CHAPTER 3

EXPERIMENTAL (GENERAL)
3.1 Materials

3.1.1 Fibres used

Delustured polyester (polyethylene terephthalate, PET) fibres of 2 denier (g/m) linear density and 44 mm staple length were obtained from a local supplier. W3 grade jute fibres from middle portion of the reed, combed and stapled to nearly 40 mm giving an average linear density of 18 to 20 denier for the combed jute were obtained from a local jute mill. These staple polyester and stapled jute were used for manufacture of rotor-spun jute/polyester yarns.

3.1.2 Yarns used

Conventional-spun 100% jute yarn of 175 - 180 tex (g/Km) and 100% polyester yarn of 11.3 tex were obtained locally.

3.1.2.1 Yarns from jute/polyester (J/P) blends prepared by Rotor-spinning technique

Polyester (PET) staple fibres were mixed by hand with stapled jute (J) in different weight proportions giving 20 to 50% polyester in the blends. The blends were then double carded in a Shirley cotton miniature card. The carded lap was passed through the four passages of a shirley miniature roller-draft drawing frame to produce a drawn sliver of 4.7 Ktex. The drawn sliver was then fed into a laboratory model rotor spinning machine fitted with Platt’s 887 rotor box to yield rotor-spun J/P yarns of 140 - 150 tex having 6.25 turns (mechanical twist) per cm. The rotor
speed, opening roller speed and the delivery speed maintained were 25,000 rpm and 7,000 rpm and 22 - 45 m/min respectively.

3.1.2.2 Yarns from Jute/Polyester (J/P) blends prepared by conventional (apron draft) spinning technique

Conventional-spun J/P blended yarns were prepared using commonly employed jute processing machinery and processing sequences. Slivers from jute finisher card and carded polyester top were blended in a typical jute draw frame at the second drawing stage. After the usual 2nd drawing operation, two more successive drawings in the second drawing unit were performed giving equal drafts and equal doublings in each of them in order to enhance homogeneity of the blended yarns. After final drawing in the finishing (third) drawing unit, the sliver was spun into J/P blended yarns using a conventional apron draft jute spinning machine.

3.1.3. Fabrics used

Jute (J) fabric

Ex-powerloom, plain weave, jute fabrics with yarns of 195 tex as warp and 214 tex as weft having 63 ends and 59 picks per dm and weighing 264 g/m² on the average were obtained from a local jute mill.

Jute/Cotton (J/C) union fabric

Ex-handloom, plain weave, 75 : 25 (w/w) jute/cotton union fabric having cotton yarns (29.5 tex) as warp and jute yarns
as weft was used. The fabric had the following characteristics viz. 173 ends/dm, 102 picks/dm and it weighed 227 g/m² on the average.

Jute/viscose (J/V) blended fabric

60 : 40 jute-viscose blended fabric having the following specifications was used: weave-plain; Area-density (fabric weight) - 307 g/m²; Picks/dm - 72; Ends/dm - 60; Warp count - 140 tex; and weft count - 160 tex.

3.1.4 Chemicals used

L. R. grade sodium hydroxide, hydrogen peroxide (50%), sodium silicate, Nonidet-P-40 (a nonionic detergent), acrylamide, formalin (40% formaldehyde), potassium persulphate (K₂S₂O₈), acetic acid, butanol, xylene, sulphuric acid, sodium chlorite, trisodium phosphate and magnesium chloride were appropriately used in the experiments as and when required.

3.1.5 Resins and other textile finishing chemicals

3.1.5.1. DMDHEU resin

Textile finishing grade 1,3 dimethylol - 4,5 dihydroxy ethylene Urea (DMDHEU) resin precondensate, obtained from Ahura Chemicals (Ahuramine - YX, having 50% solid content) was used. Its chemical structure is shown in fig 3.1(a).

3.1.5.2 UF resin

Aqueous urea-formaldehyde (UF) resin, i.e. mixed methylol urea (58% solid content), water soluble grade, was
Dimethylol-dihydroxy ethylene urea (DMDHEU) or N-N' dimethylol-4,5 dihydroxy ethylene urea (a)

Urea-formaldehyde (UF) or N-N'-dimethylol urea (b)

Partially butylated urea-formaldehyde (BUF) resin or Partially butylated di-methylol urea (c)

Acrylamide-formaldehyde (AMF) resin or N-methylol acrylamide (d)

Fig. 3.1: Chemical structure of resins used.
obtained from a local supplier. Chemical structure of dimethylol urea is shown in fig 3.1(b).

3.1.5.3 BOF resin

Water insoluble partially butylated urea-formaldehyde (BOF) resin (56% solid content) obtained from a local manufacturer was used in an emulsion form. The resin was diluted in 1 : 1 xylene/butanol mixture and then emulsified in water containing the nonionic emulsifier (0.5% Nonidet P40), giving a solid content of 10% for the emulsion. Chemical structure of partially butylated dimethylol urea is shown in fig 3.1(c).

3.1.5.4 Preparation of Acrylamide-formaldehyde (AMF) resin

33g (1.1 mole) of formaldehyde (83 cc of formalin solution) was initially neutralised using dilute NH₄OH solution and 71g (1 mole) of AR grade acrylamide dissolved in 100 ml water was mixed with it and the pH of the mixture was maintained at 9.0 with dropwise addition of a dilute NaOH solution; then the mixture was allowed to react at 50°C in a water bath for 2h. The mass was then cooled to obtain water soluble acrylamide - formaldehyde (AMF) precondensate resin with 24% - 30% solid content. Its chemical structure is shown, fig.3.1(d).

For analysis and characterisation purposes, the water soluble AMF resin precondensate was partitioned by constant shaking with warm ethyl-acetate. The ethyl acetate solution of the AMF resin was then cooled to ice temperature; the precipitated
resin was filtered, washed with acetone and dried in vacuum at 40°C.

3.1.5.5 Additives in resin finish

A textile grade, commercially available emulsion of acrylate polymers of low degree of polymerisation made available by Ahura Chemicals (Ahuracryl-PVN having 18% solid content) was also used selectively as additive in AMF resin finish. Also acrylamide monomer was used selectively as additive.

3.2 Methods

3.2.1 Dewaxing

Dewaxing of selected jute fibre/fabric samples was done by soxhlet extraction for 6h using ethyl alcohol-benzene mixture (1:2, vol/vol) as the dewaxing solvent. The samples were then washed with alcohol and finally with distilled water and then dried in air.

3.2.2 Scouring

Selected jute and blended/unionised jute fabrics were scoured as and when required using 4 g/lit of sodium carbonate solution (2% on weight of fabric) and 1 g/lit of a nonionic detergent and heating at 80°C for 30 min for all-jute (J) and jute/viscose (J/V) blended fabrics, and heating at boil for 60 min for jute/cotton (J/C) union fabric. The scouring was done in a laboratory jigger using a fabric/liquor ratio of 1 : 5 (w/v) and the average pH of the bath was kept at 8.0 - 9.0 in each case.
After scouring, the fabrics were neutralised and thoroughly washed with hot and cold water in succession. Fabrics subjected to chemical treatment using strong NaOH at any step were not scoured; in that case the fabric was simply rinsed for 15 min with hot water (70 - 75°C) containing 1 g/lit of a nonionic detergent and then washed and dried.

3.2.3 Chemical texturing of jute or blended jute yarns/fabrics

Chemical texturing of jute, is viewed as an useful step in jute processing. To achieve texturing effect, jute and relevant blended/unionised (jute/viscose and jute/cotton) fabrics of suitable dimension were dipped under slack condition in a stainless steel bath containing aqueous 18% (w/w) sodium hydroxide solution using a fabric/liquor ratio of 1 : 10 (w/v) for a specified time period (30 min or 60 min) at room temperature (25 ± 2°C).

To utilise the advantages of mercerisation effect on cotton warp yarns in the J/C union fabrics, the treatment with 18% NaOH was also done under a constant stretch on a small ebonite pin-frame.

After the NaOH (pre)treatment, each of the fabric pieces was washed under running tap water, neutralised by dipping in 1% acetic acid solution, rinsed with water and finally, dried in air. Jute/Viscose fabric was also additionally washed with saturated brine solution before neutralisation. Considering the differences in the effect of NaOH solution on jute part and on
polyester part, rotor-spun and conventional-spun jute/polyester (J/P) yarns were however treated with different concentrations (1 - 22%) of NaOH solution at different temperatures (5 - 100°C) for a specified time period (1 h).

3.2.4 Bleaching

All-jute and jute blended (J/V) or unionised (J/C) fabrics were subjected to bleaching using the following recipe and procedure, so as to achieve optimum whiteness without much adverse effect on the strength and other textile related properties (all weights of additives in bleaching are on the basis of weight of fabric).

Recipe for bleaching

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen peroxide</td>
<td>- 3% for all-jute fabric and 2% for J/C and J/V fabrics</td>
</tr>
<tr>
<td>Sodium silicate (78°TW)</td>
<td>- 6%</td>
</tr>
<tr>
<td>Tri sodium phosphate</td>
<td>- 2.5%</td>
</tr>
<tr>
<td>Sodium hydroxide</td>
<td>- 0.25%</td>
</tr>
<tr>
<td>Fabric to liquor ratio (w/v)</td>
<td>- 1 : 6</td>
</tr>
</tbody>
</table>

After addition of $H_2O_2$ and other chemicals in the jigger, the temperature of the bath was gradually raised to $80°C - 95°C$ within 20 minutes and then bleaching was continued for 30 min or 60 min as the case may be. The pH of the bleaching bath was maintained at 10.0 - 11.0 for all-jute and J/C fabrics and at 8 - 8.5 for J/V fabrics. After bleaching, the fabric was rinsed with
hot and cold water followed by treatment with dilute solution of acetic acid to remove excess alkali if any, and finally washed with water. The bleached fabric was then squeezed in a padding mangle and finally dried in a drier at 80°C for 10 minutes.

3.2.5 Sequential chemical pre-treatments

The chemical (pre)treatments of fabrics using NaOH and H₂O₂ for achieving texturing and bleaching effect, were also performed sequentially (one after the other) on a given fabric.

The two alternative sequences are:

(A) **Sequential texturing and bleaching** (STB) : treatment with 18% NaOH followed by H₂O₂ treatment.

(B) **Sequential bleaching and texturing** (SBT) : treatment with H₂O₂ followed by 18% NaOH treatment.

3.2.6 Progressive removal of hemicellulose from jute

Progressive removal of hemicellulose from jute to different extents was accomplished by treating jute fibres/fabrics with NaOH solutions of different concentrations (5, 9, 13 and 18%) for ½ h, at room temperature (25 ± 2°C) employing (fibre/fabric) : liquor ratio of 1 : 50, followed by successive washing with 1% acetic acid and water and drying in air.

3.2.7 Progressive removal of lignin from jute

Progressive removal of lignin from jute to different extents was accomplished by treating jute fibre/fabric with 0.7% NaClO₂ employing (fibre/fabric) : liquor ratio of 1 : 50 for
different time periods (5, 10, 25, 50 and 100 min.) at pH 4, at boil followed by washing with 2% solution of NaHSO₄ at 50°C for 30 min and then with water and finally drying in air.

3.2.8 Resin post-treatments

Bleached all-jute fabrics were subjected to AMF resin post-treatment employing different catalyst systems for effecting methylol-methylol and methylol/cellulosic-alcohol condensation and radical polymerisation (leading to different degrees of grafting and leading to some crosslinking) separately and simultaneously using different percentages of the resin under different conditions. Conditions of AMF resin finish were optimised and the resin finished fabrics were characterised.

All-jute or blended/unionised jute fabrics were presoaked with K₂S₂O₃ solution of known concentration, if required, prior to application of AMF-resin without or with (a) specific dose of a selected additive and (b) MgCl₂. K₂S₂O₃ picked up by the fabric would be expected to effect radical polymerisation utilising the vinyl groups of the acrylamide-formaldehyde resin moieties; such resin moieties may remain unbound or differently grafted on the segments of different constituents of jute, along with acidic condensation via methylol groups of AMF resin during the pad-dry-cure process in presence of MgCl₂. Presoaking of K₂S₂O₃ solution and AMF-resin application on jute fabric were done separately using a laboratory two-bowl padding mangle (two dip-two nip) adjusting squeeze pressure to...
enable a wet pick up of 80%. Aqueous solutions of AMF resin of known concentrations containing no MgCl₂ or specific doses of MgCl₂ (acidic curing catalyst) were used separately for padding or subsequent to padding with K₂S₂O₈ as stated above. The padded fabrics after resin application were subjected to drying (80°C, 10 min.) and curing (2 - 20 min., at 100 - 160°C) in a laboratory stenter employing hot air. Unless otherwise mentioned, fabrics treated with all other resins (UF, BUF and DMDHEU) as and when necessary, were dried at 80°C for 10 min and were cured at 150°C for 4 min. The resin finished fabric was then hot soap washed following ISO-II method²⁰ (5 g/lit soap solution at 50°C for 45 min) followed by water-wash at room temperature, finally dried in air and duly conditioned²¹ at 65 ± 2% relative humidity and at 27 ± 2°C for 48 h (as per IS : 6359-1971) before testing. For application of other resins or resin combinations (other than AMF resin) presoaking of the fabric with K₂S₂O₈ solution was not necessary and hence avoided and rest of the processing parameters and conditions were by and large kept unchanged.

3.2.9 Heat setting of textured jute/polyester (J/P) blended yarns

Heat setting without any further treatment for the chemically textured jute/polyester (J/P) blended yarns was accomplished by heating the yarn samples at 150 to 200°C for 4 min.
3.2.10 Dyeing of selected jute fabrics

Bleached and AMF resin treated jute fabrics were subjected to dyeing in a dye bath using a reactive and an acid dye following standard procedures as given below:

**Dyeing with the use of a reactive dye**

Procion brilliant Red M.8B dye (CI reactive red - 11) obtained from Atic Industries, Gujrat, was used for this study. For 1 g of fabric, 0.01 g of the dye was used in 100 ml of aqueous 5% Na₂SO₄ solution. The dyeing was done at 50°C initially for 1 h after which alkali fixation was achieved on addition of aqueous NaOH solution in two equal installments over next 30 minutes period so as to give an overall 1% NaOH concentration in the final solution. The dyed sample was then squeezed, neutralised with dilute acetic acid solution and soap-washed in a separate bath as per ISO-I washing method (5 g/lit soap solution at 40°C for 30 min). The soap-washed sample was further washed with distilled water and dried.

**Dyeing with the use of an acid dye**

Atul crocein scarlet M0 (CI Acid red-73) obtained from Atic Industries, Gujrat was used for this purpose. For 1 g of fabric sample, 0.01 g of the dye was used in 100 ml of 5% Na₂SO₄ solution and requisite volume of 2% HCOOH solution was added to adjust pH at 4 - 4.5. The dyeing was carried out for 1 h at 100°C after which the dyed sample was squeezed, neutralised with dilute
NaOH solution and then soap-washed as per ISO-I washing method\textsuperscript{20},
water washed and dried in air.

3.3 Testing and evaluation of fibres, yarns and fabrics

3.3.1 Determination of linear density and bulk density of
jute/polyester (J/P) yarns

Yarn linear density expressed in 'tex' (g/Km) was
determined by using a mass of yarn in a hank of 50 turns formed
at a constant tension and measuring the mass and noting the hank
length for the yarns set under a predetermined decrimping load for
crimped or textured yarns on top of the usual pretension load of
0.035 g/tex used for the untextured yarns\textsuperscript{20}. With increasing bulk
(volume) of the yarn on texturing, the bulk density follows a
lowering trend. The bulk density of chemically textured J/P yarns
were calculated by measuring the major diameter (a) i.e. breadth
of the thread and minor diameter (b) i.e. thickness of the thread
with the help of a precision travelling microscope under the
thread flattening condition by winding the test yarn under a mild
tension on a uniform glass rod of known diameter; Hamilton's
formula\textsuperscript{23} as given below was used for calculation of bulk density
from these measurements. An average of ten measurements ((a) and
(b)) was used.

\[
\text{Bulk density} = \frac{\text{yarn tex}}{a \times b - 0.2146b^2} \times 10^3 \text{ g/cc.}
\]
3.3.2 Determination of shrinkage of yarns or fabrics on chemical treatment and on washing

Linear shrinkage of a yarn or fabric subsequent to a treatment or washing (ISO II method, 5 g/lit soap-wash at 50°C for 45 min using fabric : liquor ratio 1 : 100) was calculated by measuring the difference in the lengths of a given segment of the test yarn or fabric before and after the given treatment/wash following standard procedures\(^24\). An average of 5 measurements in each case was used for calculation. The shrinkage observed is expressed as percentage of original length in each case. For fabrics, warpway and weftway shrinkages were measured separately.

3.3.3 Measurement of tensile properties of yarn and fabric

Tenacity and breaking extension of yarns (conditioned\(^21\) at 65 ± 2% Rh, at 27 ± 2°C temperature for 48 h as per IS : 6359-1971) were determined\(^25\) as per ASTM-2256-75, using 1445 CRT Zwick Universal Tensile Testing Machine (test yarn length = 30 cm; pretension load = 0.1 N; rate of extension = 5 cm/min). An average of 25 tests was used to express the results.

Fabric tenacity, breaking extension and modulus corresponding to 1% extension (initial modulus) of selected fabric samples (also conditioned as above) were measured\(^26\) as per IS : 1969-1968 in the said tensile testing machine. Average of 10 tests in warp and/or weft direction were taken to express the relevant properties for each sample. Test conditions employed are given
3.3.4 Crimp level and crimp stability of textured all-jute and blended jute yarns

Number of crests and troughs present in a given length of a textured yarn was measured under a constant tension in a projection microscope, and the observed crimp frequency i.e. number of crimps per cm was noted from the projected images of the yarn, counting the number of crests per extended length, as defined by Brand et al.\textsuperscript{27,28}

The initial length ($L_i$) of selected pieces (0.60 – 0.65 m) of textured yarns were measured with a pretension load\textsuperscript{28} of 0.035 g/tex. Each of these was then subjected to tensile loading for 2 min with a predetermined decrimping load and the corresponding extended length ($L_2$) in each case was measured. Crimp % was calculated\textsuperscript{28} as follows

$$\text{Crimp} \% = \frac{L_2 - L_i}{L_2} \times 100$$

3.3.4.1 Testing for crimp stability on mechanical loading

After immediate measurement of the extended length the decrimping load was removed from the yarn and the (creep) recovery was allowed for the next 2 min and thereafter, the final length ($L_a$) of the yarn was measured. Crimp stability (%) of the said sample size = 50 mm x 20 mm (ravelled strip specimen); traverse speed = 100 mm/min; and pretension level = 0.2 N.
textured yarns was then calculated as follows:

\[
\text{Crimp stability (\%)} = \frac{(L_2 - L_4) - (L_a - L_4)}{(L_2 - L_4)} \times 100
\]

The results reported are average of 20 tests for each sample.

3.3.4.2 Testing for crimp stability on washing, heating and resin finishing

Jute or blended jute yarns were subjected to soap-wash as per ISO-II (5 g/l soap solution at 50°C for 45 min) method for five successive wash cycles. After each cycle of soap-wash, crimp stability (crimp recovery, \%) was measured following the method described above. Similarly, subsequent to different resin treatment or heat setting of the relevant yarns the crimp stability was measured.

3.3.5 Measurement of bending length

Fabric stiffness, as expressed by the bending length, was measured as per IS: 6490 - 1971 (cantilever test) in a Sasmira stiffness tester with a specimen size of 200 mm x 25 mm.

In this test, the rectangular strip is extended beyond the edge of the platform of the stiffness tester and the free edge of the fabric is allowed to bend under its own weight until the free edge makes an angle 41.5° (already marked in the instrument) with the horizontal platform. The bending length is obtained using instrument calibration. Higher the bending length, stiffer is the fabric. Bending lengths for warp and weft direction of the fabric specimens were measured separately.
3.3.6 Determination of abrasion resistance

Evaluation of flat abrasion resistance of a selected fabric was done in an electronically controlled abrading instrument (SDL-Crockmeter) using a standard (IS-715) emery paper as the abrading surface fitted to the reciprocating abrader head and worked under a load of 900 g over a linear track length of 10 cm in a given direction (warp or weft) to generate a specific wear following the principle described in AATCC-93-1974. The abrasion resistance of the selected fabric sample was given by a digital display of the number of cycles of flat abrasion leading to the appearance of the specified nature of wear (first appearance of hole on the fabric by wear and tear of its yarns).

3.3.7 Measurement of crease recovery angle

The crease recovery angle of a selected fabric sample was measured using a Sasmita crease recovery tester in accordance with ASTM-D 1295-67 (1972). 10 samples for the selected fabric, each of size 4.0 x 1.5 cm, were cut out. Each cut out fabric piece was folded midway along the length and pressed with 500 gm load (pressure = 167 g/cm²) between two plastic plates for 5 min. The pressed specimen was then transferred to Sasmita crease recovery tester and the angle recovered by the folded specimen after 5 min of unloading was measured. Average crease recovery in each of warp and weft direction (for 10 specimens in each direction) was computed.
3.3.8 Measurement of fibre shedding

Fibre shedding propensity of all-jute fabrics was evaluated by measuring the weight loss in mg on abrading a fixed area of a selected jute fabric sample using another piece of the corresponding fabric as the abrader for a fixed number (100) of cycles of abrasion using an electronically controlled abrading instrument (SDL-Crockmeter) following the method used earlier.39

3.3.9 Examination of yarn structure by optical microscopy

Structural change in jute/polyester rotor-spun and conventional-spun yarns on chemical texturing using 18% NaOH was examined by optical microscopic analysis. The relevant microphotographs were duly taken using an Ernst Leitz microscope with a camera attachment at 100 magnification setting the yarn sample on a glass slide with a pre-fixed mild tension (0.035 g/tex) using appropriate focus.

3.3.10 Scanning Electron Microscopy

Surface morphology of bleached jute (BJ) with or without pad-dry-curing with AMF resin using different catalyst systems was examined according to prescribed procedures using a scanning electron microscope (Cambridge Instrument Ltd., MK-II). Selected fibre samples taken out from the relevant fabrics were subjected to coating with gold-palladium alloy using a sputter coater.34 The observations were made at an operating voltage of 20 KV using a magnification of 20 µm.
3.3.11 FTIR Spectroscopy

Selected fibre (finely crushed) or resin (powder) samples (3 mg) were examined in a double beam FTIR spectrophotometer (Perkin-Elmer, model-1615) using KBr disc technique.35

3.3.12 Differential Scanning Calorimetry (DSC)

DSC analysis of bleached jute and jute treated with AMF resin under a suitable catalyst system, was done using a Shimadzu differential scanning calorimeter (DSC-model-50), under nitrogen flow (flow rate - 50 cm³/min) at a heating rate of 10°C/min, using a sample size of 2 - 3 mg.

3.3.13 Determination of moisture regain

Moisture regain of selected oven dried fabric samples was determined according to ASTM-629-59T, (IS-199-1973).

3.3.14 Analysis of nitrogen content

Nitrogen content of selected fibre/fabric system was estimated following the usual Kjeldahl method.

3.3.15 Measurement of whiteness index, yellowness index and brightness index

Whiteness index of selected jute and relevant fabrics was measured in the Hunter lab scale and yellowness index was measured in the ASTM (E-313, 1973) scale employing a 2020 plus Macbeth reflectance spectrophotometer and making use of the appropriate formula and relevant software.
Brightness index\textsuperscript{28,39} for the relevant jute fabrics was obtained from comparison of the reflectance of the test specimen at a particular wavelength (420 nm) against the same of a standard white surface (a standard glazed white tile giving a reflectance of 100%). The measurements of reflectance values were done in a 2020-plus Macbeth reflectance spectrophotometer.

3.3.16 Estimation of dye uptake and colour strength

\textit{Surface dye uptake}

Dye uptake as estimated in terms of K/S (Kubelka Munk-function\textsuperscript{20,22}) value is based on measuring the surface reflectance of each dyed fabric sample at the respective $\lambda_{\text{max}}$ (in a Macbeth 2020-plus reflectance spectrophotometer) and converting the reflectance value to (Kubelka Munk-function) K/S value as detailed below using the appropriate formula and relevant software attached to the computer aided equipment following standard procedures

$$\frac{(1 - R_{\lambda_{\text{max}}})^2}{2R_{\lambda_{\text{max}}}} \propto C_D$$

where, $K = \text{coefficient of absorption}$,
$S = \text{coefficient of scattering}$
$R_{\lambda} = \text{reflectance of the substrate at } \lambda \text{ wave length}$
$\lambda_{\text{max}} = \text{Maximum absorbance wavelength}$
$C_D = \text{Concentration of dye}$

The K/S value is viewed as an index for surface dye uptake
Colour strength

K/S values of textured and untextured jute/polyester blended yarns (one component, polyester being coloured) were also measured to compute the relative colour strength of yarn surface to assess the preferential position of the coloured component on yarn surface on relevant chemical treatment/texturing; the yarns were parallely wound on a card board to form a continuous sheet of layers of yarn for reflectance measurement.

Relative colour strength(%) = \( \frac{K/S \text{ of the treated yarn surface}}{K/S \text{ of the untreated yarn surface}} \times 100 \)

Bulk dye uptake

Exhaustion of dye from the dye bath taken as a measure of bulk dye uptake was estimated spectrophotometrically following standard procedures using a UV-2000 Hitachi uv-visible absorption spectrophotometer, and dye content in fibre, \([D]_f\) in g/Kg of fibre (a measure of bulk dye uptake) was calculated using those dye exhaustion data.

3.3.17 Analysis of photostability

Selected jute fabric samples of a specified size were placed in position before the uv lamp (MBT lamp) in a fade-o-meter from Shirley Development Limited, UK for photoexposure for different time periods (20, 40, 80, 160 and 320 h). One half portion of each fabric sample was appropriately covered with a piece of black paper (to provide a control section for visual assessment of photo-fading) before placing the same in the
fade-o-meter. Whiteness index values for the photoexposed samples were measured and a fall in the property parameter on photo exposure was noted. A lower drop is taken as a higher order of photostability.

3.3.18 Isolation of different major constituents of jute for compositional characterisation

3.3.18.1 Determination of Fats and Waxes

Jute fibre (2 - 5g) was extracted with about 250 - 300 ml of a ethyl alcohol and benzene (1 : 2, V/V) in a soxhlet apparatus for 72 h to remove fats and waxes from the fibre. The fibre was then dried, washed with ethyl alcohol and distilled water in successive steps before final drying under vacuum at 50°C. The dried fibre was then weighed and the loss in weight on 100 g of oven-dry raw fibre was used to express the fat and wax content (%).

3.3.18.2 Determination of lignin

2g of dewaxed (dried) fibre was treated with 100 ml of 72% H₂SO₄ at about 20°C for 2 h to dissolve the carbohydrate fractions of jute. The acid was then diluted with distilled water to 3% sulphuric acid and was gently boiled. The solution was then cooled and the residual matter containing the lignin part was allowed to settle. It was filtered through a weighed sintered-bed glass crucible (IG-3), washed with distilled water, dried and
weighed. Lignin content (%) was expressed on the basis of oven dry (initial) jute fibre.

3.3.18.3 **Determination of \( \alpha \)-cellulose and hemicellulose**

2 g of jute fibre was treated with 0.7% NaClO₂ solution at boil for 2 h at pH = 4 - 5 for delignification. Thereafter the material was filtered on a tared sintered-bed glass crucible, washed with distilled water and dried under vacuum before weighing. The residual fraction constituted \( \alpha \)-cellulose and hemicellulose. The hemicellulose fraction of the residue was dissolved out by treating the same with about 200 ml of 18% (w/w) NaOH solution at 20° - 25°C for 60 min. The residual material constituted the \( \alpha \)-cellulose fraction. It was washed thoroughly with water, dried under vacuum and weighed. The loss in weight on NaOH treatment accounted for the hemicellulose content. \( \alpha \)-Cellulose and hemicellulose contents were expressed in weight percent based on oven dry (initial) jute in each case.

3.3.18.4 **Determination of Ash**

Ash content was determined by ashing 2 g of (initial) jute by taking it in a platinum crucible and appropriately heating it successively over a bunsen burner for 2 h and in a muffle furnace at about 650°C till a constant weight was obtained on cooling in a desiccator. Ash content was also expressed in weight percent based on oven dry (initial) jute.
3.3.19 Determination of copper number of selected jute samples

Selected jute samples were subjected to estimation of copper number (a measure of aldehydic group content in the cellulose molecular chain) as per Schwalbe - Braidy method as described below.

A weighed quantity (2.5 g) of bone dry jute fibre sample taken out of the relevant yarn or fabric was cut into small pieces and taken in a 100 cc conical flask to which was added a boiling mixture of 5 cc of aqueous solution - A (100 g per litre of crystalline copper sulphate) and 95 cc of solution - B (50 g of NaHCO₃ and 350 g of crystalline Na₂CO₃, 10H₂O per litre) freshly prepared. After stirring, the mouth of the flask was covered and the flask was kept immersed up to the neck in a rapidly boiling water bath for 3h. The contents were then filtered by suction through a sintered-bed glass crucible (IG₃) and the residue was successively washed with hot dilute Na₂CO₃ solution (10 g Na₂CO₃/lit) and then with distilled water; the filtrate and the washings were discarded. Over the residue on the filter was poured 50 cc of hot solution-C (100 g of ferric alum and 140 cc concentrated H₂SO₄ per litre) divided into four equal parts and the residue was then washed with 2 N hot dilute sulphuric acid. The combined ferric alum and acid washings were titrated with solution-D (0.04N potassium permanganate solution); a control experiment was also performed by titrating 50 c.c. of ferric alum
solution (solution-C) using the said KMnO₄ solution (solution-D).
The copper number was then calculated as follows:

\[
\text{Copper number} = \frac{(V_1 - V_2) \times 0.04 \times 0.06357}{W} \times 100
\]

\(V_1\) = ml of the 0.04 N, KMnO₄ solution required for the control experiment.

\(V_2\) = ml of the 0.04 N, KMnO₄ solution required for the test sample.

\(W\) = oven dry weight in g of the fibre sample.

3.3.20 Determination of -COOH group content of selected jute samples

Selected samples of jute were analysed for -COOH groups present in them iodometrically as per IS : 1560 (part - I) - 1974, after making the samples cation free by treatment with 0.5N HCl at room temperature for 2 h followed by washing with distilled water to an acid free condition. For estimation of -COOH functional groups, the above mentioned cation free and air dried fibre sample (0.5 g) was added to 50 ml of a freshly prepared aqueous solution containing KI and KIO₃ (13.83 g KI + 3.57 g KIO₃ per litre) in CO₂ free distilled water together with 20 ml of N/100 thiosulphate solution in a well fitted glass stoppered conical flask. The fibre was spread out evenly in the liquid with glass rod and the flask was shaken thoroughly for about 30 min. The excess of thiosulphate (Na₂S₂O₃) was then titrated with N/100 iodine solution and starch indicator. A control experiment was
performed under identical conditions without using a fibre sample and the titre value for the control experiment was taken into account in calculating the -COOH content expressed in meq/100 g of the fibre.

\[
-\text{COOH gr content (meq/100 g)} = \frac{(V_1 - V_2) \times N \times 100}{M}
\]

where,

- \( V_1 \) = ml of iodine solution required for the control experiment
- \( V_2 \) = ml of iodine solution required for the test fibre sample
- \( N \) = Normality of iodine solution
- \( M \) = Oven dry mass in g of cation free jute fibre.
REFERENCES


14. 'Chemical processing of jute fabrics for decorative end use - part-I, (Bleaching treatment for jute); IJIRA, Calcutta (1977) 5.


31. AATCC (American Association of Textile Chemist and Colorist)
   for testing and materials, philadelphia, pennsylvania, 1976, 219.
33. A. B. Sengupta and T. Radhakrishnan, in New ways to produce
   Textiles, Textile Institute, Manchester, UK, 1972, 116.
34. Instrumental analysis of cotton cellulose and modified cotton
   (1957) 998.
37. V. Sundaram, Handbook of method of tests, Cotton
   Technological Research Laboratory (ICAR) Bombay, 1979, 207, 212.
38. R. S. Hunter, in 'Measurement of Appearance' Interscience,
   whiteness and yellowness of near white opaque material, in
   (1947) 446.


