27. Dyeing Principles and Dyes for Wool Fabrics

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Learning objectives

By the end of this lecture, you should be able to:

• Outline the classes of dyes suitable for the dyeing of wool fabrics.
• Explain the purposes of dyeing assistants and auxiliaries used in the dyeing of wool fabrics.
• Describe the mode of action of levelling agents.
• Outline the various factors that affect the dyeing performance of wool fabrics.
• Describe the various methods employed for the determination of the colourfastness of wool dyeings.

Key terms and concepts

Dye, dyestuff, acid dyes, metal complex dyes, chrome dyes, reactive dyes, dyeing auxiliaries, levelling agents, level dyeing, approach, adsorption, diffusion, migration, fixation, exhaust dyeing, continuous dyeing, wash fastness, rub fastness, light fastness.

Introduction

Wool is a protein (animal) fibre and its physical and chemical properties can vary between breeds, and between sheep within the same breed. Other factors such as the condition of the sheep, their diet and the prevailing environment during the growth period can have a great influence on the dyeing properties of wool. In addition, wools also vary in their base colour and have differences between tip and root that affect dye uptake. These variations are overcome, to some extent, by blending. Even so, no one blend will dye the same as another blend and hence the dyer must take special care in the selection of dyestuffs, auxiliaries and dyeing method.

Dyeing can be conducted in loose stock form, or as sliver, yarn or fabric. This lecture provides and overview of wool dyeing with particular reference to the dyeing of wool fabrics. The topic of wool dyeing is a very extensive one, and therefore can only be dealt with quite briefly in this lecture.

The general references for this lecture are (Parton, 2002) and (Lewis, 1992). The latter reference is a very comprehensive book on the subject of wool dyeing, with contributed chapters from ten experts in the field.

27.1 Dyestuff classes for wool fabrics

Dyes employed in the colouration of wool fabrics are generally supplied as powders, granules or in liquid form. They are soluble in water, so that wool dyeing recipes are aqueous based. Wool dyestuffs have been designed to have a strong affinity, i.e. a strong attraction, for the protein of the wool fibre. These forces of attraction include ionic attraction (viz. Coulomb’s Law – opposite charges attract each other), van der Waal’s forces, hydrophobic forces, etc.

In general, wool dyes are classified according to their chemical type and method of application. The main classes of dyes currently used for the dyeing of wool fabrics are:

• Acid dyes,
• Metal complex dyes (premetallised dyes),
• Chrome dyes, and,
• Reactive dyes.
Other classes of dyestuffs, such as direct dyes, vat dyes, sulphur dyes, basic dyes and disperse dyes are not suitable for application to wool for a variety of reasons including poor colourfastness to washing and light. The highly alkaline reductive dyebaths used to apply vat and sulphur dyes to cotton would severely damage wool.

**Acid dyes**

Acid dyes are so-called because, in the history of dyeing, protein fibres (wool, mohair, silk, etc) were pre-treated with acid prior to dyeing. The acid pre-treatment of the protein fibres “released” the dye sites, thereby increasing the forces of attraction between the acid dye and the fibre.

Protein fibres are polypeptides, with long polymer chains terminated at one end with an amino group and at the other end with a carboxylic acid group, as represented below.

\[
\text{H}_2\text{N} \quad \text{--------------------} \quad \text{COOH}
\]

However, for a variety of chemical and thermodynamic reasons, these end groups exist in nature as charged pairs, viz. an ammonium cation and a carboxylate anion, as represented below. These ion pairs are also known as Zwitter Ion pairs.

\[
{+\text{H}_3\text{N} \quad \text{--------------------} \quad \text{COO}^-} \quad {\text{H}_3\text{N} \quad \text{--------------------} \quad \text{COO}^-}
\]

The polymer chains do not carry any net charge because each ammonium cation is balanced by an adjacent carboxylate anion, as represented below.

\[
{+\text{H}_3\text{N} \quad \text{--------------------} \quad \text{COOH}} \quad {+\text{H}_3\text{N} \quad \text{--------------------} \quad \text{COOH}}
\]

\[
\uparrow \quad \uparrow \\
\text{Cl}^- \quad \text{Cl}^- 
\]

It should be noted that the fibre is not charged positive, because the counter anion of the acid, chloride anion in the above example, balances the charge.

Thus we have freed up the ammonium cations which can now act as dyesites to attract the negatively charged dye anions via Coulomb’s Law.

The dye anion is far more hydrophobic in character than the chloride anion and so the dye anion progressively displaces the chloride anions from the ammonium cation dye sites. So, after dyeing, the situation inside the fibre would be:

\[
{+\text{H}_3\text{N} \quad \text{--------------------} \quad \text{COOH}} \quad {+\text{H}_3\text{N} \quad \text{--------------------} \quad \text{COOH}}
\]

\[
\uparrow \quad \uparrow \\
\text{Dye}^- \quad \text{Dye}^- 
\]

Of course, the counter anion will depend on the selected acid, e.g. sulphuric acid (sulphate anions), formic acid (formate anions) and acetic acid (acetate anions), but the principal is the same.
Thus high levels of salt, usually as sodium sulphate (Glauber’s Salt), can be used as a levelling agent. The sulphate anions compete with the dye anions for the dyesites, slowing down the dyeing process, and allowing time for the dyestuff to level throughout the wool mass.

Acid dyes have been divided up into four classes on the basis of the strength of the acid required to achieve adequate dyeing affinity. In general, the weaker the acid employed to achieve the dyeing, then the higher is the colourfastness to washing, but the more difficult it is to achieve a level dyeing.

In addition, the dyeing mechanism changes as we progress from Class 1 to Class 4 acid dyes. A very simplistic explanation would be that Class 1 Acid Dyes are attracted to the fibres mostly via the Coulombic mechanism whereas the Class 4 Acid Dyes are attracted to the fibres mostly via the hydrophobic mechanism. The four classes of acid dyes are summarised in Table 27.1.

Table 27.1 Acid Dye Classes for Wool Fabrics. Source: Pailthorpe, 2006.

<table>
<thead>
<tr>
<th>Dyeing Class</th>
<th>Description</th>
<th>Acid</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Level dyeing, or equalising acid</td>
<td>Sulphuric Acid</td>
</tr>
<tr>
<td></td>
<td>dyes</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Fast acid dyes</td>
<td>Formic Acid</td>
</tr>
<tr>
<td>3</td>
<td>Acid milling dyes</td>
<td>Acetic Acid</td>
</tr>
<tr>
<td>4</td>
<td>Supermilling dyes</td>
<td>None</td>
</tr>
</tbody>
</table>

2:1 pre-metallised dyes for wool, silk and nylon would most probably be Class 3 or Class 4 acid dyes. Acid dyes have average to good colourfastness properties on wool.

Metal complex dyes
Metal complex dyes, sometimes referred to as premetallised dyes or premets, have been synthesised by the dyestuff manufacturer with the inclusion of a chelating metal atom. In the case of wool premets, the metal is usually chromium although cobalt has also been used in the past. Metal complex dyes have good wet colourfastness properties and good colourfastness to light. The presence of the metal atom in the molecular structure is believed to be responsible for the improved light fastness.

Metal complex dyes are classified into two groups:

- 1:1 metal complex dyes. These dyes have been synthesised using one dye molecule and one metal atom and are usually applied from a strongly acid bath (pH 2).
- 2:1 metal complex dyes. These dyes have been synthesised using two dye molecules and one metal atom and are usually applied from a neutral to weakly acid bath (pH 6-7). 2:1 metal complex dyes are about twice the molecular size of 1:1 metal complex dyes and hence are more difficult to level on wool.

Chrome dyes
In the first instance, chrome dyes are acid dyes that have been selected on the basis of their molecular structure as being able to chelate (complex) with chromium (III). Thus chrome dyeing is a two stage process:

- The dyestuff is first applied in much the same way as an acid dye using acetic acid at the boil.
- The dyebath is cooled down to about 80°C, after which time the dichromate is added to the dyebath. The temperature is then raised to the boil to complete the chelation process.

The chromium is usually added to the dyebath in the form of sodium or potassium dichromate. Sodium dichromate is hygroscopic whereas potassium dichromate is not hygroscopic but more expensive. Whilst the dichromate anions are absorbed by the wool; the dye-metal complex is formed with chromium (III). Thus the dichromate anions must be reduced by the wool as follows:

\[
\text{CrO}_7^{2-} + 14\text{H}^+ \rightarrow 2\text{Cr}^{3+} + 7\text{H}_2\text{O}
\]
Clearly, therefore, some oxidation damage take place to the wool, but this is minimised by dropping the temperature to 80°C before the addition of the dichromate.

The chromium (III) not only combines with the dye molecules, but also with the wool, making the chrome dyeings very fast to both wet treatments and to light.

Various so called “low chrome” dyeing recipes have been devised that reduce the amount of chromium (III) in the effluent to less than 1 ppm. Even so, chromium (III) in the dyehouse effluent can have a severe environmental impact on aquatic life and hence many countries now prohibit the use of chrome dyes.

**Reactive dyes**

Reactive dyes are so named because they chemically react with the wool protein via either nucleophilic substitution or addition reactions. In this way a covalent bond (a permanent bond) is formed between the reactive dye and the wool fibre. Reactive dyes are capable of providing brilliant shades; but the down side is that they are expensive as compared to acid dyes.

Many reactive dyes for wool are based upon halogen chemistry, i.e. chlorine and bromine. Many countries now place very strict restrictions on the levels of AOX (Absorbable Organo Halogens) permitted in dyehouse effluents; so tight controls must be imposed on wool dyehouse effluents.

An example of a nucleophilic addition reaction that does not produce AOX in the effluent is that of the β-sulphatoethyl sulphone based reactive dyes; that are, in effect, blocked vinyl sulphone derivatives. The reactive group is activated at elevated temperatures, even under mildly acidic conditions as follows:

\[
\text{OH}^- + \text{D-SO}_2\text{CH}_2\text{CH}_2\text{OSO}_3^- \text{Na}^+ \rightarrow \text{D-SO}_2\text{CH=CH}_2
\]

Where: D represents the chemical constitution of the chromophore of the dyestuff.

The nucleophile in the wool, being the $\text{–NH}_2$ amino group, then reacts with the vinyl sulphone reactive group by nucleophilic addition as follows:

\[
\text{WOOL – NH}_2 + \text{D-SO}_2\text{CH=CH}_2 \rightarrow \text{WOOL – NH-CH}_2\text{CH}_2\text{SO}_2\text{-D}
\]

The reactive dyestuff has now become permanently fixed to the wool substrate. Because of this permanent fixation, reactive dyes can be difficult to level and hence levelling agents (usually amphoteric in nature) must be used.

**Summary of dye properties**

Table 27.2 summarises the properties of the various dyes used for the dyeing of wool fabrics. The dyer will choose the dyestuff, or combination of dyestuffs, that will match the required shade, meet the colourfastness specifications required for the end use and be cost effective.

**27.2 Dyeing auxiliaries**

Dyeing auxiliaries, also known as dyeing assistants, play many important roles in the dyeing of wool fabrics, including achieving:

- The correct shade,
- A level dyeing,
- Adequate colourfastness, and,
- Minimal damage to the wool.
The dyebath may also contain speciality chemicals that will assist the dyeing process in other ways, e.g. anti-foaming agents, de-aerating agents, etc.

In the dyeing of wool fabrics, the achievement of a level dyeing is of critical importance. Levelling agents therefore play a critical role in the application of dyes to wool fabrics.

Table 27.2 Summary of dye properties. Source: Pailthorpe, 2006.

<table>
<thead>
<tr>
<th>Dye type</th>
<th>Shade range</th>
<th>Levelling ability</th>
<th>Wash fastness</th>
<th>pH range</th>
<th>Relative cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid levelling</td>
<td>Bright pastels</td>
<td>Excellent</td>
<td>Poor</td>
<td>2 – 3.5</td>
<td>Cheap</td>
</tr>
<tr>
<td>Acid milling</td>
<td>Bright pastels</td>
<td>Poor</td>
<td>Very good</td>
<td>6 – 7</td>
<td>More than levelling</td>
</tr>
<tr>
<td>1:1 Metal complex</td>
<td>Dull, dark</td>
<td>Good</td>
<td>Good</td>
<td>2</td>
<td>Moderate</td>
</tr>
<tr>
<td>2:1 Metal complex</td>
<td>Dull dark</td>
<td>Poor</td>
<td>Very good</td>
<td>6 – 7</td>
<td>More than 1:1</td>
</tr>
<tr>
<td>Reactive</td>
<td>Bright pastels</td>
<td>Poor</td>
<td>Excellent</td>
<td>3 then 6-7</td>
<td>Expensive</td>
</tr>
</tbody>
</table>

Levelling agents

Levelling agents work in essentially two ways; they either:

- Compete with the fibre for the dyestuff, or,
- Compete with the dyestuff for the fibre.

In the former case the levelling agent forms a temporary complex with the dye in solution, thereby slowing down, or retarding, the adsorption of the dye by the fibre. As the dyeing proceeds the retarding agent/dye complex gradually breaks down allowing the dye to be adsorbed by the fibre.

In the latter case the levelling agent enters the fibre and temporarily occupies the dye sites. By adding large amounts of the levelling agent, e.g. sodium sulphate, the equilibrium is shifted, favouring increased dye in solution. This equilibrium is shown in the scheme below where Wool$^{2+}$ represents a cationic dye site and D$^{2-}$ is a dye anion. As we increase the concentration of sulphate anions, we “push” the equilibrium to the left hand side of the equation, with more free dyestuff in solution.

$$\text{Wool}^{2+} \cdot \text{SO}_4^{2-} + D^{2-} \leftrightarrow \text{Wool}^{2+} \cdot D^{2-} + \text{SO}_4^{2-}$$

27.3 The dyeing process

The dyeing process follows a time/temperature profile that is appropriate for the selected dyestuff and dyeing auxiliaries. Typical time/temperature profiles are given in Figure 27.1 for a conventional dyeing recipe (yellow) and a “rapid” dyeing recipe (red).

The conventional dyeing sequence (yellow) usually begins at about 25-50°C with the addition of the dyeing auxiliaries including wetting agent, acid, salt, levelling agent, etc. The liquor is circulated for 10-15 minutes to allow sufficient time for the dyeing auxiliaries to be uniformly absorbed by the wool. Not only has the wool absorbed the dyeing auxiliaries, but also water from the dyebath, causing the wool to swell. The swelling of the wool fibres makes it much easier for dyestuffs to penetrate the molecular structure of wool.

Once the dyer is satisfied that equilibrium has been reached, the pre-dissolved dyestuff is added to the dyebath from a separate mixing tank. At this stage the dyebath is still at about 25-50°C. The dye molecules now approach and interact with the wool fibre surface, as shown schematically in Figure 27.2. The temperature of the dyebath is now slowly raised to the boil (100°C) over a period of 30-45 minutes, during which time the dye diffuses into the body of the wool fibres.
The dyes may diffuse directly through the scales of the wool into the cortex. This is known as transcellular diffusion and is depicted in Figure 27.3 by the orange arrow. Alternatively the dye may diffuse through the intercellular cement between the scales. This is known as intercellular diffusion and is depicted in Figure 27.3 by the blue arrows.

Boiling is continued for a further 30-60 minutes, subject to dye type, to achieve migration of the dye both within and between fibres, thereby achieving a level dyeing.

Finally the dyes are fixed within the wool fibres by either physical or chemical means. Acid dyes and premets are fixed by simply stopping migration. The dyer achieves this by dropping the dyebath and rinsing in cold water. Chrome dyes are fixed by the addition of dichromate to the dyebath; while reactive dyes are fixed by causing the chemical reaction to take place between the reactive dye and the wool fibre.
27.4 Dyeing methods

There are essentially two dyeing processes employed in the dyeing of wool fabrics, being:

- Exhaust dyeing, and,
- Continuous dyeing.

In exhaust dyeing we begin the process with a set volume of dye liquor; as determined by the liquor ratio. The liquor ratio, also known as the liquor goods ratio, is the volume of liquor (litres) in any treatment to the weight of material being treated. Subject to the design of the dyeing machine; the liquor ratio can vary from 3:1 to 50:1 (and higher). A low liquor ratio is sometimes referred to as a “short” liquor ratio. On the other hand, a “long” liquor ratio is a high liquor ratio. During the dyeing process, the dyestuff moves from the dyebath to fibre (as discussed above) and is said to “exhaust” onto the fibre. Exhaust dyeing is used for the dyeing of wool fabrics in dyeing machines such as winches, beam dyeing machines, jigs and jet dyeing machines. These machines are covered in more detail in Lecture 31.

In continuous dyeing operations the dye liquor is applied by passing the fabric through a trough of dye liquor followed by pad mangle, spraying with dye liquor and foam application. Rather than liquor ratio, we now speak in terms of pick-up. The pick-up is usually quite low, being equivalent to a liquor ratio of 1:1 or less. Obviously the very “short” liquor ratio leads to substantial savings in water, energy and effluent costs. The dye impregnated fabric is then passed through a steamer to fix the dye to the fabric, followed by washing off. Continuous dyeing methods are mostly used for the dyeing of large lengths of fabric for mass produced articles; and are rarely employed for the dyeing of wool fabrics. Thus continuous dyeing methods will not be considered further in these lectures.

27.5 Colourfastness properties

Consumer complaints about textiles and articles made from textiles fall generally into three categories. In order of most frequently occurring, these are:

- Manufacturing faults, e.g. poor seam strength,
- Poor colourfastness, and,
- Poor dimensional stability (shrinkage in use).
Thus colourfastness is important to both the manufacturer and the consumer.

**Colour fastness testing**
The major problems with colourfastness testing are:

- The specification of colour and the measurement of colour differences.
- Devising test methods which give reproducible test results and which are representative of, or imitate, user conditions.
- The decision as to the degree of colour change that is tolerable.

The specification of colour and its measurement are large topics on their own and are beyond the scope of this lecture. Only sufficient will be said about them to permit colourfastness testing to be understood.

In the first instance it suffices to say that, whilst objective instrumental measurement of colour and colour differences is probably the ideal method of assessing colour changes in processing and end use, spectrophotometers, colorimeters, etc are expensive pieces of equipment and are not always available to the manufacturer. The consumer certainly does not have available to them such instrumentation and relies on their eye (a subjective measurement) to assess colour changes and/or colour differences.

From a practical or commercial point of view there are two aspects of colour change in which the consumer is generally interested:

- a change of shade or hue of the material, and,
- staining, by dye transfer, of other materials with which the fabric may have had contact.

Thus, two assessment scales have been developed, grey in colour, for assessing these two aspects of colour change. These scales are known as "Grey Scales". Both are based on the visual acuity of what is called the standard observer.

**Grey scales**
Grey Scales for assessing a change of shade (or colour) consist of 5 pairs of grey coloured chips each representing a specified visual difference. The differences are specified in standard NBS units as given in Table 27.3. (NBS - National Bureau of Standards [USA]).

<table>
<thead>
<tr>
<th>COLOUR DIFFERENCE</th>
<th>FASTNESS RATING</th>
</tr>
</thead>
<tbody>
<tr>
<td>NBS UNITS</td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>5</td>
</tr>
<tr>
<td>1.5</td>
<td>4</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>6</td>
<td>2</td>
</tr>
<tr>
<td>12</td>
<td>1</td>
</tr>
</tbody>
</table>

A rating of 5 is represented on the scale by two identical neutral grey chips mounted side by side; each having a total reflectance over the visible spectrum of 12±1%. The colour difference is zero.

Fastness ratings 4 to 1, inclusive, are represented by a "reference" chip identical to those used in 5, paired with similar but lighter neutral grey chips, the visual differences in pairs being in geometric steps of colour difference or contrast as given in Table 27.3.

Grey Scales for assessing staining consist of 1 pair of white, and 8 pairs of grey and white colour chips each representing a visual contrast. The differences are again given in NBS units and numerical fastness ratings.

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Table 27.4 Colour Differences for Assessing Staining.
Source: National Bureau of Standards, USA.

<table>
<thead>
<tr>
<th>COLOUR DIFFERENCE NBS UNITS</th>
<th>FASTNESS RATING</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>5</td>
</tr>
<tr>
<td>2</td>
<td>4-5</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>6</td>
<td>3-4</td>
</tr>
<tr>
<td>8</td>
<td>3</td>
</tr>
<tr>
<td>12</td>
<td>2-3</td>
</tr>
<tr>
<td>16</td>
<td>2</td>
</tr>
<tr>
<td>24</td>
<td>1-2</td>
</tr>
<tr>
<td>32</td>
<td>1</td>
</tr>
</tbody>
</table>

A fastness rating 5 is represented on the scale by two identical white chips mounted side by side having a total reflectance of not less than 85%. The colour difference is zero.

Fastness ratings 4/5 to 1 inclusive are represented by a reference white chip identical to that used for rating 5, paired with similar but neutral grey chips, the visual difference in the pairs being in steps of colour difference or contrast as shown in Table 27.4.

The Grey Scales are used as follows:

- For assessing colour change, a piece of the original untested material is placed side by side with the tested material and, under standard conditions of examination, the difference between tested and untested is compared by eye with the colour differences represented by the steps of the Grey Scale. The fastness rating of the test specimen is then that number on the Grey Scale which corresponds to the contrast between the original and treated specimens. If the rating to be given lies between two of the contrasting chips on the grey scale, e.g. between 3 and 4, then it is given an intermediate rating expressed as 3 to 4 (3 1/2 or 3/4).

- For assessing staining, a piece of the unstained undyed textile and the treated undyed piece are placed side by side and, under standard conditions of illumination, are compared with the Grey Scale for staining for visual differences. The fastness rating on the Grey Scale of the specimen is that number on the Grey Scale corresponding to the contrast between the original and stained specimens.

Since perfect colourfastness is an unobtainable ideal, what levels of fastness with respect to both colourfastness and staining are permissible in a textile material?

Most manufacturing specifications, as well as some consumer or performance specifications, do contain clauses specifying colourfastness to various agencies. In general, however, 4 with respect to colour change and 3-4 with respect to staining are suggested as being the satisfactory limit beyond which an inordinate level of consumer complaint is likely to occur. Ratings above 4 for colour change are regarded as being satisfactory performance and ratings below 4 are regarded as unsatisfactory; and likely to result in consumer complaint. The rating also helps in decisions with respect to the severity of the test to be applied and it especially enables sensible decisions with respect to the care instructions to be selected for care-labelling purposes.

For example, a fabric that has a rating of 2, in a washing test equivalent to machine laundering, may have a rating of 4, or even 5, in a test equivalent to hand washing. Thus, in this example, for satisfactory performance in use, the garment manufacturer should select a care label instructing the consumer to “Handwash”. They would be foolish to select one permitting or instructing the consumer that machine washing was satisfactory.
**Wet fastness testing**

Colourfastness testing generally consists of three steps:

- Sample preparation,
- Test method, and,
- Assessment of the result.

Generally one pattern from a given lot, provided that it includes all shades or colours, is usually sufficient. If the material to be tested is fabric, it is usually sandwiched between two other undyed materials (in most cases undyed cotton and undyed wool, or cotton and a multi-fibre fabric) and sewn so that both colour change and staining can be tested.

The tests, in general, tend to imitate user conditions. They must therefore be simple and also reproducible. Since it is difficult to accurately reproduce all user conditions, standard tests and standard materials (for staining) are therefore used in conjunction with the test sample.

The International Standard ISO 105 - "Textiles - Tests for Colourfastness" lists 54 (fifty four) separate methods for colourfastness testing as well as 4 (four) standards on general principles of colourfastness testing.

Obviously, all of these tests cannot be described in detail in this lecture. Consider, as an example of colourfastness testing, Australian Standard AS.2001.4.15 "Determination of Colourfastness to Washing" which describes 22 (twenty two) separate methods covering simulated hand-washing and machine laundering of various degrees of severity, at different temperatures and using either soap, soap and soda, soap and bleach, detergent, detergent and soda, detergent and perborate, etc.

Tests are carried out in either a "Washwheel" or a "Launderometer", a machine in which containers holding the specimens, and which have a capacity of 500 ml, can be rotated end over end about 40 times per minute in a constant temperature water bath. Test temperatures range from 40 ± 2°C to 95 ± 2°C. Washing times range from 30 minutes to 240 minutes. In some of the more severe tests up to 25 stainless steel balls are included to provide severe mechanical buffeting of the test specimens.

Composite test specimens, prepared as previously described, are placed in the selected test solution at a liquor ratio of 50:1 by weight. The sealed container is placed in the machine and rotated for the appropriate time at the appropriate temperature.

After washing, the specimen is removed from the container and rinsed in cold, running, filtered tap water for 2 min. Squeezed lightly to remove excess water, the seams of the composite specimen are unpicked and the specimen dried in air not exceeding 60°C.

Specimens are then pressed lightly (to remove creases) with a warm hand iron and reconditioned in a standard atmosphere. Assessment for colour change and staining of undyed cloths then takes place using the Grey Scales and the methods already described.

**Colour fastness to rubbing**

Colourfastness to rubbing is determined by rubbing the dyed specimen with a dry, undyed cotton cloth and with a wet, undyed cotton cloth under standard conditions.

The testing machine is called a Crockmeter and it may be either motorised or manually operated. As can be seen in Figure 27.4, the white cotton cloth is placed over a cylindrical wooden or metal peg. The total vertical force exerted by the peg on the specimen is 9.0 ± 0.2 Newtons; and the length of a single stroke is 103 ± 5 mm. The peg is then moved back and forth over the test specimen for ten cycles. The staining of the undyed (white) cotton cloths is then evaluated using the standard Grey Scales for staining.
Colourfastness to light

The changes in hue of coloured materials, especially dyed fabrics, and their degradation (mainly loss of mechanical properties) on exposure to radiation, and in weathering, are so obvious that it is not surprising that they are frequently the cause of consumer complaint and have also been the subject of scientific investigation since the earliest period of modern chemistry.

The sources of radiation used for colourfastness testing are:

- Daylight - with and without protective glass covers.
- Artificial light sources:
  - carbon arcs,
  - Xenon high pressure discharge tubes,
  - Fluorescent tubes, and,
  - MBTL lamps - (mercury vapour, tungsten filament internally phosphor coated lamp.)

Global radiation is the sum of direct radiation from the sun and reflected radiation from the sky on the horizontal plane. There are many variables in outdoor exposures; viz:

- Geographical latitude of the exposure site - the closer to the equator usually the more severe.
- Altitude - the higher above sea-level the more severe.
- Local Geographical Features - temperature, wind (drying), proximity of water (dew).
- Random year to year variation – this can cause as much as a 2:1 effect in successive years at the same location.
- Seasonal Variation - winter exposures may be only 1/7th as severe as summer exposures.
- Orientation of the sample - South V's North, Angle to Horizontal.
- Sample Insulation - backed samples (insulated) often degrade faster than unbacked ( uninsulated) samples.
- Operating Cycles - hours of sunlight, condensation, rain, humidity, etc.
- Operating Temperature - higher temperatures usually cause greater degradation.
- Material being exposed.

Hence it is more common to conduct lightfastness testing using an artificial light source, e.g. the MBTL lamp shown in Figure 27.5.
Blue scales

The ISO (International Standards Organisation) blue fabrics are a series of seven (7) blue dyed wool fabrics numbered one (1) to seven (7) of known fading behaviour when exposed to light. They are intended to be uniformly spaced by a factor of 2, meaning that each standard should require an exposure twice as severe as the one below it to show the same degree of change. Blue #1 has almost zero fastness to light, it shows a slight but perceptible change after an exposure of only a few hours in bright sunshine. Blue #7 shows little or no change after quite long periods exposed to bright sunshine. The test procedure is as follows:

- Mount test specimens and Blue standards.
- Cover with an opaque mask so that half of each specimen and half of each standard is covered.
- Expose continuously using the chosen light source.
- As irradiation proceeds, make observations of the specimen and Blue standards. When a change in colour of the test specimen can just be perceived note the number of the Blue standard just showing a perceptible change.
- Continue the exposure until Blue standard 7 shows a change in colour equal to Grade 4 on the Grey Scale.
- Compare the change in colour on the test specimen with the change of colour of the standards and record the number of the standard showing the same colour change.

Figure 27.6 provides an example of an exposed set of Blue Scales, with Blue #1 at the top and Blue #7 third from the bottom. The bottom most two samples are the test samples. It can be readily seen that the tested blue sample (2nd from the bottom) has excellent light fastness (negligible fading); while the maroon sample has faded slightly and would be rated 5-6.
The minimum lightfastness requirements for a particular textile product depend upon the expected solar exposure of the product in use. Thus textiles for indoor use can have low lightfastness ratings whereas textile products intended for outdoor use or window dressings require much higher lightfastness ratings.

Table 27.5 provides some examples the minimum lightfastness ratings required for various textile products. For textile products with lightfastness ratings of 4 or less, the Care Label should contain the warning “Dry in the Shade”.

Table 27.5 Minimum Light Fastness Ratings for Selected Products.  

<table>
<thead>
<tr>
<th>TEXTILE PRODUCT</th>
<th>FASTNESS RATING</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nightwear, Underwear</td>
<td>4</td>
</tr>
<tr>
<td>Socks, Blankets, Pullovers, Cardigans, Suits, Tunics</td>
<td>4</td>
</tr>
<tr>
<td>Scarves, Gloves, Shirts, Blouses</td>
<td>4-5</td>
</tr>
<tr>
<td>Skirts, Overcoats, Upholstery Fabrics</td>
<td>5</td>
</tr>
<tr>
<td>Carpets, Curtain Fabrics</td>
<td>6</td>
</tr>
</tbody>
</table>
Readings


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Summary

Summary Slides are available on CD

Wool fabrics can be readily dyed with acid dyes, premetallised dyes, chrome dyes and reactive dyes to achieve a wide palette of colours with good colour fastness properties. Subject to the class of dyestuff, the dyebath will also contain additives such as acids, salts, levelling agents, anti-foaming agents, etc.

Dyeings typically start at 20-50°C with the addition of the dyeing auxiliaries. The dyeing machine is run for about 10 minutes after which time the pre-dissolved dyestuff is added. The temperature is then raised to boil over 30 minutes and held at the boil for 30-60 minutes. Finally, the dyed fabric is thoroughly rinsed in cold water.

In the case of chrome dyes, the dyes are fixed by the addition of potassium dichromate. Reactive dyes are fixed by initiating the chemical reaction between the reactive dye and the nucleophilic dye sites.
References


National Bureau of Standards, USA., (NBS). Standards used by NBS to assess colour differences, change of shade and staining. NBS, Gaithersberg, USA.


Glossary of terms

<table>
<thead>
<tr>
<th>Term</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adsorption</td>
<td>Temporary holding of dye molecules on the surface of a fibre</td>
</tr>
<tr>
<td>Affinity</td>
<td>The degree of attraction of a dye molecule for a fibre</td>
</tr>
<tr>
<td>Approach</td>
<td>The movement of dye molecules towards the fibre due to its affinity</td>
</tr>
<tr>
<td>Colour matching</td>
<td>The procedure of comparing dyed samples with a standard in order to achieve the correct shade in dyeing</td>
</tr>
<tr>
<td>Continuous dyeing</td>
<td>Dyeing of a fabric or carpet as it passes continuously through a vessel of dye liquor</td>
</tr>
<tr>
<td>Diffusion</td>
<td>Movement of dye molecules into the fibre</td>
</tr>
<tr>
<td>Dispersion</td>
<td>Separation of clumps into tiny particles that can then spread throughout the liquid</td>
</tr>
<tr>
<td>Dyeing assistant</td>
<td>A chemical added to the dyebath to assist in the dyeing process</td>
</tr>
<tr>
<td>Dyebath</td>
<td>The solution, usually water, containing the dyes, dyeing assistants and any other chemicals required for dyeing</td>
</tr>
<tr>
<td>Dyestuff</td>
<td>An alternative term to dye</td>
</tr>
<tr>
<td>Exhaust dyeing</td>
<td>The use of a dyebath of a relatively large liquor-to-goods ratio in which the fibres are immersed for some time, allowing the molecules to leave the bath and attach to the fibres</td>
</tr>
<tr>
<td>Exhaustion</td>
<td>The leaving of the dye from a dyebath and becoming attached to the fibre being dyed</td>
</tr>
<tr>
<td>Fastness</td>
<td>The ability of a dyed substrate to retain its colour under the action of light, washing, rubbing, perspiration, etc.</td>
</tr>
<tr>
<td>Fixation</td>
<td>A dye molecule becomes firmly fixed in one site</td>
</tr>
<tr>
<td>Glauber’s Salt</td>
<td>Sodium Sulphate</td>
</tr>
<tr>
<td>Ion</td>
<td>A group of atoms (i.e., molecule) that carries a net positive or negative charge</td>
</tr>
<tr>
<td>Levelness</td>
<td>The degree of uniformity of dyeing throughout a batch</td>
</tr>
<tr>
<td>Liquor-goods ratio</td>
<td>The ratio of liquor to goods (on the basis of the respective masses) in a dyeing process</td>
</tr>
<tr>
<td>Migration</td>
<td>Dye molecules moving to other sites to achieve a more level dyeing</td>
</tr>
<tr>
<td>pH</td>
<td>A measure of the hydrogen ion concentration in an aqueous solution. If the hydrogen ion concentration is high (i.e., acid conditions) pH → 1; if the hydrogen ion concentration is low (i.e., pH → 14); a neutral solution such as pure water has pH 7.</td>
</tr>
<tr>
<td>Polar molecule</td>
<td>A molecule which has positive and negative charges on opposite sides (or at opposite ends)</td>
</tr>
<tr>
<td>Solution</td>
<td>A liquid containing molecules dispersed throughout it that were originally in the solid or gaseous state</td>
</tr>
<tr>
<td>Substrate</td>
<td>The undyed material (fibres, yarn, sliver, top, fabric, carpet) submitted to a dyeing process</td>
</tr>
</tbody>
</table>