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United Nations Industrial Development Organization

Expert Group Meeting on Quality Control in the Textile Industry

## QUALITY CONTROL IN THE FINISHING OF COTTON $\frac{1}{2}$

by

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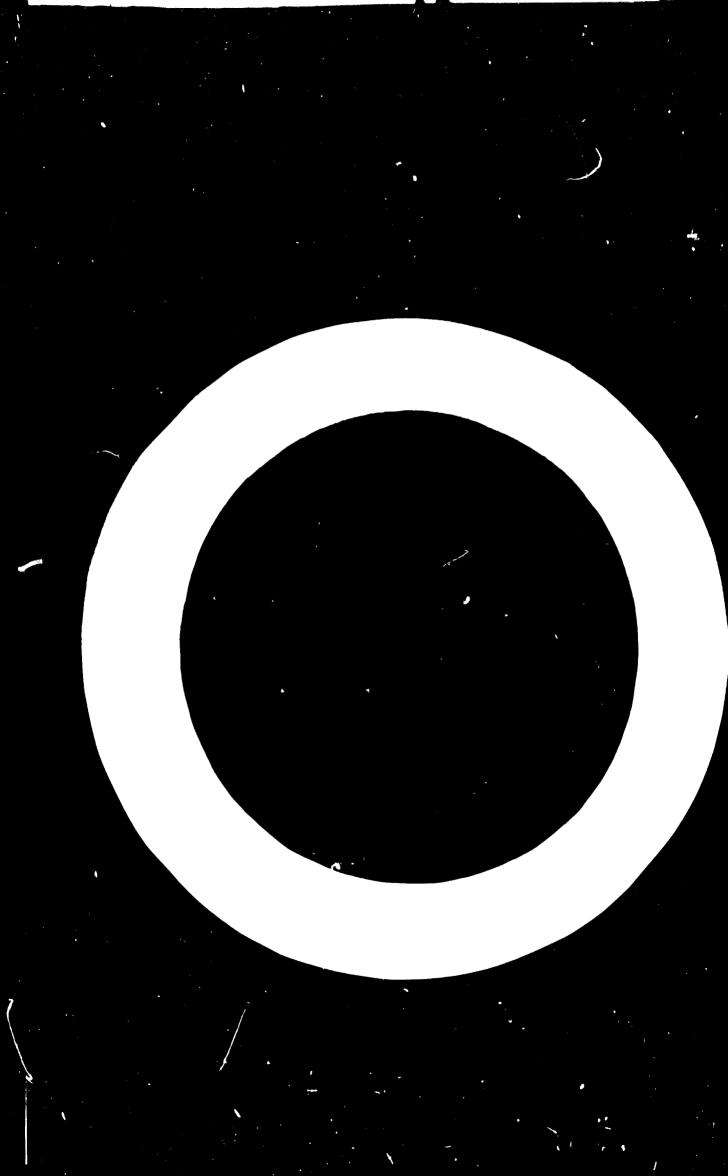
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In any industry, systematic quality control is necessary to maintain product quality and to control costs. To be effective without being exorbitant in cost the quality control programme has to be planned with care and imagination to suit the specific requirements of a given industry. It is also necessary to give serious thought to the most appropriate position for the quality control laboratory in the organisational structure and to develop proper reporting systems so that the information supplied by this laboratory is utilised to the maximum advantage of the organisation.

This paper deals with quality control in the chemical processing departments of textile mills, recommends various activities which this function can usefully perform, suggests the most important checks and controls of quality that need to be regularly undertaken and describes briefly suitable test procedures which can be adopted.

Chemical processing of textiles refers to the processes of bleaching, mercerizing, dyeing, printing and finishing. Inasmuch as these processes convert the loom state fabric into its marketable form, the care with which they are carried out will influence the marketability of the fabric and the price that it

can fetch. Furthermore, expenditure on dyes, auxiliaries, chemicals etc. in a textile mill is a significant element of the cost of production. From both these considerations, it is vital that quality control is followed in a regular and systematic manner to ensure that materials of the appropriate quality are supplied to this department at economic costs and that the various processes that are carried out by this department maintain required standards of quality.

For convenience, the paper is divided into three sections, each dealing with a particular activity which is considered necessary and desirable for effective quality control. These activities are : (1) Testing of Raw Materials; (2) Quality Control of Good9-in-Process; and (3) Inspection of Finished Products.

#### (1) TESTING OF RAW MATERIALS

A textile mill consumes various types of dyestuffs, finishing agents, auxiliaries and chemicals in its chemical processing departments. Expenditure on these materials (in an average Indian mill) amounts to approximately 5 to 8 per cent of the total manufacturing cost. It is, therefore, very important that they are purchased at the most economical cost. Besides the cost aspect, the quality of processing is influenced by the quality of the materials supplied to the departments. Materials which are sub-standard or which do not satisfy the exact requirements can lead to finished textiles which are defective or which do not match

the market requirements in terms of the prescribed shade of colour, feel and handle of fabric, or particular functional properties. Such a situation would mean either reprocessing of the defective textiles at extra cost or marketing the product at a discount. In either case, significant financial losses to the mill are involved.

It is frequently observed that decisions regarding purchase of these materials are made purely on the basis of price without taking quality into consideration. In most materials used for textile processing, the active ingredient has to be necessarily mixed with diluents and is, therefore, subject to variation in concentration unless the manufacturer or supplier of these materials employs ricorous quality control. Decisions about the most economic purchase must, therefore, take into account both price and quality aspects.

Since an average mill purchases a large number of dyestuffs and chemical products, it is necessary to plan the quality control programme for testing of raw materials with some discrimination so that the testing load does not become uneconomical. For regular testing, such items need to be picked out which are either the more expensive ones or which are critical in terms of their effect on processing quality. In the following paragraphs, the testing of such selected materials is discussed.

#### DYESTUFFS

The important qualities for which dyes need to be tested regularly are strength, purity and compatibility. A dyestuff is

never marketed as a pure chemical product since this is unsuitable for industrial dyeing or printing purposes. The commercial dye is invariably a mixture of the pure dye with dextrin and a salt such as Glauber salt. Dye manufacturers, therefore, market the same dye in different qualities in which the proportion of the pure dye varies. Each quality bears a distinctive mark which expresses the strength of the active ingredient viz., the pure dye. There is no international standardisation about the strengths at which a particular dye should be marketed by different manufacturers. The designations adopted by different dye manufacturers to identify dye strength are again not standard and vary from one manufacturer to another. Thus, even if a standard and reliable source of dyestuff supply is assured, it still becomes necessary to compare competitive samples of a dye from different manufacturers, to determine their relative strengths and hence to find out which would be the cheapest and most suitable dye. Often the supply of dyestuffs is not so reliable, especially when there is a shortage of a particular product, a situation which occurs not infrequently in developing countries.

#### Dye Strength

Different methods are available for evaluating the strength of a commercial dye. These can be classified into (1) matching, and (2) chemical or optical analysis. A mill uses dyestuffs belonging to many different classes such as direct, reactive, azoics, vat, soluble vat etc. The shade matching technique is applicable to all these classes of dyestuffs and is widely adopted in many mills.

Skeins containing standard weights of yarn are dyed under identical conditions with equal concentrations of the different dye samples which are to be evaluated and compared using conditions and procedures of dyeing recommended for the particular dyestuff. Machines such as the Marney Dyeing Machine are available which permit several dyeings to be done simultaneously under strictly controlled conditions of temperature, rate of stirring, etc. Since the different dyeings are done at the same time, under identical conditions, the depth of dyeings obtained is strictly dependent on the concentration of the active dye in the commercial product. The dyeings, after suitable washing, drying and conditioning, are compared either visually or by reflectince measurements. Having judged differences in strengths, if any, between the different samples of dyestuffs, a second set of comparative dyeings is undertaken in which one of the samples is taken as a standard and the concentration of the others suitably chosen to get the best match in shade. This procedure is repeated till dyeings which are comparable in depth are obtained. From the concentrations of the different sample dyes required to produce the same depth of dyeing, one can determine their relative strengths. Knowing the price and the relative strengths, it is thus easy to select the most economical product

Though this t onnique is apparently simple and widely applicable, it has certain limitations. Firstly, visual or even spectrophotometric comparisons cannot reliably detect differences in depths of dyeing lower than about 5%. Secondly, there are often small tone differences in the dyeings produced by competitive samples of the same dyestuffs from different suppliers. When the tone of dyeings

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is different, it is difficult and even hazardous to compare their strengths. In spite of these limitations, a judicious and careful use of this technique is very important in making decisions about purchase of dyestuffs.

In some cases, it is possible to use a simpler and objective method which depends on measurement of optical density of solutions of the dyestuffs. This method is eminently suited for direct and reactive dyes, which are all soluble in water and which produce in their aqueous solutions the same colour that will be finally obtained on the textile. The method, however, is not applicable for azoics and soluble wats for both of which the actual dye is developed or formed on the textile. Azoics are formed on the textile material by coupling of a Naphthol with a diazotized base or its salt. In the case of soluble vats, again, the dyestuff itself is practically colourless, and the actual dye is formed on the textile material by a development process using sodium nitrite and sulfuric acid. In the case of vat dyes, there is a different type of difficulty in adopting optical density measurement as a method of comparing competitive samples of dyes. This is because of the fact that dyes of different manufacturers may differ in their chemical structure and yet produce the same shade. Because of their different chemical structures, the colour of their leuco vats, "be form in which they are soluble, may be different. Comparison of optical densities is thus not possible.

In some cases, gravimetric or volumetric chemical analysis can also be used. Thus, naphthols can be volumetrically estimated against

standard solutions of an appropriate diazotised base and vice versa. Soluble wats can be estimated gravimetrically by precipitating the parent wat dye from the ester form of the soluble wat. These methods can, however, be applied only when the sample consists of a single dye and is not a mixture.

#### Dye Purity

Occasionally, a sample which is supplied as a single dye may actually be a mixture of more than one dye. For example, a little orange or pink dye may be added to a yellow or brown dye to give a slightly reddish tone to the dyeing which could be misjudged as a deeper dyeing. The purity of a dye sample can be checked by a variety of methods.

For vat dyes, a small quantity of the sample can be sprinkled in a porcelain dish containing concentrated sulfuric acid or containing the blank vat consisting of caustic soda and sodium hydrosulfite. Advantage is taken of the fact that the adulterant dye often develops a different colour than the main dye in the above reagents. In a slightly modified indirection of this method, a solution of the dye sample is prepared in caustic soda and hydrosulfite and a drop of it put on a filter paper kept under a glass plate with a small hole in the centre. Next an eluent mixture of pyridine, water, caustic soda and hydrosulfite is added to the filter paper. The glass plate is covered by a small watch glass to prevent premature oxidation of the leuco vat. The reduced vat solution on the filter paper is allowed to oxidise

after a few minutes. If the dye is a mixture, it will separate into different components and show rings of distinctly different colours on the filter paper. For soluble vats, a small quantity of the sample in powder form is blown on a wet filter paper and subsequently developed in a bath containing sodium nitrite and sulfuric acid. Again if the dye is a mixture, the filter paper will show different coloured spots.

"hese rudimentary paper chromatography methods can be applied more systematically for identification of dyes in powder form. In principle, the method consists in preparing a solution of the dyestuff and spotting it on a chromatography grade Whatman No. 1 filter paper. Suitable solvents are employed as eluents for different classes of dyestuffs. For vat dyes, the eluent has the following composition: Water: Turkey red oil (1% solution): sodium hydroxide (35%):sodiam hydrosulfite:Pyridine :: 8:12:1:1:4. For vat dyes, the method described above needs a slight modification. After the filter paper strip is spotted with the leuco solution of the dye, it is suspended from a hook in the rubber stopper of a test tube containing the above eluent mixture. The filter paper strip is kept taut by attaching small glass weights at its base. The test tube is placed in a water bath at 80°C. If the sample is a mixture, separation of the dyestuff will take place in two to four hours.

For solubilised vat dyes, three differ int eluent compositions are recommended of which the second gives the best results. The eluents are : (i) Ammonia (25%):Methanol:Water :: 1:2:3,

(ii) Pyridine: Isoamyl alcohol: Ammonia (25%) :: 1.3:1:1, and
(iii) Methanol: Acetic acid: Water :: 4:1:1. For direct dyes,
the eluent recommended is : Benzyl alcohol: Dimethylformamide:
Water :: 3:2:2. For reactive dyes, three different eluent
compositions are given below : (1) Butanol: Ethanol: Formic acid:
Water :: 50:25:15:10, (2) Water: Butanol: Ethanol :: 30:40:30, and
Phenol: Formic acid: Water :: 4:1:2. Eluent No. 2 is more suitable
for mono- and dichloro triazine dyes whereas 1 and 3 are more
suitable for the vinylsulphone class of dyes.

#### Dye Compatibility

The general practice in most dyeings is to employ a mixture of dyestuffs, single shades being very rarely demanded. When a mixture of dyes is used, especially in the dyeing of yarn in package form, it is very important to ensure that the different dyes used in the mixture are compatible with one another. Dyes of good migration behaviour and comparable dyeing rates are necessary for good compatibility. For some types of dyestuffs, compatibility data are available in published literature<sup>1</sup>. For example, Fox<sup>2</sup> has observed that for method II vat dyes, the following limits are necessary for compatibility:

Rate of dysing :  $t_{30} = 30$  to 41 seconds;  $t_{50} = 122$  to 168 seconds; and  $t_{80} = 600$  to 1104 seconds. Fixation = 59 to 63%.

Figures for rate of dyeing represent the time in seconds to give 30, 50 and 80% of dye firstion which is the amount of dye fixed in a one hour dyeing in a 30:1 bath at  $50^{\circ}$ C.

For dyes where similar data may not be available, a simple dip-dyeing test is very useful to judge compatibility. The dyebath is prepared using the different dyes in required proportions. From the same dye-bath, skeins of standard weight and quality are dyed successively. If the mixture is compatible, the successive dyeings will differ only in depth and not in tone.

#### FINISHING AGENTS

Textile finishes can be broadly classified into two groups, viz., starch-based finishes and chemical finishes, particularly resin finishing. For the former type of finishes, the major ingredients are a starch and a softening or stiffening agent depending on the fabric feel and handle required. Starches are normally available in a standard quality and do not require to be tested frequently or exhaustively. Standard specifications for starches are available from various national and international bodies which also prescribe the testing procedures. It may be necessary, however, to occasionally test starch samples for their paste viscosity behaviour since the amount of size picked up by the fabric during finishing and therefore the feel and handle of the fabric produced is dependent on the paste viscosity. A convenient instrument for studying the paste viscosity behaviour of starches is the Brabender viscograph which gives detailed information about the gelatinization temperature, peak viscosity, and changes in viscosity produced by stirring, storage and changes in temperature.

For softeners and stiffening agents, no suitable test procedures are available which can give quantitative information about

the softness, stiffness and feel of the fabric. When required, it is more useful to carry out actual finishing trials on small samples and evaluate these subjectively for feel etc.

When the finished fabrics are required to be mechanically preshrunk through a process such as sanforizing which uses a rubber blanket, it is necessary to ensure that no auxiliaries used in the finishing mixture contain mineral oil which softens the rubber blanket and adversely affects both the appearance and shrinkage behaviour of the fabric.

In the testing of chemical finishing materials, the most important products are the various 'resins' which are used for anti-crease, wash-and-wear and durable press finishes. Resins most commonly employed in these finishes are of the nitrogenous type such as dimethylol urea, dimethylol ethylene urea and dimethylol dihydroxy ethylene urea. It is necessary to test these products for (1) free formaldehyde and (2) per cent active ingredient. Since the resins are used in their methylol form which is produced by reaction of the parent compound such as urea or ethylene urea with formaldehyde in a reversible reaction, a certain amount of free formaldehyde is inevitably present in the final product. It is desirable that minimum free formaldehyde is present to avoid undue embrittlement of the fabric due to formation of methylene crosslinks, as well as to reduce the problem of obnoxious formaldehyde odour. Free formaldehyde in resins can be estimated volumetrically against standard sodium sulfite whereas total formaldehyde can be estimated colorimetrically using chromotropic acid in sulfuric acid solution which produces a violet colour with formaldehyde. The percentage concentration of the active ingredient can be

determined by estimation of nitrogen using the standard kjeldahl method. These chemical tests may also be supplemented by small scale finishing trials in which samples finished with the different resin samples are compared for crease recovery, strength loss, etc.

Occasionally, it is necessary to make a qualitative identification of the resin. Some qualitative tests based on colour reactions with reagents such as phenylhydrazine, ferric chloride and sodium hypochlorite are available.

Chromatography can also be conveniently used for qualitative identification. A 50% aqueous solution of the sample resin is spotted on chromatogra, by grade thatman No. 1 filter paper. Spots of reference standard resins are also put alongside. The paper is hung in a chamber saturated with vapours of eluent containing butanol:Ethanol:Water in the ratio of 4:1:1 for a few hours and preferably overnight. The paper is removed from the chamber, dried and sprayed with alkaline ferricyanide-nitroprusside reagent which produces different coloured smots with different resins. Comparing the colour developed by the test sample with that of the reference standards, the former can be quantitatively identified. Another recent technique converts the resin into its trimethylsilyl derivative which is volatile and can, therefore, be identified by gas-liquid chromatography. Infrared spectrophotometry can also be used for qualitative identification. Different functional groups show characteristic peaks of IR absorption.

#### TIBRES

The textile processor of today has to handle materials made from several fibres which require different conditions of processing, particularly of dyeing. It is, therefore, necessary for him to be able to identify the component fibres of a textile material and at times to carry out a quantitative estimation of the proportions of these fibres in the textile.

Many types of fibres have distinctive cross-sectional shapes and can therefore be identified microscopically. Similarly, IR spectra can also be used for quick qualitative identification of different fibres which exhibit absorption peaks at characteristic frequencies according to the particular functional group(s) present in the fibre. More recently, gas-liquid chromatography has been applied for fibre identification. Many man-male fibres depolymerize to give volatile products when they are heated. Cellulosic and other fibres which do not behave in this manner can be converted into suitable derivatives which volatalise on heating. The volatile products can be identified by appropriate application of gas-liquid chromatography.

Both qualitative and quantative estimation of fibre blends can be conveniently carried out also by taking advantage of their differing solubilities in different solvents. For example, in a blend of cotton or viscose with polyester, the cellulosic fibre would completely dissolve in cold concentrated sulfuric acid leaving the polyester unaffected. In a mixture of cellulosics or polyester with wool, the latter can be dissolved in concentrated sodium hydroxide.

Employing standard procedures of gravimetric analysis, it is thus possible to estimate quantitatively the proportions of the component fibres in blends. Detailed procedures and solvent systems to be used for different binary and tertiary blends of fibres are available in national and international standards (for example, Indian Standard Nos. 1889, 1564, 2005, 2005, 2176, 2177).

#### AUXILIARIES

These are chemical reagents which are used to assist speedy and uniform reactions in various textile chemical processes. Their classification is on their functional usage rather than on chemical structures. The most important types of auxiliaries used in textile processing are desizing agents, wetting agents, dispersing agents and levelling agents.

#### Desizing Agents

The active ingredient in these is an enzyme of the diastase, pencreatic or bacterial type. Enzymes used for desizing are all of the liquifying and not saccharifying type. In other words, their action on starch pastes is to produce a random breakdown of the starch molecules producing a rapid lowering of viscosity. A simple and convenient method of evaluating desizing agents is thus to measu reduction in viscosity of a standard starch paste produced by them at different concentrations. The viscosity changes are measured at suitable intervals over a period of one to two hours. From plots of concentration vs reduction in starch paste viscosity within a given time, say, one hour, it is possible to determine equivalent concentrations of different desizing agents required to produce the same exten-

of reduction in viscosity of a given starch paste. Knowing the relative strengths of different desizing agents and their respective prices, one can choose the most economical product. Care must be taken to ensure that each product is tested under optimum conditions of pH, temperature etc. recommended for it.

#### Wetting Agents

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These are used in several processes such as scouring, mercerising, dyeing etc. where it is necessary to ensure that the reagent will be absorbed by the textile quickly and uniformly. A standard method for a comparative evaluation of wetting agents is the Draves Test which measures the sinking time of a standard hank of grey yarn under exactly prescribed conditions. (See for example, Indian Standard No. 1185). Plots of sinking time vs concentr tion of wetting agents are made for the different samples. From these, equivalent concentrations of different samples to give the same sinking time, i.e., the relative wetting efficiencies of the different samples can be easily determined. Taken together with the price factor, the most economical product can thus be selected.

In carrying out these evaluations, it is also necessary to obtain additional information about the efficiency of the wetting agent under the particular conditions of its use. Thus, for example, a wetting agent used in scouring must be efficient at temperatures near boil. Likewise, wetting agents used for mercerising must show efficient wetting in highly alkaline solutions. It is also necessary to confirm compatibility of the wetting agent with other ingredients of the particular reaction system. An anionic wetting agent will be incompatible

with cationic reagents and vice versa. Where problems of compatibility arise, the choice of wetting agents may have to be restricted to the nonionic type.

#### Dispersing Agents

Modern dyeing methods, especially for vat dyes, employ a dispersion of the dye in its pigment form instead of its solution in a reduced form. This change is necessary because of the use of continuous dyeing methods in which an important requirement is to minimise tailing effects due to substantivity and affinity of the reduced vat dye for the fibre. For level and uniform dyeing, free from specks, it is necessary to ensure that the vat pigment is in a truly colloidal dispersion which remains stable for the required length of time under the conditions of dyeing. It is necessary to use an efficient dispersing egent for this purpose.

A simple test for the efficiency of a dispersing agent is to place a drop of the dye dispersion in the centre of a filter paper disc and observe the diffusion of the dye relative to that of water. If the auxiliary is efficient and the dispersion satisfactory, the dye will be transported along with water and the filter paper will be dyed more or less uniformly. On the other hand, if the dispersion is not satisfactory, the dye will not move much beyond the centre spot and will not be transported to a significant extent with the water.

#### Levelling Agents

Certain dyes, especially in the vat series, have a very high initial rate of dyeing (called strike) and poor migration

characteristics. It is difficult to obtain level dyeing with such dyes especially when the textile is dyed in a package form. Levelling agents are used as auxiliaries in such cases to assist in producing good dyeings. Levelling agents act in one of two ways. The majority of them retard the rate of dyeing, whereas a few recent products are adsorbed in preference to the dye on the more active sites on the fibre surface. In either case, the phenomenon of strike is considerably reduced in intensity, and migration of dye from one site to another is promoted.

A simple method for comparative evaluation of levelling agents is to perform a series of dyeings on skeins of standard weight using 2, 4, 33 and 40 minutes as dyeing time. The depth of shade is compared between the 2 and 4 minute dyeings and between the 38 and 40 minute dyeings. If the levelling agent is efficient, the depth of dyeings should be comparable within each set. If the test is carried out at different concentrations of the levelling agents to be compared, one can determine the minimum concentration necessary to give the desired results for each sample.

#### OTHER MAJOR CHEMICALS

In addition to the materials discussed above, a textile processing house uses a large number of common chemicals. Of these, the more important ones are sodium hydrosulfite, caustic soda, common walt, glauber salt, soda ash, sodium nitrite, hydrogen peroxide, acids and bleaching powder. Standard analytical procedures can be employed for evaluating the purity of these chemicals.

#### WATER

Often it is not realised that water is as important a raw material for textile processing as those discussed above. Since water is used in large quantities at every step in textile processing, its quality has a marked effect on the quality of processing. In analysis of water, the usual tests viz., hardness, alkalinity, dissolved solids and total solids have to be carried out. Presence of iron also should be tested. Hardness in water is particularly objectionable since it interferes with the efficiency of various reagents. Thus, in scouring, a part of the alkali will be used up in reacting with the calcium and magnesium bicarbonates present in hard water. In dyeing, particularly package dyeing, hard water will form an insoluble scum in the dye-liquor which gets filtered off on the surface of the packages, thus creating problems of patchy dyeing If the content of dissolved solids in water is high, these may form localised deposits on the fabric when it is piled or rolled up in the wet state because of the greater exposure to air of the selvedge This again can cause defective dyeing if salts of magnesium and calcium are present in water in large quantities. Salts of iron have a catalytic action in bleaching and may, therefore, cause overbleaching and tendering of the fabric.

Many mills employ a water softening process so that water of the required quality can be supplied to the processing departments. It is necessary to check the quality of softened water regularly to ensure that the softening plant is working efficiently.

## (2) QUALITY CONTROL OF GOODS-IN-PROCESS

This is the second important aspect of the quality control programme discussed in this paper. Textile processing consists of a series of sequential steps, the efficiency achieved in one step often affecting the efficiency of the following steps. Thus, it is imperative to carry out proper quality checks at each strategic operation to minimise the danger of defects in the final product. In the following paragraphs are described selected tests for quality control which are considered to be critical for maintaining uniformly high quality.

#### Desizing

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Residual size on the desized fabric must be evaluated periodically, especially when any changes are made in the size mixing, fabric construction, desizing agent or conditions of desizing. Residual size is determined by complete desizing of the sample fabric in the laboratory, and by determining the difference in weight of the sample fabric either in the bone-dry or in conditioned state before and after complete removel of size, as tested by staining with a dilute iodine solution.

From experience, it is found that residual size should be kept below 1% on the weight of the fabric. Inefficient desizing leads to uneven scouring, giving wide variations in fabric absorbency which increases the danger of patchy and uneven dyeing.

#### Scouring

The main purpose of acouring is to remove noncellulosic impurities from the loom-state fabric and to make it more absorbent

so that the subsequent processes of bleaching, dyeing etc. can be carried out efficiently. Absorbency of the scoured fabric should, therefore, be regularly tested for each lot of fabric processed. Absorbency can be tested by a simple method in which a drop of water from a burrette is dropped from a given height on the fabric which is mounted taut in an embroidery ring and kept in a horizontal position. The drop of water is suitably illuminated and the time required for its disappearance is measured with a stop watch. A well-scoured fabric should show a mean value of 3 seconds for absorbency with a maximum of 5 seconds for any individual value.

The importance of maintaining strict control over absorbency need not be emphasised. In processes such as mercerizing and dyeing, the fabric is exposed to the chemical or dye for a fraction of a minute in which it must be uniformly absorbed and reacted with the fibre. This is possible only if the fabric has a uniform and high degree of absorbency.

Periodically it is also desirable to carry out tests for residual wax content and nitrogen content in the scoured fabric.

#### Bleaching

In the process of bleaching, the main objective is to oxidise the natural colouring matter present in cotton fibres and thus to increase the whiteness of the scoured fabric. Bleaching may be carried out either as a batch or a continuous process, employing sodium hypochlorite or hydrogen peroxide or both. Some mills still continue to use bleaching powder instead of sodium hypochlorite though the use of the latter is increasingly favoured.

The bleached fabric must be tested for whiteness and fluidity. Several reflectance meters are available which are suitable for measuring whiteness of fabrics against standard magnesium oxide which is taken as 100. Standards of acceptable whiteness vary from country to country and may range from 80 to 87 per cent.

Fluidity of the cotton fabric sample in cuprammonium hydroxide or in cupriethylene diamine is an index of the extent of chemical degradation of the cellulose molecules that has occurred in the process of bleaching. Details of the test methods are available in published literature (for example, Indian Standard No. 244). A fluidity not exceeding 8 rhes is considered satisfactory though a value upto 10 rhes may be considered permissible. Fluidity exceeding 10 rhes is a definite indication of excessive fabric degradation and demands careful study of the bleaching conditions employed.

Since the bleaching action depends on the concentration, pH and temperature of the bleaching solution employed, it is necessary for proper process control to test these characteristics at regular time intervals during the process. pH is very critical especially in hypochlorite bleaching and should be maintained at 10 to 10.5. The rate of oxidatic of cellulose increases rapidly as the pH goes down and is very high in the pH range of 7 to 8. When hydrogen peroxide is used as a bleaching agent, pH is not so critical from the point of view of fabric degradation. However, control of pH is essential here too since the rate of decomposition of the peroxide and hence the efficiency of bleading is dependent on pH. Optimum results are obtained at a bath pH of 10.5 to 11.00.

When either bleaching powder or sodium hypochlorite is used as the bleaching agent, it is necessary that the bleached fabric after thorough washing is soured with a dilute solution of sulfuric or hydrochloric acid. This step is necessary to remove excess alkali, to decompose remaining hypochlorite and, when oleaching powder is employed, to remove residual calcium from the fabric. Hydrochloric acid is preferred to sulfuric acid for souring, because being volatile, its concentration on the fabric cannot be built up in subsequent drying operations. Then bleaching powder is used, sulfuric acid is definitely to be avoided for souring since it produces, by reaction with the residual bleach liquor, colour sulphate which is insoluble in water and may, therefore, leave deposits on the fabric.

The pH of the fabric after souring and thorough washing must be tested without fail to avoid any hazards of traces of residual acid degrading the fabric during drying. Spotting the fabric with universal pH indicator is a quick and convenient test for this purpose.

#### Mercerizing

Concentration of sodium hydroxide in the mercerizing liquor is tested by standard analytical methods. It is particularly important to check for curbonate alkalinity because this is liable to be present in the caustic lye and does not contribute at all to the mercerizing effect. The quality of mercerizing is measured by determining the barium activity number and luster of the mercerized fabric. Mercerizing produces swelling of the cotton fibres and hence increases its absorption capacity for various aqueous reagents. The

barium activity number test is a measure of the increase in absorption capacity due to mercerizing and is satisfactory for routine quality control purposes. The primary objective of mercerizing is to increase the luster of the cotton fabric. Lustre is enhanced when the textile is swollen with the mercerizing solution and subsequently stretched. Since the tension applied on the cloth during mercerizing is governed largely by the design and setting of the machine, it may not be necessary to determine lustre as a regular quality check. However, occasional measurements of lustre are useful especially when the conditions of mercerizing are changed.

In addition to the above tests, it is also necessary to test the amount of residual alkali on the fabric after it is neutralized on the mercerising range. Excessive residual alkali can lead to yellowing and oxidation of the fabric. It is also necessary to determine the concentration of wash liquors in the different storage compartments of the counter current washing system normally used in mercerizing machines. Maintaining these concentrations at optimum levels is important for achieving maximum efficiency in caustic recovery.

#### Finishing

For starch-based finishes, the most important tests which should be done regularly ard viscosity of each lot of ready finishing paste, as well as paste viscosity and temperature in the sow-box periodically during the finishing of a batch of fabric. Inasmuch as the feel, handle, etc. of the finished fabric directly depend on the amount of finish on the cloth which in turn depends on paste

viscosity, it is important to maintain this property within required limits. Occasionally, it will also be necessary to test the amount of squeeze obtained across the width of the finishing mangle. This will ensure that the pick-up of finishing paste is uniform over the width of the fabric and is maintained at the desired level. Fabric feel, handle etc. are generally evaluated subjectively. Occasionally, it may be worthwhile to supplement this subjective evaluation by measurement of stiffness and drape through instruments such as the stiffness meter and Handle-O-Meter. It is also necessary to determine the whiteness of the finished fabric using visible and near ultraviolet light sources. The latter is necessary to determine the efficacy of any optical brightening agents that may have been used in finishing.

When easy-care finishes are employed, stability of the resin finishing bath must be checked constantly. The resins normally used for such finishing are either monomers or low molecular weight precondensates, both of which have a tendency to polymerise in acidic pH conditions. While there is practically no danger of this when metal salt catalysts are used, this is not the case with the other types of catalysts often employed for resin finishing. Stability of the finishing bath may also be adversely affected because of the incompatibility of the different ingredients employed. If premature polymerisation has occurred due to acidic pH conditions or if there is precipitation be ause of incompatibility of ingredients a turbidity will be produced which can be clearly observed by examining a sample from the finishing bath in a test tube.

The fabric to be finished must also be carefully tested for its pH and absorbency. The fabric should be free from any alkalinity and should preferably have a slightly acidic pH. Any alkalinity in the fabric would neutralise a part of the catalysts employed and may thereby reduce the level of resin fixation. Fabric absorbency, as measured by the method described earlier, should be about 3 seconds to ensure uniform and thorough absorption of the finishing chemicals by the fabric.

While the above tests are required to be carried out for every batch of cloth finished, additional tests are necessary on an occasional basis. The extent and uniformity of squeeze obtained in the finishing mangle must be determined for reasons already given earlier. Uniformity of drying and curing of the fabric must also be ensured to avoid uneven results. In stenters and curing ovens, there is a definit possibility of nonuniform temperatures at the sides of the machines. This would lead to variations in levels of resin fixation and therefore in fabric crease recovery. A simple staining test to essess evenness of resin fixation employs dyeing of the dried or cured fabric with a combination of a naphthol and a base. The azoic stain can be red (Naphthol A3 - Fast Red 3GL), blue (Naphthol BN -Fast Blue 2B) or yellow (Naphthol AT - Fast yellow GC). The appropriate stain is selected to provide sufficient contrast with the colour of the dyed fabric<sup>3</sup>.

Uneven resin fixation will lead to varying intensities of the stain at different portions of the fabric. It is also necessary to check periodically the efficiency of resin fixation obtained under

the conditions of finishing employed. A level of 80% is considered satisfactory. If the fixation is less than this, a detailed investigation is justified.

#### Dyeing

Care must be taken to ensure that the dye-bath is accurately prepared according to the relipe. Temperature of the dye-liquor during the dyeing process must also be measured from time to time. At the end of the dyeing, it would also be desirable to test a sample of the exhaust dye-liquor, at least visually, against a standard dye solution prepared according to the level of exhaustion expected. For example, if a 90% exhaustion is expected for a particular dye, the standard solution for comparison would be obtained by a ten-fold dilution of the original dye-liquor. For vut dyeings, it is also essential to check from time to time that required concentrations of alkali and hydrosulfite are available in the dye-bath. Test papers are available for this purpose. If the dye is used in the form of a pigment dispersion, the latter must be tested for its proper colloidal state by using the filter paper test already described earlier.

The material to be dyed should also be tested. In the case of fabric dyeing, the cloth must be tested for absorbency and for freedom from inorganic impurities, especially magnesium and calcium salts. The batching of cloth must be carefully done to avoid uneven dyeing at the selvedges when jiggers are employed. When yarn is dyed in package form, it is essential to measure the hardness of packages and to see that all the packages used in a lot are

within a narrow range of hardness. A simple determination of package density is a sufficiently reliable measure of hardness.

The padding mangle must be checked periodically for the extent and uniformity of squeeze obtained. In dyeing methods which employ padding of the fabric with the dye or a chemical, it is necessary to maintain a constant liquor level in the bath by automatic controls. Similarly in the pad-steam process of dyeing, automatic controls are necessary to maintain the concentration of the chemical bath accurately at the desired level.

At the end of a dyeing process, it is customary in most mills to cut out a small sample, dry it and compare it with the standard to see that the desired shade of dyeing is obtained. If only slight modifications are required in the shade, this can be carried out easily at this stage without the necessity of a complete redyeing.

#### Printing

Just as in the case of dyeing, it is necessary to ensure that the composition of the printing paste accurately tallies with the given recipe. Viscosity behaviour of the thickene:, particularly under conditions of high shear, needs to be studied whenever a new sample has to be evaluated. Sodium sulfoxylate formaldehyde (rongolite) used in large quantities of printing of vate must be checked for its activity. When the all-in process of printing is used for vat dyes, it is also necessary to ensure that the dried prints are stable during storage and that there is no premature decomposition of the sodium sulfoxylate formaldehyde. This can

happen if the dried prints are exposed to high humidities and temperatures. Likewise, it is necessary to see that naphtholated goods, prior to printing, are not exposed to direct sunlight during storage.

Uniformity of printing roller pressures at the two sides must be checked periodically. Proper setting of rollers to get sharp and accurate prints is a common procedure which every printer observes as a matter of routine. Conditions in the ager must also be checked periodically to ensure uniformity of temperature and freedom from air.

The fabric to be printed must be tested for absorbency and freedom from protruding threads. Proper shearing and cropping will minimise chances of the latter and considerable reduce incidence of defective prints.

#### Process Control of Blend Fabrics

Many of the tests and procedures discussed so far are applicable to process control of blend fabrics also. For these fabrics, however, there are certain special requirements which are discussed here. Since this article is concerned with finishing of cotton, the discussion in this section is with particular reference to polyester-cotton and viscose-cotton blend fabrics which are industrially by far the most important today. It is recognized that cotton may be blended also with other fibres such as cellulose acetate or acrylics. In a general article such as this, it is not possible to include specific test methods for all possible blend fabrics.

The following tests are considered necessary for maintaining proper control on processing of blend fabrics:

(a) Blend Composition :

The tone and depth of dyeing obtained will depend on the relative quantities of the component fibres of the blend, since their dye absorption characteristics are different. Periodic checks would be adequate to ensure that uniform blending is being achieved in the required proportions of the component fibres. In case of defective dyeing<sup>6</sup> especially in the nature of warp-wise or weft-wise bars, a more detailed investigation of blend composition may be required. Extra precaution is necessary if a mill is manufacturing several fibre blends and it may be useful to follow clear systems for visual identification of different blends.

(b) Inspection of the Loom State Fabrics :

While this is important for all fabric finishing, it is especially so in finishing of fabrics containing synthetic fibres such as polyester or polyamide. This is because any oil, grease or other stains on the loom state fabric are liable to be fixed permanently in these blend fabrics during processing. It is, therefore, necessary to inspect the fabric thoroughly in the loom state and remove such stains by proper spotting or solvent treatments before undertaking finishing of the fabric.

(c) The processes of cropping, shearing and singeing are critical in processing of blend fabrics containing synthetic fibres in order to achieve a high quality and appearance of the finished fabric. If these processes

have not satisfactorily removed the protruding fibres and fuzz from the fabric surface, the finished fabric is liable to pilling as well as to localised defects in fabric appearance due to formation of beads formed due to melting of the synthetic fibre fuzz at the high temperatures used in subsequent treatments such as heat setting. No instrumental test is available to evaluate the efficiency of cropping, shearing and singeing treatments. Only a visual examination is possible to determine whether satisfactory results are being achieved.

(d) In the processes of scouring, bleaching and mercerising, which are all carried out generally at an alkaline pH, it may be necessary to confirm that the man-made fibre is not being unduly degraded. For example, polyester is liable to alkaline hydrolysis whereas viscose can dissolve under certain conditions of alkaline treatment. The most effective method of minimising this risk is to follow the optimum processing parameters with respect to pH and temperature and to enforce these strictly. Excessive degradation of the fibre will be reflected in a loss in tensile strength and in weight of the fabric. Of these, the former is easier to measure as and when necessary.

#### (e) Dyeing :

In the finishing of blend fabrics, containing man-made fibre such as polyester or polyamide high temperature treatments are used in dyeing and in heat-setting. Dyeing will generally consist of two steps in which each of the fibre components is dyed with an appropriate dye type in successive stages. It is necessary to ensure that the dyes used for the cellulose component are stable at the high temperatures required for

dyeing and heat-setting the synthetic fibre component. Also methods such as the thermosol process for dyeing of these blend fabrics involve heat conditions under which the disperse dye used for dyeing the man-made fibre can sublime and stain the fabric. It is, therefore, necessary to test the dyes used for the man-made fibre component for sublimation fastness at the temperatures used in thermosol dyeing.

Garments made from polyester-cotton and similar blends may be dry-cleaned by the wearer instead of the usual soap washing. Fastness of the dyes (to dry-cleaning) is, therefore, another useful quality control test.

Most dye-stuff manufacturers provide data regarding heat stability and fastness to sublimation and to dry-cleaning their shades in cards. Generally, therefore, it would be sufficient for a mill to merely refer to these shade cards for selecting suitable dyes.

Cross-dyeing, i.e. dyeing of the two components of the blend in contrasting shades is generally not recommended for blends containing a cellulosic fibre such as cotton or viscose and a man-made fibre such as polyester or polyamide. This is because the abrasion resistance of these two classes of fibres is widely different with the result that the cellulosic component will get abraded much faster during wear leading to a peculiarly patchy appearance called frosting in the dyed fabric (or garments). If fashion requirements dictate that cross dyeing must be used, processing conditions in such fabric treatments as resin finishing should be so chosen as to minimise losses in abrasion resistance of the cellulosic component. The susceptibility of a dyed blend fabric to frosting can be evaluated reliably with the accelerator.

(f) Heat-setting is a crucial stage in the finishing of blend fabrics containing a synthetic fibre such as polyester or polyamide. In this process, the fabric is subjected for a few seconds to a temperature just below the softening point of the man-made fibre. The purpose of heatsetting is to improve the dimensional stability, dry crease recovery and pilling resistance of the fabric. Tests of these characteristics, therefore, require to be carried out on the heat-set fabric to ensure that the process is being carried out satisfactorily.

#### (3) INSPECTION OF FINISHED FABRICS

Some tests of finished products, viz., lustre and whiteness, have already been mentioned earlier. In addition, the following tests are also necessary as part of regular inspection.

All dyeings and prints must be tested for their fastness properties, particularly to washing and light. When pigments are used for dyeing or printing, dry and wet rubbing fastness must also be determined. For specific end-uses, fastness to other agencies such as chlorine, perspiration, dry-cleaning, etc. may need to be carried out.

Residual shrinkage in the fabric needs to be determined particularly when marketed as pre-shrunk.

Fabrics which have been finished for easy-care properties must be tested for dry and wet crease recovery, tensile and tear strength.

wash-and-wear rating, wash fastness of the finish and if necessary for chlorine retention. In the case of durable press finishing, additional tests are also required. Storage stability of the sensitised fabric must be determined. Abrasion resistance also has to be tested and controlled, especially if the fabric contains little or no polyester fibre. Garments made from durable press finished fabrics must be tested for crease retention, smooth drying and freedom from seam puckering.

In addition to these specific properties, the fabric in its final finished form must be inspected for defects such as stains, patchy or uneven dyeing and defective prints.

Even if a mill adopts a comprehensive programme of quality control such as described in this article, the chances of having some defects, especially in dyeing, cannot be totally ruled out. This is because of certain inherent limitations in sampling of the goods-inprocess. In a batch system of processing, the minimum lot size will be about 25,000 metres. For continuous processing, it would be much larger. Considering the time required for carrying out many of the tests given in Section 2, it would not be practicable to test more than 3 to 5 samples per lot. The quantity of samples required for most of these tests is much smaller than a metre. This means that the sample size is at the most 1 in 5,000 or 0.02%. There is a further

restriction on the representativeness of sampling. The sample can be withdrawn only at the beginning or end of a piece, i.e., at the place where two pieces are stitched together. Thus, while systematic process control will generally ensure that the desired results are achieved, there can be chances of an occasional defect going undetected.

The rest of this paper, therefore, deals with methods of examining such defects and diagnosing the source of the defect.

The most common causes of stains in dyed fabrics are due to (1) defects in cotton and yarn, or in weaving, (2) faulty bleaching and finishing, and (3) faulty dyeing.

#### Defects in Cotton, Yarn and in Weaving

Immature fibres cause neps in spinning which because of their lower dye uptake will appear as lightly dyed specks on the fabric surface. balling up of mature fibres in carding can also form neps of a different type. Fluff sometimes settles down on the textile material in spinning and weaving. In both the cases, the bundle of loosely adhering fibres will dye deeper and appear as dark specks on the fabric surface. Another source of dyeing defect is due to contamination of the fibres by lubricants and by marking inks and crayons. If these are unsaponifiable, they would not be removed in scouring and will produce lightly coloured patches or resist marks in the dyed fabric. Residual size also has a similar effect in dyeing.

Thick and thin places in the yarn, uneven denting due to poor quality of reeds, etc. give a streaky or reedy appearance to the dyed cloth.

## Faulty Bleaching and Finishing

Incomplete desizing, nonuniform scouring because of channeling, overloading, etc. in kiers, degradation of cellulose in bleaching and localised drying are common defects in the preparation of the fabric for dyeing. In mercerisation, creases in fabric, overlapping of ends on the chainless, padless type of machines, excessive foaming in the padding trough of the mercerising machines and abrasion marks from expanders have been observed to cause faulty dyeings. Often hard water is used in preparing accuring and mercerising liquors. This leads to precipitation of hydroxides and carbonates of magnesium and calcium which get deposited on the cloth and produce uneven dyeings.

## Faulty Dyeing

Leuco compounds of some vat dyes are sensitive to sunlight. If cloth dyed with the reduced vat is exposed to sunlight, the exposed portion will dye lighter. Some dyes are very sensitive to over-oxidation and will not dive the required shade unless extreme care is taken in dyeing and development. In dyeing of azoics, particular care is required for maintaining correct temperature and pH during coupling. Instability of the diazotised base may result in formation of deep stains or bronzy appearance. Exposure of naphtholated cloth to acid fumes invariably leads to stains.

To pinpoint the particular cause which led to the stain by a postmortem of the defective dyeing is by no means an easy job. However, a systematic examination as described in Table I will give useful information, which in many cases will help to diagnose the cause and location of the defect.

#### TABLE I

TESTS FOR ANALYSIS OF STAINED FABRICS

Test		What is Observed	
Exa	mination of Stain:		
1.	Visual	type; shape; repeat; colour; frequency; whether on both sides of cloth.	
2.	Under Microscope	fly; neps; mechanical damage; mildew attack; deposits.	
3.	Under Ultraviolet Light	distinctive fluorescence due to mineral oils, crayon marks, optical brightening agents.	
Ana	liytical Tests:		
1.	Absorbency	Poor or non-uniform absorbency.	
2.	Wax Content	High or unevenly distributed.	
3.	Ash content; Qualitative Tests on Ash	Salt deposits; presence of calcium, magnesium.	
4.	Fluidity; Tests for Oxycellulose and Hydrocellulose	Extent and type of cellulose degradation.	
5.	Stripping and Redyeing	Stain reappears or not.	
Plant Observations		Location of the exact origin of stains.	

This method of diagnosis is illustrated with the help of three cases selected from the large number of such investigations carried out at Ahmedabad Textile Industry's Research Association. The nature of the defect, observations from various tests and inferences drawn from them are tabulated (Tables II, III & IV) for each case. Conclusions about the cause of the defect are given at the end of the table. These were confirmed through observations on the shop floor and through the effectiveness of suitable remedial action in eliminating these defects.

CASE 1 : WHITE SHORT LI	NES ON CLOTH DYED WITH A	ZOIC COMBINATION
		Dod with w
Test	Observation	Inference
Examination of Stains: Visual a	) White streaks in warp and weft, about 1-2 inch long	

	a) White streaks in warp and weft, about 1-2 inch long	defect not likely to originate in weaving	
	b) Defect confined to single threads	or chemical processing	
Under microscope	Portion of yarn undyed	-d o-	
Under UV light	Green fluorescence	Presence of mineral oil deposits	
Analytical Tests:			
Absorbency	Poor at the white stain	Presence of hydropho- bic material	
Bleached cloth dyed in laboratory -			
a) before solvent extraction	White lines in dyed cloth	Dyeing method not faulty. Presence of	
b) after solvent extraction	Even dyeing; no defect.	oil confirmed	
Plant Observations	Improper handling of yarn after spinning. Excessive lubrication.		

TABLE II

#### TABLE III

## CASE 2 : WHITE STAINS IN DYED FABRICS

Lawn : 80 s/ 100 s; 95 x 88

Mercerised Fabric, dyed on jiggers with vats

Test	Observations	Infer ence
Examination of Stains:		
Visual	White stains only on one face of cloth	Defect may originate in an open-width process
Under UV light	No fluorescence	Mineral oil absent.
Analytical Tests:	<u></u>	والاروالي هار، وارد شوار مير خدم الدين التي والله عليه الذي و مريس مدينة من بين والله على والوو
Absorbency	Good	
Wax content	Less than 0.3 per cent	
Ash content	Low	Bleaching not faulty
Cuprammonium fluidity	Less than 8 rhes	
Stripping and redyeing	Stains reappear	Dyeing not faulty
Plant Observations	The fault was located in mercerising. Excessive foaming in padding trouch of merce- rising machine. Foam was getting trapped between two layers of cloth. Bleached samples were dyed before mercerisation; no fault was noticed, but mercerised samples dyed unevenly.	Uneven mercerisation due to foaming in pad-bath was the cause.

## TARLE IV

# CASE 3 : PATCHY DYEING IN POPLIN DYED WITH VATS

Poplin : 30s/38s, 112 x 56

Manufactory of the owner owner owner owner owner owner owner

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The second s

Dyed by pigment pad-jig develop method

Test	Observation		
Examination of Stains		Inference	
Visual	: Overall patchy appearanc with deeply dyed specks all over the cloth	<del>0</del> -	
Under microscope	Fluff or neps absent	_	
Under UV light	No fluorescence	Mineral oil absent.	
Analytical Tests:			
Absorbency Wax content	Good Less than C.3 per cent		
ish content Suprammonium fluidity	0.2 per cent Less than 8 rhes	Bleaching not faulty.	
tripping and redyeing	Uniformly redyed	Dyeing method faulty.	
lant Observations	It was found that the method of preparing pigment dispersion was not satisfactory. Pasting of dye was not carried out properly. Dispersing agent used was not efficient.		

#### CONCLUSION

A well-planned programme of testing, quality control and inspection is necessary to ensure control over raw material costs, to maintain desired product quality and to minimize the proportion of sub-standard production in finishing of cotton textiles. Suitable test procedures have been described for important raw materials and for goods-in-process, and their significance with respect to processing quality and efficiency pointed out. These tests can be easily carried out in a reasonably well-equipped textile mill laboratory. A detailed procedure for inspection of defective dyeings has been suggested for identification of the cause and source of the defect. Selected case histories have been given to illustrate how this procedure has been used to diagnose origin of defects and to decide on necessary corrective action.

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