CHAPTER 3

MATERIALS AND METHODS

3.1 METHODOLOGY OF THE PRESENT RESEARCH WORK

Fabric samples were produced starting from grey cotton fabric of light and medium weight and construction of plain weave using statistical sample size (n=6). The scoured fabrics were subjected to bleaching operations involving three types of bleaching agents (H₂O₂, SPB and enzyme) and their combinations at three concentrations (corresponding to half, three-fourth and full bleach whiteness). The bleached fabrics were dyed for one other trial. These bleached and dyed fabrics were given finishing treatments by Pad-dry-cure technique at optimized concentration (20gpl) and standardized operational parameters. Thus ‘Cool’ finished, ‘Lotus’ finished and ‘Lotus plus Cool’ finished fabric samples were developed and evaluated by conducting different characteristic tests. The test results were statistically analyzed and conclusions have been drawn.

For all the studies 100% cotton yarn of 40° Ne was used for the weaving of fabric samples. Three types of fabric substrates (A,B,C) of light and medium weight (Range of gsm:110-150) and plain construction for woven fabric samples were used. The design of experiments is as per the titles of studies listed in the Tables 3.1, 3.2 and 3.3 and Flow-charts given in Figures 3.1 and 3.2 which are covered sequentially in the following sections. 40° Ne yarn characteristics and the process parameters for weaving are given below:
3.1.1 40s Ne Warp and Weft - Yarn Characteristics:

- U% : 11%;
- (Thick + Thin + Neps) : Imperfections per Km : 70;
- CSP : 2800.

3.1.2 Fabric Weaving Particulars

LAKSHMI RUTI-C1000 Automatic Loom,

Width:180 cm (71”); Loom Speed: 200 rpm,

Fabric particulars: 96 ends x 64 picks (Type B fabric),

Reed Count: 96; Reed Space: 163 cm (64”),

Weave: Plain,

Warp and weft count: Ne 40s

Total no. of ends: 6384

Fabric particulars: 134 ends x 76 picks (Type A fabric); Reed count: 4/67

Fabric particulars: 100 ends x 80 picks (Type C fabric); Reed count: 100
Table 3.1 Samples and their Process Sequence for Studies on ‘Cool’ Finished Fabrics (Fabric Type A)

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Sample Name</th>
<th>Process Sequence</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1(C)</td>
<td>Control sample</td>
<td>Desized, Scoured and Mercerized.</td>
</tr>
<tr>
<td>S2</td>
<td>½ bleached sample</td>
<td>Mercerized, H$_2$O$_2$ bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S3</td>
<td>¾ bleached sample</td>
<td>Mercerized, H$_2$O$_2$ bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S4</td>
<td>Full bleached sample</td>
<td>Mercerized, H$_2$O$_2$ bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S5</td>
<td>½ bleached sample</td>
<td>Mercerized, H$_2$O$_2$ + SPB bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S6</td>
<td>¾ bleached sample</td>
<td>Mercerized, H$_2$O$_2$ + SPB bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S7</td>
<td>Full bleached sample</td>
<td>Mercerized, H$_2$O$_2$ + SPB bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S8</td>
<td>½ bleached sample</td>
<td>Mercerized, SPB bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S9</td>
<td>¾ bleached sample</td>
<td>Mercerized, SPB bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S10</td>
<td>Full bleached sample</td>
<td>Mercerized, SPB bleached &amp; PVA finished</td>
</tr>
<tr>
<td>S11</td>
<td>½ bleached sample</td>
<td>Unmercerized, SPB bleached &amp; PVA finished with auxiliary</td>
</tr>
<tr>
<td>S12</td>
<td>¾ bleached sample</td>
<td>Unmercerized, SPB bleached &amp; PVA finished with auxiliary</td>
</tr>
<tr>
<td>S13</td>
<td>Full bleached sample</td>
<td>Unmercerized, SPB bleached &amp; PVA finished with auxiliary</td>
</tr>
<tr>
<td>S14</td>
<td>½ bleached sample</td>
<td>Mercerized, SPB bleached &amp; PVA finished with auxiliary</td>
</tr>
<tr>
<td>S15</td>
<td>¾ bleached sample</td>
<td>Mercerized, SPB bleached &amp; PVA finished with auxiliary</td>
</tr>
<tr>
<td>S16</td>
<td>Full bleached sample</td>
<td>Mercerized, SPB bleached &amp; PVA finished with auxiliary</td>
</tr>
</tbody>
</table>
Table 3.2  Samples and their Process Sequence for Studies on ‘Lotus’ Finished and ‘Cool’ Finished Fabrics (Fabric TypeB)

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Sample Name</th>
<th>Process Sequence</th>
</tr>
</thead>
<tbody>
<tr>
<td>W01</td>
<td>Scoured Sample (Conventional)</td>
<td>Desizing – Conventional Scouring</td>
</tr>
<tr>
<td>W02</td>
<td>Scoured Sample (Bio-Scoured)</td>
<td>Desizing – Bio Scouring</td>
</tr>
<tr>
<td>W1</td>
<td>Control Sample</td>
<td>Desized, Scoured &amp; Unmercerized – Conventional H₂O₂ Bleaching – Sourcing</td>
</tr>
<tr>
<td>W2</td>
<td>Base fabric sample</td>
<td>Desizing – Bio-Scouring – Bleaching with Enzyme – Souring</td>
</tr>
<tr>
<td>W3/W9</td>
<td>15 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Finishing (L/L+C)</td>
</tr>
<tr>
<td>W4/W10</td>
<td>25 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Finishing (L/ L+C)</td>
</tr>
<tr>
<td>W5/W11</td>
<td>35 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Finishing (L/ L+C)</td>
</tr>
<tr>
<td>W6/W12</td>
<td>15 gpl lotus finished fabric</td>
<td>Desizing – Bio-Scouring – Bleaching with Enzyme – Sourcing- Finishing (L/ L+C)</td>
</tr>
<tr>
<td>W7/W13</td>
<td>25 gpl lotus finished fabric</td>
<td>Desizing – Bio-Scouring – Bleaching with Enzyme – Sourcing- Finishing (L/ L+C)</td>
</tr>
<tr>
<td>W8/W14</td>
<td>35 gpl lotus finished fabric</td>
<td>Desizing – Bio-Scouring – Bleaching with Enzyme – Sourcing- Finishing (L/ L+C)</td>
</tr>
</tbody>
</table>
Table 3.3 Samples and their Process Sequence for Studies on ‘Lotus + Cool’ Finished Fabrics (Fabric Type C)

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Sample Name</th>
<th>Process Sequence</th>
</tr>
</thead>
<tbody>
<tr>
<td>BW1</td>
<td>Control sample</td>
<td>Desizing – Scouring- Bleaching with H₂O₂ – Sourcing</td>
</tr>
<tr>
<td>BW2</td>
<td>10 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Finishing</td>
</tr>
<tr>
<td>BW3</td>
<td>15 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Finishing</td>
</tr>
<tr>
<td>BW4</td>
<td>20 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Finishing</td>
</tr>
<tr>
<td>BW5</td>
<td>25 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Finishing</td>
</tr>
<tr>
<td>DW1</td>
<td>Control sample</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Dyeing- Finishing</td>
</tr>
<tr>
<td>DW2</td>
<td>10 gpl Lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Dyeing- Finishing</td>
</tr>
<tr>
<td>DW3</td>
<td>15 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Dyeing - Finishing</td>
</tr>
<tr>
<td>DW4</td>
<td>20 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Dyeing- Finishing</td>
</tr>
<tr>
<td>DW5</td>
<td>25 gpl lotus finished fabric</td>
<td>Desizing – Scouring – Bleaching with H₂O₂ – Sourcing- Dyeing -Finishing</td>
</tr>
</tbody>
</table>
PURCHASE OF YARN

GREY FABRIC WEAVING
PLAIN WEAVE
FABRIC WIDTH = 155 CM (62”)

STUDY 1
FABRIC A
(MEDIUM WT.)
EPI=136  PPI=74
GSM=140

METRES OF WOVEN FABRIC
44
NO. OF 4 SQM SAMPLES 16

STUDY 2
FABRIC B
(LIGHT WT.)
EPI=96  PPI=74
GSM=122

METRES OF WOVEN FABRIC
20
NO. OF 4 SQM SAMPLES 07

STUDY 3
FABRIC C
(MEDIUM WT.)
EPI=100  PPI=80
GSM=130

METRES OF WOVEN FABRIC
28
NO. OF 4 SQM SAMPLES 10

CHEMICAL PROCESSING

DESIZING 138 SQM
SCOURING 138 SQM
BLEACHING 138 SQM
SOURING 138 SQM

MERCERISING 108 SQM

COOL FINISHING
(16 SAMPLES)
64 SQM

LOTUS & COOL FINISHING
(07 SAMPLES)
28 SQM

LOTUS + COOL FINISHING
(10 SAMPLES)
40 SQM

Figure 3.1  Flow Chart for Research Methodology (A)
PURCHASE OF YARN

2/40 Ne COTTON WARP (B)
40 Ne COTTON WEFT

GREY FABRIC WEAVING
PLAIN WEAVE
FABRIC WIDTH = 155 CM (62”)

STUDY 4
FABRIC B
(LIGHT WT.)
EPI=96  PPI=74
GSM=122

METRES OF
WOVEN FABRIC
20
NO. OF 4 SQM
SAMPLES 07

CHEMICAL PROCESSING

DESIZING 30 SQM

BIO SCOURING 30 SQM

BIO BLEACH 30 SQM

SOURING 30 SQM

LOTUS & COOL FINISHING
(07 SAMPLES)
28 SQM

Figure 3.2  Flow Chart for Research Methodology (B)
3.2 STUDY 1: DEVELOPMENT OF ‘3T’ MODELS FOR COMFORT EVALUATION OF ‘COOL’ FINISHED COTTON FABRICS

Bleached and finished cotton fabric samples were produced sixteen (16) in numbers, in which six fabric samples were finished with ‘Cool’ finish.

3.2.1 Materials

Cotton yarn in warp and weft of 40s Ne count with ends and picks of 136 and 74 in plain weave fabric, was used for this research study. Sodium hydroxide, hydrogen peroxide and sodium per-borate were used for scouring, mercerizing and bleaching of the above cotton fabric. Hydrophilic poly vinyl acetate, PVA (Nikhil Adhesives, Mumbai) and emulsified polyester based softener, Finish Soft V (a formulation of Padma Traders, Coimbatore) were used to impart ‘cool’ finish to the fabric.

3.2.2 Desizing and Scouring

Desizing was done using α-amylase enzyme (bacterial) of 2% owf in an industrial jigger of 50 Kg capacity. Scouring was also done in the same Table 3.4.

Table 3.4 Recipe for Scouring

<table>
<thead>
<tr>
<th>S. No</th>
<th>Chemical / Process detail</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Caustic soda (NaOH)</td>
<td>5% owf</td>
</tr>
<tr>
<td>2</td>
<td>Soda ash (Na₂CO₃)</td>
<td>1.5% owf</td>
</tr>
<tr>
<td>3</td>
<td>Wetting agent</td>
<td>0.25% owf</td>
</tr>
<tr>
<td>4</td>
<td>Temperature</td>
<td>95°C</td>
</tr>
<tr>
<td>5</td>
<td>Time</td>
<td>2 hrs</td>
</tr>
<tr>
<td>6</td>
<td>MLR</td>
<td>1:10</td>
</tr>
</tbody>
</table>
3.2.3 Mercerizing

Mercerization was done in the same industrial jigger using sodium hydroxide of 20% concentration with MLR 1:10 at room temperature followed by rinsing, washing and drying in sunlight.

3.2.4 Bleaching

The bleaching treatment was done in the same industrial jigger using $\text{H}_2\text{O}_2$ with requisite quantities of chemicals as per process particulars followed in industry, given in Table 4.1. In the novel bleaching treatment, additionally, requisite quantities of SPB are taken in combination with $\text{H}_2\text{O}_2$. The fabric was further subjected to bleaching process as per following recipes:

$\text{H}_2\text{O}_2$ and SPB bleaching with 1%, 2%, 3% and 2, 3, 4% respectively of bleaching agent on the weight of fabric (owf); pH stabilizer 1.5% owf and soda ash 3% owf were used. In the combined process, for the three concentrations of $\text{H}_2\text{O}_2$, namely, 0.5, 1, 1.5% owf additional 1, 2, 3% owf of SPB were added. The temperature of bleaching was kept at 90°C for a time of treatment of 2 hours and MLR of 1:8. Two different methods of finishing operations were carried out on five sets of bleached fabrics. In the first method of finishing, hydrophilic PVA of 20 gpl concentration was padded uniformly on the three sets of bleached fabrics (S2-S10) mentioned in Table 4.1. These fabrics were subjected to Pad-Dry-Cure technique of finishing. In the second trial of finishing, in addition to PVA, soft finish auxiliary of emulsified polyester based softener of 20 gpl concentration was used for the un-mercerized fabrics (S11-S13) and mercerized fabrics (S14-S16). Pad-Dry-Cure technique was adopted for these six fabric samples also. The drying of the finished fabric was done on a miniature stenter at 110°C with fabric speed of 30 m/min and curing at 140°C with a fabric speed of 40 m/min.
3.2.5 Sourcing

Souring was carried out using Acetic acid to neutralize the alkaline condition of the bleached fabric. The industrial jigger was used in the usual manner Table 3.5.

Table 3.5 Recipe for Sourcing

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Chemical / process detail</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Acetic acid</td>
<td>2% owf</td>
</tr>
<tr>
<td>2</td>
<td>Time</td>
<td>30 min</td>
</tr>
<tr>
<td>3</td>
<td>Temperature</td>
<td>R.T</td>
</tr>
<tr>
<td>4</td>
<td>MLR</td>
<td>1:10</td>
</tr>
</tbody>
</table>

3.2.6 Finishing

Finishing was carried out in padding mangle attached to stenter of small size. It was done to impart water absorbency and stiffness to fabric. 20 gpl PVA was used as the finishing agent and applied on the fabric by pad – dry – cure process, with MLR of 1:2 and expression of 70%.

3.2.7 Effluent Testing

Using TDS Meter, the total dissolved solids (TDS) in the effluent are measured, during the bleaching process. For other characteristics, different estimation methods are available (refer Chapter 2).
3.3 STUDY 2: COMFORT CHARACTERISTICS OF COTTON FABRICS FINISHED WITH FLUORO-ALKYL NANO-LOTUS FINISH

The influence of nano-lotus finish in conjunction with a silicone softener on the comfort characteristics of finished cotton fabrics has been studied. Fabric handle, thermal and hygral comfort in terms of total hand value, thermal insulation value, and moisture transport characteristics have been analyzed through a new expression, equated by ‘3T’ values. “Nano-Lotus finish” is a special effect, incorporated using fluoro -alkyl based emulsion, to impart functional finish to apparel fabrics. This effect has high commercial importance in view of repellency for oil and water with soil release characteristics. Fluoro-alkyl, non-ionic nano-emulsion is applied on the cotton woven fabric by pad-dry-cure technique at three different finish liquor concentrations of 15, 25 and 35 gpl with 65-70% expression at padding. The tactile, thermal and transport characteristics of finished fabrics have been critically analyzed using Kawabata Evaluation System and Thermo Lobao -model II. It is observed that the properties that show development of ‘lotus effect’ are geometric roughness, fabric stiffness, fabric compressional resilience.

3.3.1 Materials

Grey cotton plain woven fabric was used with the fabric particulars: 2/40s Ne warp, 40s Ne weft, 96 ends / inch, 74 picks /inch and 122g/m² fabric weight. Nano-chemical of fluoro-carbon family in the form of non-ionic fluoro alkyl emulsion was procured from Resil Chemicals, Bangalore to impart oil and water repellency with soil release to the fabric. Polyester resin and iso-propyl alcohol were used as binder and absorbent in the finish liquor.
3.3.2 Processing Methods

The grey cotton woven fabric was scoured, bleached, soured using winch machine. The process parameters and recipes are given below in Table 3.6.

**Table 3.6 Process Particulars**

<table>
<thead>
<tr>
<th>Scouring</th>
<th>Finishing (Stage 1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Caustic soda : 5% owf</td>
<td>Silicone softener: 2% owf</td>
</tr>
<tr>
<td>Soda ash : 1.5% owf</td>
<td>Antifoaming agent : 0.5% owf</td>
</tr>
<tr>
<td>Wetting agent : 0.25% owf</td>
<td>MLR: 1:2</td>
</tr>
<tr>
<td>Temperature : 95°C ; MLR : 1:10</td>
<td><strong>Temperature 32°C</strong></td>
</tr>
<tr>
<td>Time : 2-2.5 h</td>
<td></td>
</tr>
</tbody>
</table>

**Bleaching**

<table>
<thead>
<tr>
<th>Finishing (Stage2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen peroxide : 3% owf</td>
</tr>
<tr>
<td>Stabilizer : 1.5% owf</td>
</tr>
<tr>
<td>Soda ash : 3% owf ; Time: 2h</td>
</tr>
<tr>
<td>Temperature : 90°C; MLR: 1:8</td>
</tr>
<tr>
<td>Polyester resin : 10 gpl</td>
</tr>
<tr>
<td>Isopropyl alcohol : 10 gpl</td>
</tr>
<tr>
<td>MLR : 1:2</td>
</tr>
</tbody>
</table>

**Souring**

<table>
<thead>
<tr>
<th>Temperature 32°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetic acid : 2% owf; Time:20min</td>
</tr>
<tr>
<td>Temperature : 32°C ; MLR : 1:8</td>
</tr>
</tbody>
</table>

The nano-lotus finishing of the cotton fabric was carried out in two stages. In the first stage the cotton fabric was treated with silicone softener of organo-modified poly-siloxane from Resil Chemicals, Bangalore using tumble washing machine to obtain a high degree of softness and fullness to
the fabric. This is also because the fabrics finished by fluoro-alkyl nano-lotus finish tend to become brittle and stiff, and a softener can make the fabric soft and supple to the required degree at the pretreatment stage itself instead of in the finishing. In the second stage, the silicone finished cotton fabric was immersed into the lotus finishing liquor and processed by pad-dry-cure technique. The drying of the finished fabric was carried out at 110°C with 30 m/min speed, and then it was followed by curing at 140°C with 40 m/min speed using miniature stenter.

3.3.3 Test Methods

The nano-lotus finished fabric characteristics, such as tensile, shear, bending, compression, surface friction, variation in roughness and air resistance, were tested using Kawabata Evaluation System (KES).

Thermal properties, namely, thermal insulation value (TIV) and thermal energy (q max), were measured on Thermo-lobao model II as per ASTM D1518-85 standard procedure. Moisture transport was measured in the same instrument for all fabric samples and it was expressed as the weight of moisture in gram/m² of fabric per second. To assess the degree of finish, the fabric weight gain % of finished fabric samples was determined.

To evaluate the degree of whiteness, the CIE whiteness index (WI) values of the control and nano-lotus finished fabrics were recorded using Premier spectrophotometer Colour Scan model SS 5100A.

Water – repellency test was carried out on ten fabric samples of the size 20cm X 20cm for each concentration of finish, cut from control fabric and nano-lotus finished fabrics. The initial weights of the fabric samples were recorded and known quantities of water droplets were allowed to fall on the fabric samples and their final weights were measured. To evaluate water
repellency characteristics of the finished fabric, ASTM D-2721 (1989) Standard procedure was used.

Soil release test was performed on ten samples of size 5cm X 5cm cut from control and nano-lotus finished fabrics. These fabrics were soiled with charcoal-machine oil paste. Out of the ten samples treated with 15gpl finish concentration, five samples were subjected to soaping for a period of 90 min. After soaping, the fabric samples were rinsed, washed, dried and compared with soiled control fabric samples as per AATCC 130-1981 standard procedure. Since the nano-lotus finish affects the hydrophilic nature of the cotton fabric, the assessment of thermal and hygral comfort of the fabric was carried out. Thermal and hygral properties are of paramount importance for the hygroscopicity of the nano-lotus finish. It is observed that nano-lotus finishes are hydrophobic in character and due to special chemical constitution of some of the finishes they exhibit super-hydrophobicity. Thus the present study acclaims practical relevance and importance in regard to the study of these characteristics.

Fabric samples of all finish concentrations were subjected to Scanning Electron Microscope (SEM) exposures at different magnifications apart from control fabric sample. This was carried out to ascertain the presence and the extent of finish deposits on the surface of the treated fabric.

Fourier transform infra-red (FTIR) spectra were obtained on finish chemical and on fabric treated with 15gpl finish concentration as it amply met the requirement of water-repellency. FTIR spectral analysis was carried out to ascertain the identical chemical nature of peaks and bands in the two spectra, which is independent of concentration.

Thermal Insulation Value (TIV) represents thermal resistance, resulting in cool touch feeling and warm feeling to body, for a fabric meant
for apparel. Moisture Vapor Transport is yet another characteristic important for comfort and thus the prime characteristics, namely, THV, TIV and Transport of moisture (MT) can well define the combination of handle and comfort. Other secondary characteristics representative of handle are drape, bi-axial stress relaxation, tensile modulus, crease recovery to mention a few. Similarly, in the case of comfort the secondary characteristics are air-permeability or breathe-ability, wicking height. It gave rise to the ‘evolution’ of ‘3T models’ in the present work, by taking into consideration a combination of three prime or secondary variables which are mutually independent measures. However, the secondary characteristics are dependent on the prime characteristics.

Tensile testing of the fabric samples was conducted on Instron fabric tester. Drape testing of the fabric samples is another method of evaluating the handle characteristic of a finished fabric.

The whiteness on the basis of CIE color indices of the fabric samples were assessed by computer colour matching system attached with a color scan and spectro-photometer. Soil release test was carried out by the standard procedure of AATCC Spray test for evaluating the multi functional characteristics of finished fabrics.

An advanced technique of characterizing the water / oil repellency of a lotus finished fabric sample is by the DRA test, which determines the dynamic rolling angle on the fabric by a droplet of water or oil. Use of a crease recovery tester was made to conduct this test. FTIR Spectral Distribution studies were conducted for identifying the chemical groups and elements present in the finish applications on the fabric samples. SEM studies were carried out for analyzing the surface characteristics of the treated fabrics.
3.4 STUDY 3: STUDIES ON CONVENTIONAL BLEACHED AND REACTIVE DYED COTTON FABRICS FINISHED WITH NANO-‘LOTUS’ FINISH IN CONJUNCTION WITH ‘COOL’ FINISH AUXILIARY AND CROSS LINKED WITH DMDHEU BASED RESIN

3.4.1 Materials

The bleached fabric of Fabric Type C was used for this study and it was finished at four different concentrations of 10 gpl, 15 gpl, 20gpl & 25 gpl of Fluoro-alkyl nano-‘lotus’ finish combined with ‘cool’ finish auxiliary, namely, hydrophilic polyester resin based emulsion of 20 gpl concentration. The finish liquor was also made up with silicone softener and DMDHEU based cross-linking agent each of 20gpl concentration.

3.4.2 Test Methods

The ten (10) fabric samples of bleached and dyed varieties given with the above treatment were subjected to studies of low stress mechanical properties on KES and comfort related characteristics on Thermo-Lobao Model II. The different test methods used for testing all the fabric samples are detailed in section 3.6.

3.5 STUDY 4 STUDIES ON CONVENTIONAL BLEACHED, BIO-SCOURED AND BIO-BLEACHED COTTON FABRICS FINISHED WITH NANO- ‘LOTUS’ AND ‘LOTUS PLUS COOL’ FINISHES

3.5.1 Materials

In the study, starch sized, plain woven 100% cotton fabric, with the fabric particulars 2/40s Ne warp count, 40s Ne weft count, 96 ends / inch, 74 picks /inch and 122g/m² fabric weight (Fabric Type B) was used.
3.5.2 **Enzymatic Desizing**

Starch sized fabric was treated with a commercial amylase (Rapidase L40) with 1g/l, 70ºC for 60 min, at pH 5 in 0.1 M acetic buffer.

3.5.3 **Enzymatic Scouring:**

The fabrics were scoured with an alkaline pectinase enzyme, Bio – Scour N (from Resil Chemicals, Bangalore) of enzyme activity 20 APSU/g cotton in 0.05 mole phosphate buffer at pH 8, for two hours at 55º C in the presence of 0.1% of non-ionic surfactant Sandozin NLA (Sandoz).

3.5.4 **Standard Bleaching Process**

The fabrics were bleached with the following recipe: Silicon 3.5% owf., Soda ash 1% owf., Sodium hydroxide 1% owf, 35% (vol.) Hydrogen peroxide – 4% owf., for 1 hour at 90º C.

3.5.5 **Bleaching of Bio-Scoured Fabrics**

Cotton fabrics, scoured with alkaline pectinase were bleached (pH 10-11, 90º C, 60 min) with enzymatically produced peroxide (0.408 g/l). The results of whiteness from the bleaching with enzymatically produced peroxide were comparable to the results obtained with a standard bleaching process (0.17 g/l hydrogen peroxide according to the recipe – 4% owf., 35% H₂O₂).

3.5.6 **Chemicals Used**

Sodium hydroxide, Wetting oil, Hydrogen peroxide, Peroxide killer, Scouring enzyme and Stabilizer.
3.5.7 Scouring Enzyme and Stabilizer

Bio-scour N is the commercial scouring enzyme of Resil Chemicals, Bangalore. It is unstable in alkaline pH. The temperature should be around boil. A stabilizer admixed with the Enzyme formulation has its role in preventing from decomposing Hydrogen peroxide in diluted alkaline medium.

3.5.8 Bio-Scouring

<table>
<thead>
<tr>
<th>Component</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bio-Scour N</td>
<td>2 % o.w.f</td>
</tr>
<tr>
<td>Wetting Agent</td>
<td>0.5 % o.w.f</td>
</tr>
<tr>
<td>Hydrogen Peroxide</td>
<td>2 % o.w.f</td>
</tr>
<tr>
<td>Temperature</td>
<td>95º–98º C</td>
</tr>
<tr>
<td>Time</td>
<td>45 min. – 1 hr.</td>
</tr>
<tr>
<td>MLR</td>
<td>1:8 to 1:10</td>
</tr>
</tbody>
</table>

3.5.9 Bio-Bleaching

This process is similar to Bio-Scouring. The only difference is the concentration of Hydrogen peroxide. In Bio-Bleaching, the concentration of H₂O₂ is 8% owf., along with Bio-Scour N and Wetting agent. In this process the use of number of chemicals are minimized. Thus it occurs to be eco-friendly. Given below is the recipe for Bio-Bleaching: MLR =1:8 to 1:10.

<table>
<thead>
<tr>
<th>Component</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bio-Scour N</td>
<td>2% o.w.f</td>
</tr>
<tr>
<td>Wetting Agent</td>
<td>0.5% o.w.f</td>
</tr>
<tr>
<td>Hydrogen Peroxide</td>
<td>8% o.w.f</td>
</tr>
<tr>
<td>Temperature</td>
<td>95º–98º C</td>
</tr>
<tr>
<td>Time</td>
<td>45 min</td>
</tr>
</tbody>
</table>
3.5.10 Finishing

The bleached fabrics were subjected to finishing treatment with Fluoro-alkyl nano-‘lotus’ finish at three different concentrations of 15gpl, 25gpl and 35gpl similar to the Study 2.

3.6 TEST METHODS

The different test methods adopted for the studies are detailed below in sections 3.6.1 to 3.6.20.

3.6.1 Fabric Weight Loss (%)

The fabric weight loss % of different samples have been estimated using the formula given below:

\[
\text{% weight loss} = \frac{W_1 - W_2}{W_1} \times 100
\]  \hspace{1cm} (3.1)

where, \( W_1 \) and \( W_2 \) are the weights of the scoured (base fabric-S1) and bleached fabric samples (S2-S16), respectively.

3.6.2 Fabric Weight Gain (%)

To assess the degree of finish, the fabric weight gain % of finished fabric samples were determined. The fabric weight gain % of different samples has been estimated using the formula given below:

\[
\text{Fabric weight gain %} = \frac{W_2 - W_1}{W_1} \times 100
\]  \hspace{1cm} (3.2)

where, \( W_1 \) and \( W_2 \) are the weights of the bleached control fabric \( (W_1) \) and finished fabric samples \( (W_2) \) respectively.
3.6.3 **Wettability Area Test (BS 4554)**

A known quantity of water was sprinkled on the fabric sample and the circular area of water absorbed by the fabric after a time lapse of one minute was measured, using a one foot scale.


The wicking heights for all the fabric samples were measured in warp and weft directions (in cm) as per AATCC test procedure.

3.6.5 **Air Resistance and Air Permeability (BS 5636)**

Air permeability is defined as the volume of air in milliliters which is passed in one second through 100 mm$^2$ of the fabric at a pressure difference of 10mm head of water. It is expressed as (cc)/sec/100mm$^2$. The reciprocal of air permeability is air resistance, which is defined as the time in seconds for 1 ml of air to pass through 100mm$^2$ of fabric under a pressure head of 10mm of water. ASTM Standard procedure (D73.7-96) was used.

3.6.6 **Thermal Insulation Value (TIV)**

Thermal properties, namely, Thermal Insulation Value (TIV) and thermal energy (q max) were measured on Thermo-labao model II as per ASTM D1518-85 standard procedure. Thermal Insulation Value (TIV) is the percentage saving in heat loss from a surface, due to covering it with the fabric, which is measured using the following formula:

$$T.I.V = \frac{100 \ (H_o - H_c)}{H_o} \quad \text{(in tog units)} \quad (3.3)$$
where, \( H_0 \) = the heat lost per second from the uncovered surface, and \( H_c \) = the heat lost per second from covered surface.

\[ 1 \text{ tog} = 0.1^\circ \text{C} \text{ m}^2/\text{W} \]

Thermal transmittance involves heat transfer from the ambient to the fabric sample. This is another thermal property designated as \( q_{\text{max}} \) and expressed in the units of W/cm\(^2\). This is measured as thermal energy (W) per cm\(^2\) of the fabric specimen for 10\(^\circ\)C rise in fabric temperature. ASTM Standard procedure (D1518 – 85) was used.

### 3.6.7 Moisture Transport (ASTM E96-80)

Moisture transport was measured in the same instrument for all fabric samples and it was expressed as the weight of moisture in grams per square metre of fabric per second.

### 3.6.8 Fabric Drape Test (AATCC EP5)

The drape % (F) is measured for all the fabric samples using the formula given below:

\[
F = \left( \frac{A_s - A_d}{AD - Ad} \right) \quad (3.4)
\]

where,

- \( A_D = \) The area of the specimen
- \( A_d = \) The area of the supporting disc and
- \( A_S = \) The actual projected area of the specimen
3.6.9 Crease Recovery Test

Two sets of fabric samples of sample size five each in warp and weft directions were subjected to Shirley Crease Recovery Angle Test. The combined (W+F) angles of sample size five each were recorded.

3.6.10 CIE Whiteness Index Test (Using Premier Color Scan 5100A)

The whiteness index (WI) of the control, ‘cool’, ‘lotus’ and ‘lotus plus cool’ finished fabrics were tested using Premier Spectrophotometer, Colorscan - Model No. SS 5100A.

3.6.11 The Kawabata Evaluation System (KES-F)

The fabric properties such as tensile, shear, bending and compression behaviour of both control and finished cotton fabrics were analysed using Kawabata Evaluation System (KES-F). The instrument was operated with following modules such as FB1 – for Tensile and Shearing, FB2- for Bending, FB3- for Compression, FB 4- for Surface friction and variation in geometric roughness (as per AATCC EP5).

3.6.12 Instron Fabric Tensile Strength Test

Fabric strip strength tests were carried out for all fabric samples according to the ASTM standard test method.

The mean breaking load and extension were measured for all samples. A new measure of fabric tensile modulii at 10mm extension for warp and weft directions (5% of total sample length of 20cm) was carried out for all the samples. The average of these results was used to compute the tensile modulus at 500gf and 200gf tensile loads seperately. After the load test, the relaxation % in warp and weft directions was measured and recorded.
3.6.13 Soil Release Test

Soil release test was performed on ten samples of size 5cm x 5cm cut from control and ‘Lotus plus Cool’ finished fabrics. These fabrics were soiled with charcoal-machine oil paste. Out of the ten samples treated with finish, five samples were subjected to soaping for a period of 90 minutes. After soaping, the fabric samples were rinsed, washed, dried and compared with soiled control fabric samples as per AATCC 130-1981 standard procedure.

3.6.14 Water and Oil Repellency Test – Spray Test

ASTM D- 2721 (1989) Standard procedure was referred. Sample size of 20cm X 20cm was cut from the control and ‘lotus plus cool’ finished cotton fabrics for oil and water repellent test. The initial weights of the fabric samples were tabulated and known quantities of water droplets were sprayed over the fabric samples. Filter papers equal to sample size were cut and placed over the fabric samples to blot water droplets. Then a known weight was placed over the fabric sample for a period of one minute. After one minute, the filter paper was removed and the final weights of the fabric samples were determined. From the initial and final readings the degree of water repellency was evaluated. The fabric samples were also compared with AATCC Spray test standard photographs, and graded as per standard spray ratings.

There are a few instruments which measure air-resistance in place of air permeability. One such instrument is KES -F.8-AP1 Air permeability tester. In this instrument a constant rate of air flow is generated by the piston motion of plunger/ cylinder mechanism and passed through the specimen into the atmosphere. The suction and discharge periods of air are 5 seconds each and the air pressure loss caused by the air resistance of the specimen is
measured by a semiconductor differential - pressure gauge. The air resistance of the specimen, 'R' is directly indicated on a digital panel meter.

\[ R = \frac{AP}{V} \]  

(3.5)

where,  \( AP \) - Pressure difference (Pa) and  \( V \)  - Rate of air flow ml/m\(^2\)-S.

3.6.15 Water Repellency (Spray Test)

The fabrics, depending on end uses, should have the ability to keep water out (e.g. Jerkin) or to keep water in (e.g. hose pipe). On the other hand, some fabrics must exhibit the ability to absorb water and dry rapidly, towel being an obvious example.

The Spray Test - In this test, a small size, mock rain shower is produced by pouring water through a spray nozzle. The water falls on to the specimen which is mounted over a 6 inch. diameter embroidery frame and fixed at an angle of 45". To carry out the test, 250 ml of water at 70F (21C) is poured steadily into the funnel (Fig. 3.3). After spraying is finished, the sample holder is removed and the surplus water removed by tapping the frame six times against a solid object.

![Figure 3.3 Spray test](image)

Figure 3.3 Spray test
The tapping is in two stages, three taps at one point on the frame and then three times at a point diametrically opposite. The assessment of the fabric's water repellency is given by the 'spray rating'. After removal of surface fibre is accomplished, the fabric surface is examined visually. The American Association of Textile Chemists (AATCC) and colorists recommend the use of photographs against which the actual fabric appearance is compared. The ratings are as follows:

- 100  No sticking or wetting of the upper surface
- 90   Slight random sticking or wetting of the upper surface
- 80   Wetting of upper surface at spray points.
- 70   Partial wetting of whole of upper surface
- 50   Complete wetting of whole of upper surface
- 0    Complete wetting of whole of upper and lower surfaces

The mean of the five ratings is reported.

3.6.16 The Drop Test or Drop Penetration Test / Wettability test

The drop test is a count of the number of drops required to penetrate through to the underside of the fabric when all the drops fall on to the same spot. The basic apparatus is shown in Fig.3.4. The fabric specimen is clipped on to a glass plate with a piece of filter paper sandwiched between the fabric and the glass.
Figure 3.4 The drop or drop penetration test

3.6.17 FTIR Analysis

Fourier Transmission Infra-Red (FTIR) spectra were obtained on finish chemical and on fabric treated with finish. This was carried out to ascertain the identical chemical nature of peaks and bands in the two spectra, which is independent of concentration.

3.6.18 SEM Analysis

Fabric samples of all finish concentrations were subjected to Scanning Electron Microscope (SEM) exposures at magnifications (2000x) in addition to control fabric sample. This was carried out to ascertain the presence and the extent of finish deposits in the form of ‘whiskers’ on the surface of the treated fabric.

3.6.19 Statistical Analysis of Test Results

The statistical analysis of test results was carried out for the one way Analysis of Variance (ANOVA), two-tail correlation analysis and t-tests
made on the parametric variables of the tested fabric samples. For all variables, statistical significant differences at 95% or 99% confidence levels or both were assessed and recorded.

3.6.20 Modeling Studies

The test results of bleached and dyed fabric samples were analyzed and compared for ‘3T’ modeling, ANN modeling and subjective assessment studies by innovative techniques.

3.7 NOVEL BLEACHING PROCESS

A novel bleaching process has been attempted in this work to study the effects of combined bleaching using industry popular hydrogen peroxide and the identical but eco-friendly sodium perborate (SPB) on the finished fabric handle and comfort properties. A comparison has also been made with these individual bleaching agents under the same study, detailed in Chapter 4.

3.7.1 Mechanism of Combined Bleaching of SPB with H$_2$O$_2$

Sodium perborate tetrahydrate (NaBO$_3$.4H$_2$O) is prepared by reaction of sodium borate with hydrogen peroxide. It is considered as a borate containing hydrogen peroxide of crystallization (NaBO$_2$.3H$_2$O.H$_2$O$_2$). When it is dissolved in water, sodium perborate releases the hydrogen peroxide as shown in equations 1 and 2.

\[
\text{NaBO}_3.4\text{H}_2\text{O} \leftrightarrow \text{H}_2\text{O}_2+\text{Na BO}_2+3\text{H}_2\text{O} \quad (3.6)
\]

\[
\text{NaBO}_2.3\text{H}_2\text{O}.\text{H}_2\text{O}_2 \leftrightarrow \text{H}_2\text{O}_2+\text{NaBO}_2+3\text{H}_2\text{O} \quad (3.7)
\]

Several popular theories have stated that the active oxygen (nascent oxygen) is the reactive species in hydrogen peroxide bleaching. This nascent
(atomic) oxygen is claimed to separate easily from perhydroxyl anion (HO$_2^-$) in accordance with the equation 3.

\[
\text{HO}_2^- \rightleftharpoons \text{HO}. + [\text{O}]
\]  

(3.8)

When hydrogen peroxide decomposes, some of the oxygen is released. It is in electronically exited state (singlet oxygen). Singlet oxygen is the active substance in peroxide bleaching. Hydrogen peroxide is present in aqueous solution in dissociation equilibrium with the perhydroxyl anion (HO$_2^-$) and the peroxo dianion (O$_2^{-2}$) (equations 4 & 5).

\[
\text{H}_2\text{O}_2 \rightleftharpoons \text{HO}_2^- + (\text{H}^+) \]  

(3.9)

\[
\text{HO}_2^- \rightleftharpoons \text{O}_2^{-2} + (\text{H}^+) \]  

(3.10)

The perhydroxyl anion may further generate other active species according to the equation 6.

\[
\text{HO}_2^- + \text{H}_2\text{O}_2 \rightarrow \text{HO}_2^- + \text{HO}^- + \text{HO}^- \]  

(3.11)

The perhydroxyl radical (HO$_2^-$) may also dissociate to form the radical anion O$_2^{-2}$, known as superoxide, an active bleach agent.

### 3.7.2 Technical Advantages of the Novel Bleaching Process

As SPB is found compatible and comparable with industry popular hydrogen peroxide in regard to handle and comfort characteristics, it may be envisaged that technically compatible, eco-friendly feasible and economically beneficial results may be obtained with SPB in combination with enzymes suitable for one step process of desizing, scouring and bleaching.