CHAPTER 3

EXPERIMENTAL PLAN

3.1 INTRODUCTION

The present chapter describes about the selection of textile fabrics and natural sources for dye extraction and application. The major dyes are selected from fruit sources that are berberis vulgaris (Bv), bixa orellana (Bo), terminalia chebula (Tc), punica granatum (Pg), allium cepa (Al) and citrus paradisi (Cp). The experimental plan is broadly classified into three major divisions.

The first division describes about the extraction and application of major dyes. These dyes are applied to pretreated natural textile fabrics (cotton, silk and wool).

The second division describes the application of combinational dyeing for shade variation on the textile fabrics. The dyes selected for shade variation are indigofera tinctoria (If), rubia cardifolia (Rc) and pterocarpus santallinus (Pp).

The third division describes the application of combinational dyeing to impart antimicrobial property to the dyed textile fabric. The natural sources selected to add functional property are vitex negundo (Vn), emblica officinalis (Eo), aloe barbadensis (Ab) and azadirachta indica (Ai). Below given Figure 3.1 indicates the procedure of the experimental plan in general.
The final stage of the experimental plan deals with the evaluation of the natural dyes and the natural dyed textile fabrics for FTIR analysis, color fastness testings, antimicrobial assessment and K/S color strength analysis.
3.2 SELECTION OF MATERIALS

3.2.1 Textile Fabrics

Textile fabrics such as Cotton, Silk and Wool with the following specifications were used. Cotton: 1/1 plain weave, 60/50 ends and picks per inch, 60s count, desized, scoured and bleached. Silk: 1/1 plain weave, 80/70 ends and picks per inch, 90s count, degummed. Wool: 1/2 twill weave, 40/40 ends and picks per inch, 50s count, desized, scoured and bleached (Datye and Vaidya 1984).

3.2.2 Major Natural Dyes

The main natural sources selected for the study were fruit source such as berberis vulgaris (Bv), bixaorellana (Bo), terminilia chebula (Tc), punica granatum (Pg), allium cepa (Al) and citrus paradisi (Cp). Table 3.1 shows the major dyes selected for dyeing.

**Table 3.1 Selection of major natural sources**

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Common name</th>
<th>Botanical name</th>
<th>Symbol</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Barberry</td>
<td>Berberis vulgaris</td>
<td>Bv</td>
</tr>
<tr>
<td>2</td>
<td>Annatto</td>
<td>Bixa orellana</td>
<td>Bo</td>
</tr>
<tr>
<td>3</td>
<td>Myrobalan</td>
<td>Terminalia chebula</td>
<td>Tc</td>
</tr>
<tr>
<td>4</td>
<td>Pomogranate</td>
<td>Punica granatum</td>
<td>Pg</td>
</tr>
<tr>
<td>5</td>
<td>Onion</td>
<td>Allium cepa</td>
<td>Al</td>
</tr>
<tr>
<td>6</td>
<td>Grape</td>
<td>Citrus paradisi</td>
<td>Cp</td>
</tr>
</tbody>
</table>

3.2.3 Natural Dyes for Shade Variation

The supporting natural sources used for the combination to bring the shade variation were indigofera tinctoria (If), rubia cardifolia (Rc) and
pterocarpus santallinus (Pp). Table 3.2 shows the natural dyes selected for shade variation.

Table 3.2 Selection of natural dyes for shade variation

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Common name</th>
<th>Botanical name</th>
<th>Symbol</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Indigo</td>
<td>Indigofera tinctoria</td>
<td>If</td>
</tr>
<tr>
<td>2</td>
<td>Madder</td>
<td>Rubia cardifolia</td>
<td>Rc</td>
</tr>
<tr>
<td>3</td>
<td>Red sandal wool</td>
<td>Pterocarpus santallinus</td>
<td>Pp</td>
</tr>
</tbody>
</table>

3.2.4 Natural Sources to Impart Functional Property

The natural sources added to bring the functional property were vitex negundo (Vn), emblica officinalis (Eo), aloe barbadensis (Ab) and azadirachta indica (Ai). Table 3.3 shows the natural sources selected to impart functional property.

Table 3.3 Selection of natural sources to impart functional property

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Common name</th>
<th>Botanical name</th>
<th>Symbol</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Nocchi</td>
<td>Vitex negundo</td>
<td>Vn</td>
</tr>
<tr>
<td>2</td>
<td>Amla</td>
<td>Emblica officinalis</td>
<td>Eo</td>
</tr>
<tr>
<td>3</td>
<td>Aloevera</td>
<td>Aloe barbadensis</td>
<td>Ab</td>
</tr>
<tr>
<td>4</td>
<td>Neem</td>
<td>Azadirachta indica</td>
<td>Ai</td>
</tr>
</tbody>
</table>

3.3 COMBINATION OF NATURAL SOURCES

The above given chart shows the number of natural dyed samples obtained from the combination of supporting natural dyes (If, Rc and Pp) and (Vn, Eo, Ab and Ai) to the major dyes Bv, Bo, Tc, Pg, Al and Cp to impart shade variation and functional property respectively. The three main stages
show the application of natural dyes and its combination on textile fabrics for different variations. The number of dyed sample got in the first stage is one for each natural dye. The second stage shows the combination of natural dyes applied to achieve shade variation and the number of dyed samples obtained is six. In third stage natural dyed samples are obtained by combining the natural dye sources for shade variation as well as to impart functional property. For each major natural dye thirty five natural dyed samples were obtained separately for cotton, silk and wool fabrics as shown in Figure 3.2.

![Figure 3.2 Flow chart of combination of natural sources](image_url)
3.4 EXTRACTION OF NATURAL DYE SOURCES

Natural dye solutions of major dye ingredients berberis vulgaris (Bv), bixaorellana (Bo), terminilia chebula (Tc), punica granatum (Pg), allium cepa (Al) and citrus paradisi (Cp); and supporting dyes indigofera tinctoria, rubia cardifolia, pterocarpus santalinus; and the value added natural sources such as vitex negundo, emblica officinalis, aloe barbadensis and azadirachta indica were extracted by aqueous method as per the established technique. All the natural dyes were extracted with varying amount of the dye material (10-15% owm) in different time intervals (20-60 min).

3.5 APPLICATION METHOD OF NATURAL DYES

This division describes the application method of various dyes applied to cotton, silk and wool fabrics for shade variation and functional property. Sodium chloride, sodium carbonate and potash alum were used as dyeing assistants. The maximum usage condition of these dye assistants were set as 2.5% (owm) for good dyeing. The application condition is different for each and every dye applications on textile fabrics as discussed below.

3.5.1 Berberis Vulgaris and its Combination on Cotton, Silk and Wool Fabrics

Sodium chloride, sodium carbonate and potash alum are used in natural dyeing with Vn combination and the concentration of these were 1%, 0.5% and 1.5% for cotton and wool, 1%, 0.5% and 1% for silk. Sodium chloride and potash alum of 0.5% and 1.5% for cotton and wool, 0.5% and 1% for silk were used for the combination of dyeing with Eo, Ab and Ai. The first level of combination required only potash alum of 0.5% for all the three fabrics. The temperature was maintained at 60° for berberis vulgaris dye alone, and for the combination dyeing the temperature was varied to obtain
the best dyeing result. The duration of the natural dyeing was 30 minutes for berberis vulgaris dye alone whereas the duration increases upto 90 minutes there afterwards based on the combination for cotton, silk and wool fabrics.

3.5.2 Bixa Orellana and its Combination on Cotton, Silk and Wool Fabrics

Dyeing assistants sodium chloride, sodium carbonate and potash alum were used in natural dyeing with Eo, Ab and Ai combination and the concentration of these were 0.5%, 1.5% and 0.5% respectively for cotton, silk and wool fabrics. Sodium chloride and sodium carbonate in 0.5% and 2% for cotton and wool fabrics and sodium chloride and potash alum with concentration of 0.5% and 2% were used for silk fabric dyeing. The first level of combinations required only sodium carbonate of 1.5% to give best result. The temperature maintained was 60° for bixa orellana dye alone, and for the combination dyeing the temperature was varied for good dyeing. The duration of the natural dyeing is 30 minutes for bixa orellana dye alone and the duration increased there afterwards upto 90 minutes based on the combination results needed.

3.5.3 Terminalia Chebula and its Combination on Cotton, Silk and Wool Fabrics

Dye assistants sodium chloride, sodium carbonate and potash alum with 2%, 0.5% and 0.5% are used in natural dyeing with Vn combination and dye assistants sodium chloride and potash alum was used for the dye combinations with Eo, Ab and Ai with concentrations 1% and 0.5% respectively for cotton, silk and wool fabrics. The first level of combinations with If, Rc and Pp required the dye assistants sodium chloride and potash alum with 1% and 0.5% respectively for natural textiles to obtain good results. The temperature was maintained at 60° for berberis vulgaris dye
alone, and for the combination dyeing the temperature was varied for good dyeing. The duration of the natural dyeing is 30 minutes for the berberis vulgaris dye alone whereas the duration increases there afterwards upto 90 minutes based on the combination results needed.

3.5.4 Punica Granatum and its Combination on Cotton, Silk and Wool Fabrics

Sodium chloride, sodium carbonate and potash alum are used in natural dyeing with Vn in concentrations 1%, 1% and 0.5% for cotton and 1%, 0.5% and 0.5% for silk and wool fabrics. Combination of dyeing with Eo, Ab and Ai required 0.5% of sodium chloride and potash alum separately for cotton, silk and wool fabrics. The first level of dyeing combinations with If, Rc and Pp required 0.5% of sodium chloride and sodium carbonate. Silk and wool fabrics required only sodium chloride of 0.5% for good results. The temperature was maintained at 60° for berberis vulgaris dye alone, and for the combination dyeing the temperature was varied for good dyeing. The duration of the natural dyeing is 30 minutes for the punica granatum dye alone whereas the duration increased there afterwards upto 90 minutes based on the combination results needed.

3.5.5 Allium Cepa and its Combination on Cotton, Silk and Wool Fabrics

Sodium chloride, sodium carbonate and potash alum are used in natural dyeing with Vn combination with concentration 1.5%, 0.5% and 0.5% for all cotton, silk and wool fabrics and sodium chloride and potash alum with 1% and 0.5% for cotton and 0.5% of the both were used for silk and wool for the first level of combinational dyeing with If, Rc and Pp. Combination of Al with Eo, Ab and Ai for cotton required 1% and 0.5% of sodium chloride and potash alum and 0.5% of both the dye assistants for silk and wool for good
results. The temperature was maintained at 60° for Allium cepa dye alone, and for the combination dyeing the temperature was varied for good dyeing. The duration of the natural dyeing is 30 minutes for the allium cepa dye alone whereas the duration increases there afterwards upto 90 minutes based on the combination results needed.

3.5.6 Citrus Paradisi and its Combination on Cotton, Silk and Wool Fabrics

Sodium chloride, sodium carbonate and potash alum were used in concentrations 1%, 0.5% and 0.5% in natural dyeing with Vn combination for cotton, silk and wool. The first level of combinational dyeing with If, Rc and Pp required 0.5% of sodium chloride and potash alum separately for good results. Combination of dyeing with Eo, Ab and Ai required 0.5% of sodium chloride and potash alum separately. The temperature was maintained at 60° for Cp dye alone, and for the combination dyeing the temperature was varied for good dyeing. The duration of the natural dyeing is 30 minutes for the berberis vulgaris dye alone whereas the duration increases there afterwards upto 90 minutes based on the combination results needed.

3.6 EVALUATION OF NATURAL DYES AND NATURAL DYED FABRICS

3.6.1 FTIR Analysis of the Natural Dye Extract

FTIR 8400’s Shimadzu spectrophotometer, one of the most commonly used spectroscopic technique was used to analyze the chemical groups present in the natural dyes. It identifies the chemical bonds in a molecule by producing an infrared absorption spectrum (Y.R Sharma 2006). During FTIR analysis, a spot on the specimen is subjected to a modulated IR beam and the resulting FTIR spectral pattern was then analyzed with the peaks obtained.
3.6.2 Color Fastness Analysis of the Natural Dyed Textile Fabrics

The natural dyed samples were washed under condition IIIA of AATCC Test Method 124-2001 (2003) to determine the color change effect of natural dyed fabrics. Light fastness tests were carried out according to AATCC Test Method 16 E-1998 (2003). The samples were exposed to 5, 10 AFUs (AATCC Fading Unit) to determine the color change AATCC 16-1998 (2003). AATCC standardized crock meter was used to determine the rubbing fastness of natural dyed fabrics under wet and dry condition to assess the color change and staining property AATCC 61-1996 (2003).

3.6.3 Antimicrobial Assessment of the Natural Dyed Textile Fabrics

AATCC Test Method 100-1999 (2003) was used to determine the antimicrobial activity of the dyed samples (cotton, silk and wool). Escherichia coli (E.coli), a gram-negative bacterium, was selected due to its popularity of being selected as a test organism and its resistance to common antimicrobial agents. Staphylococcus aureus (S.aureus), a pathogenic gram-positive bacterium, was used because it was the major cause of cross-infection in hospitals and it is the most frequently evaluated species. The gram negative E. coli and the gram-positive S. aureus cultures were left to grow in specific conditions and used to test the antimicrobial effect of all the dyed and undyed samples. The dilution medium was nutrient broth and the neutralizer was sodium hydroxide. To evaluate the antimicrobial activity the numbers of reduction colonies were compared between the dyed and undyed samples after incubation. The results are expressed as percent reduction of bacteria by \[ R (%) = 100(A-B)/A. \] Where A and B are the numbers of bacteria recovered from the undyed and dyed samples after inoculation and incubation.
3.6.4 K/S Analysis of the Natural Dyed Textile Fabrics

Colorimetric data of natural dyed textile fabrics were determined using a Datacolor SF 600 plus spectrophotometer interfaced to a PC. Measurements were taken regarding colour presence, brightness, dullness and colour intensity with the specular component of the light excluded and the UV component included using illuminant D65 and 10° standard observer. Each fabric was folded once so as to give two thickness and average of five readings were taken each time AATCC 135-1985 (2003).