample. The first method is based on the fact that oils can usually show fluorescence clearly with UV light. The second method is based on the fact that oils can dissolve solvent dyes but not acid dyes and other water-soluble dyes.

The total content of oils, fats and waxes on fabrics can be extracted with 1,1,1-trichloroethane using the Soxhlet extractor. By comparing the fabric weight before and after extraction, the weight percentage of oils, fats and waxes can be easily calculated.¹⁹ It was reported that supercritical CO_2 can be used instead of the toxic organic solvent to extract the commercial finishes from polyester, nylon and polypropylene fibres.²⁰

5.3.3 Synthetic compounds

Many synthetic compounds can be used as lubricants and cohesive agents. In the



5.6 Chemical structure of silicones.

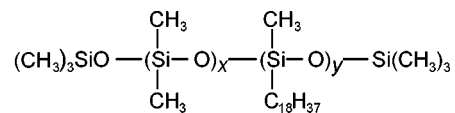
lubricant group, many are esters of organic acids, such as octyl isononanoate, decyl adipate and tridecyl stearate. In the cohesive agent group, many are non-ionic surfactants, such as ethoxylated decyl alcohols, ethoxylated oleyl alcohol and their derivatives. Similar to the case of acrylics, these synthetic compounds vary widely. It is difficult to analyse them chemically using a simple wet chemical mechanism. However, the methods mentioned in [Chapter 4](#), Section 4.3.3 and Section 5.3.2 here can meet most requirements for the textile applications.

5.3.4 Silicones

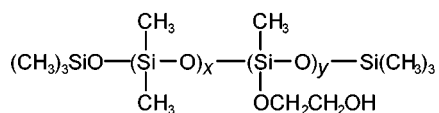
Silicones are a special group of polymers containing --Si--O-- as the backbone of the structure, as shown in Fig. 5.6. The R groups in the structure may be the same or different chemical groups. The most popular are methyl, phenyl and chlorophenyl groups. Silicones can be used as high-performance lubricants in spandex spinning, sewing threads treatment, knitting operation and rope manufacturing.

However, they are not used for yarns that are dyed in the later process because silicone lubricants are difficult to remove, and silicones removed from the yarns can deposit on the surface of machine parts, causing many processing problems.

The most-used silicone lubricants in the fibre finishing applications are polydimethylsiloxane, polymethylphenylsiloxane, oleophilic silicone/organic copolymer (Fig. 5.7a), and poly(glycol silicone copolymer) (Fig. 5.7b). The analysis of silicones can be conducted by FTIR. A strong and often broad absorption in the $1000\text{--}1100\text{ cm}^{-1}$ region can be attributed to the Si–O vibration. A peak at 1100 cm^{-1} shows the Si–O–Si vibration. A sharp peak at 1260 cm^{-1} indicates the Si–CH₃ stretching absorption.²¹

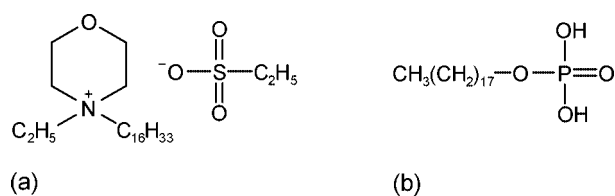


(a)



(b)

5.7 Chemical structures of the two most used silicone lubricants.



5.8 Chemical structures of two common antistats.

5.4 Other additives

In a good fibre finishing formulation, lubricants are always the most important component. However, lubricants alone cannot make the formulation work perfectly. Usually, an antistatic component is included to control the static charge generated during the spinning process, an antimicrobial agent is used to control the growth of microorganisms, an antioxidant is used to prevent the formulation from oxidation damage and a defoamer and an emulsifier are also used to facilitate the production of the final finish. These components, often used in a small quantity, are not necessarily all used together, depending very much on the application and formulation.

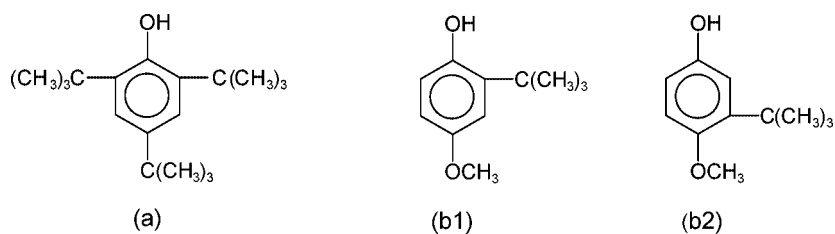
5.4.1 Antistatic agents

'Antistatic agent' can also be called 'antistat' in the industry. The best antistatic agents used in fibre finishes are quaternary amines and alcohol phosphate esters. For example, *N*-cetyl-*N*-ethylmorpholinium ethylsulphate shown in Fig. 5.8a and stearyl alcohol phosphate shown in Fig. 5.8b are representative antistats.

Figure 5.8a is clearly a cationic surfactant. Indeed, the structure shown in Fig. 5.8a can be used as a multifunctional agent, being an antistat, as a cationic surfactant, as a lubricant and even as an antimicrobial agent. The detection of this type of quaternary amines can be followed by the methods described in [Chapter 4](#), Section 4.3.2. Figure 5.8b is an alcohol phosphate that can be easily neutralised as the $-OH$ group connected to the P atom can be dissociated to show the required charge conductivity. When neutralised, it is actually an anionic surfactant. Similar to quaternary amines, stearyl alcohol phosphates can be used as emulsifiers as well as being excellent antistats. Accordingly, they can be detected using the methods presented in [Chapter 4](#), Section 4.3.1.

5.4.2 Antimicrobials

There is a wide range of chemicals suitable for antimicrobial application to fibre finishes. However, for practical reasons, amine-based chemicals, especially quaternary amines are used most because they can show multifunctionality as mentioned in Section 5.4.1.



5.9 Chemical structures of (a) BHT and (b) BHA.

5.4.3 Antioxidants

Antioxidants are used to protect the finishes and the treated fibres from attack by oxygen and heat. Many antioxidants are used in rubber, plastic, composite, paint and coating/laminating materials. Hindered phenols are widely used as antioxidants, especially in the food industry. Two representative ones are butylated hydroxytoluene (BHT, 2,6-di-*t*-butyl-4-methyl-phenol as shown in Fig. 5.9a) and butylated hydroxyanisole (BHA, a mixture of 2- and 3-*t*-butyl-4-hydroxyanisole, shown in Fig. 5.9b1 and b2, respectively).

Apart from the alkylated phenols, phenyl- β -naphthylamine, acetone diphenylamine reaction products and alkylated diphenylamines are also used in different industrial applications and they can absorb UV light. Using proper solvents, these antioxidants can be both qualitatively and quantitatively analysed. Table 5.1 shows the solvents and test conditions for determination of antioxidants.⁷

Sometimes, chelating agents are used to diminish the catalytic effect of metal ions which, if present in the finish system, can promote the autoxidation reaction. EDTA is one of the most commonly used chelating agents. EDTA sodium salt can chelate nickel ions thus preventing the formation of red nickel dimethylglyoxine. Two solutions are needed, one being 0.008% nickel sulphate and the other 1% dimethylglyoxine in alcohol.⁴

5.4.4 Defoamers

Defoamers are used in fibre finishes in order to inhibit the formation of foam during the manufacturing and application of the finishes. Silicones and fluorochemicals are outstanding defoamers. They have very low surface tension and limited solubility in many organic compounds. They can quickly reduce the local surface tension of bubbles to create an imbalance of surface tension which leads to the easy rupture of bubbles. Silicones can be analysed using FTIR. The analysis of fluorochemicals is difficult because fluorochemicals, especially perfluoro compounds, are resistant to many reagents. One microdetermination of fluorine by alkali fusion in a metal bomb was reported.²² Since it is too complicated and specialised apparatuses are used, this method is not introduced here.

Table 5.1 Solvents and test conditions for determination of antioxidants

Antioxidant	Solvent	Detection wavelength (nm)
Phenyl- β -naphthylamine	Toluene	309
Acetone diphenylamine reaction products	85% Methylcyclohexane 15% Ethanol	288
Alkylated diphenylamines	Methylcyclohexane	288

5.4.5 Emulsifiers

Emulsifiers are used to help immiscible components become compatible with each other to form an easily applicable finish, usually in a liquid form. Since emulsifiers are surfactants, they can be tested using the methods presented in [Chapter 4](#), Section 4.3.

5.5 References

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6

Chemical analysis of fabric finishes and performance-related tests

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6.1 Introduction

Textile finishing is the final process in converting greige fabricated textile materials into a condition that allows formation of garments, home furnishings and other consumer goods. Chemical finishing, that is, applying chemicals to textiles to achieve specific performance results, is a major part of the finishing process. Desirable physical and chemical properties of the textile can be enhanced and undesirable properties minimized. In order properly to control, evaluate and optimize the chemical finishing process, analysis of the finished fabric and performance evaluation are necessary. The following sections discuss the various methods that can be used to test chemical finishes and treated fabrics.

6.2 Analysis of fabric finishes

The identification of chemical finishes on fabrics can serve several purposes. Often a fabric with an unknown finish needs to be analyzed for forensic or competitive reasons. Fabrics that do not perform as expected need to be analyzed to determine if the correct finish was applied in the proper concentration. Regular analysis of production fabrics forms a basis for process improvement through application of statistical process control methods.

The special character of textile finishes can make chemical analysis less than straightforward. A typical chemical finish for textiles has numerous components including the active agent, surfactants, softeners and miscellaneous processing aids. In addition, many finishes undergo chemical changes during the finishing process, that is, the cross-linking reactions that occur in durable press finishing. If the fabric has been washed after finish application either as part of the finishing process or by a consumer after purchase, the water-soluble components may have been partially or completely removed. The most useful approach is often to prepare fabrics with known finishes and use these as internal standards when analyzing unknown or questionable fabrics.

Table 6.1 Infrared frequencies associated with chemical finishes

Absorption frequency (cm ⁻¹)	Chemical bond
3700–3300	O–H
3500–3200	N–H
2967–2857	C–H
2252–2062	C≡N
1750–1735	C=O (esters)
1725–1700	C=O (saturated aliphatic acids)
1725–1705	C=O (saturated aliphatic ketones)
1680–1630	C=O (amides)
1600–1500	C=C (aromatic)
1570–1515	N–H (secondary amides)
1250–1150	C–O (esters)
~ 1250	P=O
720–730	C–H

The following sections discuss a variety of analysis methods that can be used to examine textile finishes. The discussion is divided into two sections, one for methods that can be used with fabrics without separation techniques and the other for techniques that require extraction of the finish or finish component of interest from the fabric.

6.2.1 Non-extractive methods

Infrared spectroscopy

One of the most useful analytical methods for examining chemical finishes on fabrics is infrared spectroscopy.^{1–3} By using a reflectance infrared sampling process, the finish does not need to be separated from the fabric. In this technique, the fabric sample to be analyzed is exposed to infrared radiation in the 600–4000 cm⁻¹ range. After reflection, the resultant energy spectrum is recorded. The chemical bonds in the sample absorb specific wavelengths of the incident radiation depending on the nature of each molecule's vibrations. Specific absorption frequencies in the infrared region have been assigned to particular chemical bonds. Table 6.1 lists some of the frequencies associated with chemical finishes used with textiles.

By studying the absorbed frequencies from a sample, the presence or absence of specific functional groups can be inferred.

Of the various infrared techniques available, Fourier transform infrared (FTIR) spectroscopy has become the most useful for textile finish analysis. The entire spectrum is recorded simultaneously and scanning is not necessary. In this way, many spectra can be recorded and averaged in a short time. FTIR utilizes a computer interface to store and manipulate the recorded spectra. The spectra can then be easily compared with libraries of standard spectra to aid in the identifica-

tion of the fabric finish. FTIR is often combined with the attenuated total reflectance (ATR) data collection technique. The ATR technique involves placing the fabric sample horizontally on a zinc selenide crystal. The incident infrared radiation enters the crystal at one end and exits the other after multiple internal reflections between the sample surface and the crystal interior. The radiation typically only penetrates the sample to a depth of a few micrometers, so the technique is limited to surface studies. An example of the use of FTIR with textile finishes is given by Morris *et al.*⁴ The authors determined the amount of a durable press finish (dimethyloldihydroxyethylene urea or DMDHEU) on finished fabric by first generating fabrics with known levels of DMDHEU and then analyzing them with both FTIR and elemental nitrogen analyses. The percent DMDHEU found by FTIR compared very favorably with the amounts found by nitrogen analysis.

Another very useful technique similar to infrared spectroscopy is near infrared (NIR) spectroscopy.⁵ With this method, radiation in the 4000–12 500 cm⁻¹ range is employed. This radiation is energetic enough to excite overtones and harmonics of molecular vibrations. The method can be used to determine organic functional groups such as O–H, N–H, and C=O quantitatively if adequate calibration samples are available. The instrumentation used with NIR makes it quite suitable for in-process monitoring of textile finishing processes.⁶ NIR has also been used to determine DMDHEU and DMDHI (1,3-dimethyl-4,5-dihydroxy-2-imidazolidinone) concentrations on cotton fabric.⁷ In this study, excellent correlation was found between the NIR results and elemental analyses.

X-ray fluorescence

When a material is exposed to a beam of high-energy X-ray radiation, the inner shell electrons of the material's atoms are ejected by the absorbed X-rays and outer shell electrons fall into the vacant orbitals, emitting characteristic fluorescence.⁸ The intensity of each individual fluorescence is proportional to the amount of that element present. The radiation for X-ray fluorescence is generated with tungsten, molybdenum or chromium target tubes in high-voltage electrical fields. X-ray fluorescence instrumentation can provide concentrations of suitable elements accurate to 0.1% in less than 1 min. No special sample preparation is needed, however, calibration with known standards should be done if quantitative results are desired. The elements that are most suitable to be studied by X-ray fluorescence are those with atomic number 12 (magnesium) and higher. Flame-retardant finishes are particularly convenient to analyze with this technique since they usually contain elements in that atomic number range (i.e. phosphorous, chlorine, bromine, antimony). A study has shown that X-ray fluorescence and elemental analyses gave comparable results for a series of phosphorus-based flame-retardant fabrics.⁹

X-ray photoelectron spectroscopy (XPS), sometimes called electron spectroscopy

for chemical analysis (ESCA), is an analysis technique similar in some ways to X-ray fluorescence.¹⁰ In XPS, the sample to be analyzed is exposed to high energy X-rays generated from magnesium and aluminum targets by high voltages. As in X-ray fluorescence, inner shell electrons are ejected when the X-rays are absorbed. However, in the XPS technique, the ejected electrons are collected and their energies measured. Only surface atoms can be studied by XPS owing to the short range of the electrons involved. In addition, the sample chamber must be kept under ultra high vacuum conditions (10^{-9} millibar) to avoid interference from molecules in the atmosphere adsorbed on the sample surface. The ejected electron energies are characteristic of the binding energies of the atoms on the material's surface. The shapes of the peaks in the electron energy spectrum are influenced by chemical bonding, so XPS can provide both elemental and chemical bond information. The only elements not able to be detected by XPS are hydrogen and helium. This technique is particularly useful in studying textiles that have been plasma treated, since the treatments are restricted to the surface of the material. In a recent study of cotton treated with a fluorochemical containing atmospheric plasma,¹¹ XPS showed that both CF_4 and C_3F_6 plasmas introduced C–F bonds at the fabric surface, with the C_3F_6 treatment providing a higher level of modification. Fewer COH and COOH groups were also detected after the plasma treatments.

6.2.2 Extractive methods

Separating a chemical finish from the textile to which it has been applied can be a daunting task. Some finishes, especially those intended to be non-durable might simply be removed by rinsing with the appropriate solvent. However, finishes that are expected to provide performance over the life of the textile cannot usually be removed that easily. In these situations, the entire finish–textile assembly must be broken down by chemical or thermal means and the resulting components analyzed.

Elemental analysis

Elemental analysis is probably the oldest method that has been used to analyze textile finishes. It is still the 'gold standard' in that all newly developed methods are judged by how well results from the new method compare to results from elemental analysis. Traditional wet chemical methods, such as Kjeldahl nitrogen analysis,¹² continue to have a place in finish analysis. Elemental analysis can be qualitative or quantitative, depending on the need.

Qualitative elemental analysis

Spot tests are very useful in determining the presence or absence of particular elements. Several good references are available for elements commonly used in

textile finishes.^{13,14} A few tests, for example, iron or copper that are present in large amounts can be done directly on the fabric. However, the usual practice is to prepare solutions, either from fabric extracts or from fabric digestion. These solutions can be concentrated by evaporation if necessary and the appropriate masking agents added. The specific reagents for a particular element are added and a distinctive color change or precipitation indicates the presence of that element.

Quantitative elemental analysis

In atomic spectroscopy,¹⁵ a material is exposed to high-energy conditions (2000–6000 °K) in a flame, furnace or plasma. The resulting gaseous atoms can absorb or emit ultraviolet (UV) or visible radiation in quantities proportional to their concentration. Atomic absorption (AA) measures the absorption of radiation from atoms produced from a solution injected into a flame. A hollow-cathode lamp containing the element under investigation emits radiation into the flame. Any atoms of that element in the flame absorb that radiation and decrease the intensity of the lamp in proportion to their concentration. With appropriate calibration curves, accurate concentration levels of the element in the original material can be determined. Most metals found in textile finishes, such as magnesium, aluminum and zinc, can be measured. Some disadvantages of AA are that only one element at a time can be analyzed and that a separate hollow-cathode lamp is needed for each element. Inductively coupled plasma spectroscopy (ICP) measures the emission of radiation by atoms generated by injecting a liquid sample into a plasma. This method allows for the simultaneous determination of multiple elements. In addition, more elements likely to be found in textile finishes can be detected with ICP including halogens (except fluorine), phosphorous, sulfur and silicone.

Formaldehyde tests

Formaldehyde is an important constituent of many textile finishes, especially finishes for cellulosic textiles. As the main chemical route to cross-linking cellulose, formaldehyde or a reactant made from formaldehyde is found in a variety of finishes, ranging from durable press agents to water repellents.

Although a very useful and versatile chemical, formaldehyde also causes breathing difficulties and headaches if inhaled, causes eczema and allergies with skin contact and is a suspected human carcinogen. As a result, several countries have enacted laws to control the level of free or easily released formaldehyde in textiles. These levels are specified with particular test methods since there can be significant differences in results obtained with different test methods. Formaldehyde in durable press-treated fabrics can exist in several forms since the formaldehyde reactions are all equilibrium reactions and the 'bound' formaldehyde can be converted to free formaldehyde to varying degrees depending on the test conditions. Therefore, when analyzing fabric for formaldehyde, the test

method used must be reported with the data. Some of the more common fabric test methods for formaldehyde¹⁶ are summarized below.

A qualitative test for the presence of formaldehyde is to add a small piece of the fabric to 3 ml of a solution of 10 mg chromotropic acid per 100 ml of 72% sulfuric acid. The solution is warmed in a test tube to 100 °C for 1 min. A red to violet color indicates the presence of formaldehyde.

In the USA, the regulatory focus is on consumer and worker exposure to formaldehyde vapors released from the fabric, so the test method specified is AATCC Test Method 112-2003.¹⁷ In this method, 1 g of fabric is suspended over 50 ml of distilled water in a sealed quart jar. The jar is placed in an oven for either 4 h at 65 °C or 20 h at 49 °C. Any formaldehyde vapors generated are absorbed by the water. An aliquot of the formaldehyde–water solution is taken and analyzed colorimetrically using the Nash reagent.¹⁸ Typical levels of formaldehyde found in properly processed fabrics treated with modern cross-linking reagents are less than 100 ppm. The Nash method is based on the reaction of acetylacetone with formaldehyde and an ammonium salt to form a yellow complex with an absorbance maximum at 414 nm. The mild conditions of the reaction (pH 7, 5 min at 58 °C) eliminate many potential interferences.

In Japan, the concern for formaldehyde is the dermatological effect of wearing formaldehyde-containing textiles and the specified test method reflects that concern. Japanese Law 112-1973¹⁹ provides a procedure for extracting 1 g of fabric with 100 ml of distilled water at 40 °C for 1 h. An aliquot of the water is then analyzed colorimetrically using Nash reagent. The maximum level of allowable formaldehyde is 75 ppm in children's and adult clothing, no formaldehyde is allowed in infant wear.

Chromatographic methods

Several reviews of the use of chromatographic methods to analyze textile chemicals have appeared.^{20,21} Since most textile finishes are either polymeric in nature or covalently bonded to the textile, the finishes must be separated from the fabric and solubilized prior to chromatographic analysis. These separation techniques tend to break down the finish into its component parts making unambiguous analysis difficult. Some of the more useful chromatographic methods are summarized below.

Pyrolysis chromatography involves heating samples to temperatures in the range of 600–1000 °C in a very short period of time (ms). The volatile products of this pyrolysis can then be analyzed by gas chromatography (PY–GC), mass spectrometry (PY–MS) or their combination (PY–GC–MS). In gas chromatography, adsorption–desorption between the gaseous material and the solid column packing causes retention time differences between the various components allowing their identification by comparison of retention times to standards. By placing a mass spectrometer at the exit end of the column, additional information can be

learned about the components. Fabrics treated with phosphorous²² and bromine-containing flame retardants²³ have been studied by these procedures.

High-pressure liquid chromatography (HPLC) allows the separation of soluble materials into component parts based on the partitioning of the materials between a mobile (solvent) and stationary (column packing) phase. With proper choice of solvent and column packing, separation of many types of materials is possible. HPLC has been used successfully to determine reagent residues on fabrics treated with durable press finishes^{24,25} as well as determining formaldehyde levels in aqueous solutions obtained by the AATCC Test Method 112.²⁶

6.3 Finish performance tests

Chemical finishing is an important area in textile processing and requires a diligent fabric testing program to maximize the benefits of the chemical treatments. Many chemical finishes have an optimum level of application, too much chemical can be wasteful, too little can compromise the desired fabric properties. The development of the appropriate performance test methods is just as important to the commercial success of a chemical finish as the development of the finish itself. A test method is appropriate if it provides useful, reproducible results that correlate with actual 'real world' performance. The best test methods utilize simple, inexpensive equipment with easy to follow procedures and yield precise, accurate data.

Organizations such as the Association of American Textile Chemists and Colorists (AATCC) and the American Society for Testing and Materials (ASTM) in the USA, the British Standards Institute in the United Kingdom, the Deutsches Institut für Normung (DIN) in Germany and the International Organization for Standardization (ISO) headquartered in Switzerland publish test methods that can be used to evaluate the performance of chemical finishes.

The following sections will discuss some of the more important test methods in use for fabrics with durable press, flame-retardant, soil release, repellent, UV protective, antimicrobial, anti-insect, anti-felting, hand building and weighting finishes.

6.3.1 Durable press testing

Cellulosic textiles, although valued for their comfort, have the unfortunate disadvantages of wrinkling while being worn and wrinkling and shrinking during laundering. Chemical finishes have been developed that can provide dimensional stability, wrinkle resistance and shape retention to cellulosic and cellulosic blend fabrics. These finishes are referred to as 'easy care', 'wrinkle resistant', 'permanent press' or 'durable press' finishes. These fabric property improvements are achieved by cross-linking the cellulosic polymer chains into the desired configuration.²⁷ A variety of tests has been developed to determine the effectiveness of the treated fabrics. Some of the more common procedures are discussed below.

Performance tests

AATCC Test Method 135-2003²⁸ determines the dimensional changes of fabrics when subjected to controlled home laundering. Different wash temperatures, agitation cycles, rinse temperatures and drying procedures can be chosen for any particular home laundering condition. Several options for sample preparation are available based on fabric size. The fabrics are marked with benchmarks prior to laundering and the distance between the marks is compared before and after laundering. The percentage dimensional change can then be calculated.

AATCC Test Method 124-2001²⁹ is a procedure that attempts to standardize the home laundering process. Choices of hand- or machine laundering, wash cycles, wash and rinse temperatures and drying procedures are provided. The method specifies washing machine, load size, detergent composition, tumble dryer and visual rating procedures. Any fabric construction can be evaluated, but the most meaningful results are obtained with woven fabrics. Three fabric samples (each 38 × 38 cm) are laundered and dried with a ballast load (total weight of 1.8 kg). After conditioning, the appearance of the samples is then visually compared to plastic replicas (AATCC three-dimensional smoothness appearance replicas) that allow ratings on a 1–5 scale, with 5 being the smoothest appearance. A typical commercially acceptable rating is ≥ 3.5 .

AATCC Test Method 128-1999³⁰ determines the tendency of fabrics to wrinkle under carefully controlled laboratory conditions. Fabrics made from any fiber or fiber combination can be evaluated. A special apparatus (AATCC wrinkle tester) provides consistent wrinkling force to the 15 × 28 cm preconditioned fabric samples with a 3500-g weight. After being wrinkled for 20 min, the samples are allowed to hang vertically for 24 h under standard conditions and then compared to plastic replicas (AATCC three-dimensional wrinkle recovery replicas) to obtain a smoothness rating on a 1–5 scale. An average of nine determinations is reported.

AATCC Test Method 66-2003³¹ provides a quantitative measure of a woven fabric's ability to recover from induced wrinkling. Fabric samples (15 × 40 mm, six with the long dimension parallel to the warp direction of the fabric and six with the long dimension parallel to the filling direction) are folded and compressed under controlled conditions of time and force to create a wrinkle. The wrinkled sample is then suspended in one of two recommended testers (AATCC wrinkle recovery tester or James H. Heal crease recovery angle tester) for a controlled recovery period after which the recovery angle is measured. The sum of the average warp recovery angle and the average filling recovery angle is reported. The test can be done with dry or wet samples.

The ISO test method 2313: 1972³² is essentially the same procedure as option 2 of AATCC Test Method 66-2003.

AATCC Test Method 88B-2003³³ is designed to evaluate the smoothness appearance of fabric seams after laundering. Any fabric with any type of seam is suitable for testing with this method. Fabric samples (38 × 38 cm) are cut in half

and then seamed back together using the chosen seaming method. A choice of hand- or machine washing, machine wash cycles, wash temperatures and drying procedures is provided. Two sets of AATCC photographic seam smoothness replicas (one for single needle seams, one for double needle seams) can be used to rate the seam appearance on a 1–5 scale, with 5 having the smoothest appearance.

AATCC Test Method 88C-2003³⁴ allows the evaluation of the durability of intentional pressed-in creases to home laundering. Creased fabric samples are laundered and dried under controlled conditions similar to those in AATCC Test Method 124-2001. The washed and dried samples are then visually compared to plastic replicas (AATCC three-dimensional crease replicas) which allow ratings from 1–5, with 5 being the sharpest crease.

AATCC Test Method 143-2001³⁵ applies the washing, drying and evaluation procedures of test methods AATCC TM 124, 88B, 88C to apparel and other textile products.

In addition to these tests, which measure the enhanced performance properties, fabrics that have been treated with durable press treatments also need to be tested for strength (tensile and tear) and abrasion resistance since the cross-linking reaction leads to fiber and fabric strength losses. Appropriate tensile strength tests include ASTM D5034 and D1424 for woven fabrics and D3786 for knitted fabrics. Tearing strength for all fabrics can be determined by ASTM D5587 and D2261.³⁶

6.3.2 Flame retardancy testing

One of the more important functional textile finishes is the flame-retardant finish. Firefighters and military personnel have obvious needs for such protective apparel. In addition, textiles used in public buildings are required to have similar protection. Flame retardancy can be achieved by several chemical means³⁷ which usually involve phosphorous and/or bromine chemistry. The tests that have been developed for evaluating flame retardancy are typically designed for specific textiles under specific testing conditions and these conditions may or may not be comparable to real world fire situations. In addition, these tests are continually being revised. Nonetheless, many of these tests have been established by various legislative bodies and fabrics being sold into markets covered by the legislation must meet the requirements.

The United States Consumer Product Safety Commission (CPSC) has published several test methods that are used to qualify apparel for sale in the USA.³⁸ Test 16 CFR 1610 is a test for general apparel.³⁸ A fabric sample (2 × 6 inches, 5 × 15 cm) is placed on a sample rack in a draft proof ventilated chamber at an angle 45° from the horizontal and a cotton cord is placed 5 inches (13 cm) from the narrow fabric edge. A butane flame is held to the narrow edge for 1 s. The test ends when the cord is burned through or the fabric fails to ignite. The fabric passes if it does not ignite or if only one of five tested samples burns the cord after more than 4 s. There are special grading rules for raised surface fabrics.

Children's sleepwear have separate tests, 16 CFR 1615/1616,³⁸ depending on the size of the garment. These tests are quite similar, both impinging a methane flame for 3 s to the bottom edge of a 3.5 × 10 inch (8.9 × 25.4 cm) fabric sample suspended vertically in the draft-free test chamber. In order to achieve a passing grade, five samples of the tested fabric must have an average char length of 7 inches (17.8 cm) or less with no sample having a char length of 10 inches (25.4 cm).

The National Firefighters Protection Association (NFPA) has developed a specific flame retardancy test for the protective clothing worn by firefighters.³⁹ NFPA 1971 is a compilation of requirements for coats, trousers, helmets, gloves, footwear and interface items that make up a firefighter's protective ensemble. The flame retardancy test exposes a vertically held 3 × 12 inch (7.6 × 30.5 cm) fabric sample for 12 s to a flame produced by a special gas mixture (540 BTU/cubic foot (4806 kcal m⁻³)). An acceptable fabric will have an average char length of 6 inches (15.2 cm) or less, an after-flame of less than 2 s, and an afterglow of less than 4 s.

A very useful test for flammability is ASTM D2863-00.⁴⁰ This method provides a procedure for measuring the minimum oxygen concentration that will just support flaming combustion in a flowing mixture of oxygen and nitrogen. This minimum concentration, also known as the limiting oxygen index or LOI, can be used to quantify flammability behavior of plastics and textiles. A sample of the material to be tested is placed in an open top glass column and a flow of oxygen and nitrogen is begun at the bottom of the column. A natural gas flame is used to ignite the top of the sample. The amount of oxygen is adjusted until the flaming sample just continues to burn. The concentration of oxygen at that point is the LOI. For example, materials that will continue to burn in air have LOIs of ~20. Materials that have good flame retardancy can have LOIs of 30 or greater, meaning that they will burn in an atmosphere of 30% oxygen, but not in air.

Recently the State of California has passed a stringent flammability requirement for mattresses sold in the state beginning in January 2005. Since the size of the California market is so large, California standards tend to become national standards as manufacturers decide for business reasons to have all of their products comply with California law. The requirement (Technical Bulletin 603 of the Bureau of Home Furnishings and Thermal Insulation, Department of Consumer Affairs, State of California) specifies a burner apparatus and testing conditions for mattresses. The measured quantity is the total heat evolved 30 min after subjecting the mattress to severe flame exposure.

6.3.3 Soil release testing

Most consumers would like to be able to clean their garments effectively during laundering. The ability of textiles to release soil during laundering is a function of many factors including the nature of the soil, the mechanical action imparted by the washing machine, the composition of the detergent, the structure of the textile, the

washing temperature and the surface characteristics of the textile fiber. In order to release oily soil easily, the most difficult soil to remove from synthetic fibers, the fiber surface should be both hydrophilic and oleophobic. A variety of chemical materials have been used commercially to achieve this goal.⁴¹ Evaluating soil release finishes is best done by subjecting the fabrics to simulated home launderings under carefully controlled conditions.

AATCC Test Method 130-2000⁴² has been developed to measure the ability of fabrics to release an oily stain when laundered. Fabric samples (15 × 15 inches) (38 × 38 cm) are stained with corn oil, then a specified weight is used to force some of the oil into the fabric interior. After laundering with a specified detergent under a choice of laundering conditions, the samples are dried in a tumble dryer and compared to photographic standards (stain release replica with the usual AATCC 1–5 scale).

6.3.4 Repellency testing

Repellent finishes are important components of many protective textiles. Applications for repellent textiles range from medical textiles to raincoats. The low surface energies provided by repellent finishes can keep solid and liquid soils from adhering to treated fiber surfaces. Finishes based on hydrocarbon and silicone chemistries can yield water repellent textiles, while fluorochemicals are necessary to achieve the low surface energies needed for dry soil and oil repellency.⁴³

AATCC Test Method 22-2001⁴⁴ is a simple test for rapid screening of water repellency. The fabric sample (7 × 7 inches) (18 × 18 cm) is stretched tight in an embroidery hoop, held at a 45° angle in the test apparatus and sprayed with 250 ml of water through a specified spray head from a height of 150 mm. Photographic standards are used to evaluate any wetting pattern that is formed. A completely non-wetting fabric is given a 100 rating, while a fabric that wets completely is given a 0 rating.

AATCC Test Method 35 -2000⁴⁵ is designed to simulate a rain event. A special apparatus is used to hold the 20 × 20 cm fabric sample in a vertical position backed by a weighed piece of blotter paper. The fabric face is sprayed with water under constant hydrostatic pressure for 5 min and the blotter paper reweighed. The increase in weight of the backing paper is a measure of the resistance of the fabric to penetration by the simulated rain.

A more severe simulation of a rain event is provided by ISO 9865.⁴⁶ This test method is similar to AATCC Test Method 35 except that the water is sprayed for 10 min and the undersides of the fabric samples are rubbed while the spraying takes place. A rather elaborate apparatus is required. The appearance of the fabric face, the amount of water absorbed by the fabric and the amount of water passing through the fabric are all factors in determining the repellency rating of the fabric.

Oil repellency can be evaluated by AATCC Test Method 118-2002.⁴⁷ Drops of hydrocarbon with various surface tensions are applied to the fabric sample and the

fabrics observed to determine if wetting has occurred. The hydrocarbon series varies from mineral oil (rating of 1) to n-heptane (rating of 8). The fabric is assigned a repellency rating based on the hydrocarbon with the lowest surface tension that does not wet the fabric. For example, a fabric that is not wetted by n-heptane in this test is given a rating of 8.

INDA, the Association of the Nonwoven Fabrics Industry, has published repellency test methods specifically designed for the nonwoven fabric structure. IST 80.5(01)⁴⁸ is designed to measure a nonwoven material's ability to resist gravity-only penetration by a saline solution. This property is useful in assessing the degree of water repellency needed by nonwovens in a number of applications. A sample of the nonwoven to be tested is placed in the mouth of a quart size Mason jar containing saline solution. The jar is then inverted and placed on an electric grid that senses when the solution has penetrated the nonwoven. The time to complete penetration is recorded as a measure of repellency.

Another INDA repellency test especially useful for nonwovens intended for medical use is IST 80.8(01).⁴⁹ This test is similar to AATCC 118 in that a series of liquids with varying surface tensions are placed dropwise on the sample to determine at what point the nonwoven is wetted. The difference in this test is that the liquids used are composed of varying concentrations of alcohol and water from 100% water (rating 0) to 100% alcohol (rating 10). Methanol, ethanol and isopropanol may be used as the alcohol component. The higher the rating, the higher the level of repellency of the nonwoven.

Upholstery and carpets are often treated with repellent finishes to minimize soiling during use. Since much of the soiling that occurs is due to dry soil, a test to evaluate dry soiling behavior is useful. AATCC Test Method 123-2000⁵⁰ can be used for upholstery and carpet samples. Either 10 g of actual vacuum cleaner soil or 10 g of a synthetic soil is rotated with pebbles in a rotary ball mill with two samples (3 × 7 inches) (7.6 × 18 cm) of the fabric to be tested. After a predetermined exposure time, the fabrics are removed, vacuumed and visually compared to previously soiled standards.

6.3.5 UV protective testing

The harmful effects of exposure to ultraviolet radiation from sunlight on human skin were recognized by the medical community in the early 1990s. Long-term exposure to ultraviolet light can result in accelerated skin ageing, acne, phototoxic reactions with drugs, sunburn, skin cancer, cornea damage and DNA mutations. The textile industry has responded to this recognized need by providing chemical treatments that absorb ultraviolet light. Government agencies around the world have also responded by providing standards for clothing designed to protect the wearer from ultraviolet rays. The standards define an ultraviolet protection factor (UPF) to be included on clothing sold as ultraviolet protective. The higher the UPF, the greater the ultraviolet protection provided.

The early test methods for determining the ultraviolet protective effects of clothing involved exposing volunteers to ultraviolet radiation until their skin reddened (erythema) to a specific degree. Fortunately, instrumental methods of evaluation have been developed that eliminate the need for sunburned assistants. AATCC Test Method 183-2000⁵¹ determines the transmittance of ultraviolet light through fabric samples. Through the use of established tables and formulas, the UPF is calculated.

6.3.6 Antimicrobial testing

The growth of microorganisms on textiles can cause functional, hygienic and aesthetic problems. Fungi and bacteria are the most troublesome organisms. Fungi can cause discoloration and fiber damage, while bacteria can produce unpleasant odors and a slimy feel to fabrics. Of course, growth of pathogenic bacteria on textiles presents health hazards to the wearer and to the general public. A successful antimicrobial textile finish must be effective against the target organisms, yet not harm the wearer or the environment. Textiles marketed as biocidal, that is, capable of killing organisms, are mandated by law to meet strict efficacy requirements, whereas biostatic (growth inhibiting) textile finishes are held to much more lenient standards. In the USA, biocidal products must be registered with the Environmental Protection Agency before they can be sold.

Antibacterial tests

Qualitative and quantitative test methods have been developed for evaluating the antibacterial properties of textiles. AATCC Test Method 147-1998⁵² provides a qualitative measure of a textile's antibacterial effects. A small sample of the textile (25 × 50 mm) is placed in a sterile agar-containing plate that has been streaked with either a gram positive- or gram negative bacteria-containing solution. After incubation for 18–24 h at 37 °C, the plate is examined for bacterial growth. If the bacteria did not grow on the textile, an antibacterial effect can be claimed. Although this method is suitable for a rapid assessment, a more quantitative test is needed to determine if the effect is biocidal or biostatic.

AATCC Test Method 100-1999⁵³ was developed to provide a quantitative evaluation of a textile's antibacterial properties. A swatch of the antimicrobial treated textile (1.9 inch (4.8 cm) diameter disk) is inoculated with a bacteria-containing solution (either gram positive or gram negative) and incubated for 18–24 h at 37 °C. The swatches are then extracted and the number of bacteria in the extract determined by serial dilutions placed on sterile agar containing plates and incubated for 48 h at 37 °C. Calculations are made to find the percentage reduction of bacteria found on the antimicrobial treated textile compared to the number of bacteria found on an untreated textile of similar construction. Care must be taken

to ensure that bacteria actually grow on the untreated textile sample. This can be accomplished by incorporating a small amount of growth medium in the initial inoculating solution.

Antifungal testing

AATCC Test Method 30-1999⁵⁴ provides four methods for determining the antifungal properties of a textile. The first method involves covering the textile samples (1.5 × 6 inches) (4 × 15 cm) with fungi-containing soil and incubating the mix for 2–16 weeks at 28 °C. In the second method, cellulosic fabric (1.5 inch (4 cm) diameter disk or 1.5 × 6 inch (4 × 15 cm) strip) is inoculated with a standard fungus (*Chaetomium globosum*) solution and incubated on a sterile agar-containing plate. The third method is similar to the second except that the fungus *Aspergillus niger* is used as the inoculum. The fourth method exposes 1 × 3 inch (2.5 × 7.6 cm) textile samples to a high humidity environment for 14–28 days in a sealed jar after inoculation with a mixture of three fungi. In all methods, the extent of fungal growth is determined visually and reported as either ‘no growth’, ‘microscopic growth’ or ‘macroscopic growth’. In all the methods except the third, fabric strength losses can also be measured.

6.3.7 Anti-insect and mite testing

Although desirable from an aesthetic and comfort sense, animal fibers have the disadvantage of being damaged by a variety of insects that have the ability to digest keratin. These insects include clothes moths, carpet beetles and fur beetles. The main defenses against these insect pests are digestive poisons and nerve poisons. These materials must be used carefully to prevent harm to other non-destructive insects or to the environment in general.

To evaluate the effectiveness of anti-insect finishes, populations of the target insects must be established. AATCC Test Method 24-1999⁵⁵ provides detailed procedures for rearing and handling clothes moths and carpet beetles in the testing laboratory. Options are available for evaluating fabrics, yarns, and carpets. The textile materials to be tested (samples of 2 inch² (12.9 cm²)) are placed in glass or metal containers with ten larvae of the target species. After 14 days of incubation at 27 °C and 55% relative humidity, the samples are removed and examined for damage. The extent of the damage can be measured by weight loss of the samples or by the total mass of insect excrement generated. The number of living and dead insects is also recorded. This test method is usually used in conjunction with AATCC Test Method 28-1999⁵⁶ to evaluate potential anti-insect finishes under conditions of ordinary use.

Recently concerns over the adverse effect of house dust mite fecal matter on asthma sufferers has led to interest in textile treatments to control mite populations in mattresses, sheeting, pillows and bed covers. Dust mites are not insects but are

more closely related to spiders. Chemical treatments, both biocidal and biostatic, are available. The AATCC has a test method under development to evaluate the effectiveness of these treatments. Fabrics to be tested are inoculated with dust mites and a growth medium. The number of mites surviving after six weeks incubation at 23 °C and 74% relative humidity gives an indication of the anti-mite property of the treated textile.

6.3.8 Anti-felting testing

Wool fibers have a scaly covering that leads to an interesting phenomenon referred to as felting. When wool fabrics are laundered, the individual fibers can slide along each other in one direction (the 'with-scale direction'), but not the other (the 'against-scale direction'). This situation causes irreversible shrinkage of the fabric. Chemical treatments that address this problem can either remove the scales by chemical reaction or 'fill in' the rough areas with a polymer deposit to give a smoother surface and allow the fiber to return to its original position more easily, or 'spot weld' adjacent fibers to prevent movement in the first place. The success of 'washable wool' in the market place attests to the usefulness of these finishes.

The Woolmark Company Test Method TM31⁵⁷ provides a method of washing wool in a special washing machine designed to duplicate the mechanical action found in home laundering. The wool fabric is marked before and after being laundered under controlled conditions and the shrinkage calculated.

6.3.9 Hand building and weighting testing

Often a textile fabric requires a firm feel or hand. This can be accomplished by adding a chemical finish to increase the fabric stiffness. These finishes are usually low-cost water-soluble polymers such as starch or polyvinyl alcohol. If a durable stiffening effect is needed, vinyl acetate polymers, polyurethanes and thermo-setting resins can be used.

The effects of a fabric stiffening treatment can be quantified by the cantilever method described in ASTM D1388-96.⁵⁸ A sample of specified length and width is placed on top of the test device. The device has a horizontal surface connected to a downward incline. The fabric is pushed over the incline until the sagging leading edge just touches the inclined plane of the device. The length of fabric that has passed over the point where the horizontal and incline meet is used to calculate the fabric's flexural rigidity.

Some of the same chemicals used to stiffen fabrics can also be used to increase a fabric's weight, an important sales specification. Silk fabrics can be conveniently weighted by the application of tin salts. Measurement of the degree of weighting can be done by comparing the final weight of the fabric in g m^{-2} to the initial weight.

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