CHAPTER – 3

METHODOLOGY

Considering the modern trend in favour of a switch over to use natural
dyestuff, it is a high time to exploit natural resources of plant material for dyeing and
printing.

A combination of mordants and plant materials can go long way to serve as an
effective substitute for synthetic dyestuff. The health hazard posed by using synthetic
dyes and chemicals is great cause of concern.

The ban imposed by European countries on using azo dyes and non eco-
friendly chemicals has forced the researcher to take this opportunity.

Vegetable dyes are eco-friendly in nature and more popular among various
groups of people. There is heavy demand for natural dyes and growing popular
interest in metropolitan cities in India and abroad.

Taking this into account the investigator has selected the popular topic entitled
Dyeing and Printing of cotton and silk with selected Natural dyes.

Considering the importance, and objectives the experimental procedure was
designed.

3.1 Experimental Materials

3.1.1. FABRICS

Sources of natural fibres are wide ranging, but fall into two main categories:
Cellulosic fibres and protein fibres. Cotton as a cellulosic fibre and tussar silk as a
protein fibre were selected for the present study.

3.1.1.1 Cotton. Cotton is the most widely used of the textile fibres. It has a
combination of properties- durability, low cost, easy washability and it makes very
comfortable skin contact fabrics because of its absorbency and its good heat and
electrical conductivity. This unique combination of properties has made cotton a standard for great masses of the world’s people.

3.1.1.2 Tussar silk. It is a product of wild silk worms that are native to India and China. Silk is universally accepted as a luxury fibre. The International Silk Association of the United State emphasizes the uniqueness of silk by its slogan “Only silk is a silk”. Silk has a unique combination of properties not possessed by any other fibre: ‘Dry’ tactile hand, natural lustre, good moisture absorption, lively suppleness and draping qualities and high strength.

100% grey cotton and natural tussar silk being natural fibres are biodegradable in nature and hence preferred safe route of using natural dyes on natural fibres.

The fabrics selected for the present study were tested for understanding the various physical parameters so that the effect of the dye can be generated for the selected fabrics.

The experimental fabrics 100% cotton and tussar silk were tested according to standard procedures laid by ISO. (Booth 1982). The results obtained are given in the following table.

<table>
<thead>
<tr>
<th>Fabrics</th>
<th>Wt/sq m. (GSM)</th>
<th>Ends/ Inch</th>
<th>Picks/ Inch</th>
<th>Warp Count</th>
<th>Weft Count</th>
<th>Weave</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Cotton</td>
<td>60</td>
<td>72</td>
<td>64</td>
<td>80 Ne</td>
<td>80 Ne</td>
<td>Plain</td>
<td>Natural Creamy</td>
</tr>
<tr>
<td>2 Tussar Silk</td>
<td>45</td>
<td>72</td>
<td>96</td>
<td>60 Denier</td>
<td>50 Denier</td>
<td>Plain</td>
<td>Tussar Gold Natural</td>
</tr>
</tbody>
</table>

3.1.2. DYE SOURCES

Dye sources were finalized after the exploratory survey.

- *Carissa Carandas* Linn (Karvanda fruits)
- *Holarrhena antidysenterica* Linn (Kuda leaves)
- *Tagetes erecta* Linn (Marigold flowerheads)
3.1.3 MORDANTS

3.1.3.1 Natural mordants
- Harda fruits (*Terminalia chebula* Linn)
- Pomegranate rind (*Punica granatum* Linn)
- Babool bark (*Acacia arabica* Linn)

3.1.3.2 Metal mordants
- Alum
- Tin (Stannous chloride)
- Iron (Ferrous sulphate) \( \frac{1}{2} \) Lb. Gd. Of Qualigens.

3.1.3.3 Chemicals and Auxiliaries
- Sodium carbonate (Na\(_2\)CO\(_3\))
- Acetic acid (CH\(_3\)COOH)
- Nonionic detergent
- Sodium hydroxide flakes

All these chemicals were used (as and when) in the form received from the market.

3.2 Experimental Methods

3.2.1 PREPARATION OF FABRIC FOR DYEING
With known specification, selected fabrics were prepared for the dyeing and printing for further work.

In order to make the fabric absorbent, to obtain level dyeing and penetration of dyestuffs scouring of cotton fabric and degumming of silk fabric was done. Standard procedures were adopted for scouring and degumming.

3.2.1.1 Scouring of cotton fabric. The grey cotton material was scoured in a bath containing 2% caustic soda (NaOH), 1% T.R.O. and 1 percent soda ash (Na\(_2\)CO\(_3\)) for 3 to 4 hours at boiling temperature keeping M:L as 1:20. After processing it was washed thoroughly in hot water and then in cold water. The scoured fabric was dried in air for 48 hours.
3.2.1.2 Degumming of silk fabric. Silk fabric contains 25 to 30 percent sericin gum (o.w.m.). To remove this gum, silk fabric was dipped in 25% aqueous solution of non-ionic detergent. The solution was heated up to 80±1°C for about 60 minutes. Residual detergent was removed thoroughly by washing the treated fabric with water, initially by hot water and finally with cold water.

3.2.2 EXPLORATORY SURVEY AND COLLECTION OF DYE SOURCES

In India vegetation is in abundance and due to variation of climatic conditions in different parts of country, variety of vegetation is available. There are trees/Shrubs, which do give excellent dye sources. It was observed that the variety of trees/plants species, available in Melghat forest of Vidarbha region, have not been explored as a dye source, though these trees are abundantly available in forest. Exhaustive screening of literature revealed that, *Wrightia tinctoria* gives deep blue colour on cotton. A small attempt on extraction of dye from *Carissa congesta* fruits has also been noted. Both these plants are the members of Apocynaceae family.

It was a suggestion of taxonomist that other members of the family Apocynaceae can be tried for the extraction of dye. During the survey of Melghat forest it was observed that *Holarrhena antidysentrica*, which is very close to Wrightia tinctoria in morphological features, is abundantly available in forest region. *Holarrhena antidysentrica* is also a member of of Apocynaceae with different genus and species.

Another member of Apocynaceae i.e. *Carissa carandas* is also abundantly available in forest as well as plains of the region. *Carissa carandas* is the plant of same genus with different species from the *Carissa congesta* noted earlier. *Carissa carandas* Linn (plate-1) and *Holarrhena antidysentrica* Linn (plate-2) are the phytochemically rich plants which have not been tapped for the extraction of dyes, were selected for the present study. However, identification of plant is not an easy job as morphological...
features may get confused even though they are from different genus and species. Hence, the survey and identification of plants was done with the help of taxonomist. Collection of the *Holarrhena antidysentrica* leaves and matured fruits of *Carissa carandas* was arranged from forest. The other phytochemically rich plant *Tagetes erecta* (Marigold)(plate-3) is the member of family Asteraceae and most commonly used as a dye source, which was also selected for the study. The Marigold flower heads were collected from the nearby gardens.

3.2.3 PREPARATION OF DYE EXTRACTION

The vegetable products were washed thoroughly to remove dirt particles, fungus and insecticides (if seen). From the collected fruits, leaves and flowers a section of material was kept for shade drying.

The fresh and dried vegetable sources were then used for extraction. The Common methods available for extraction of dyes are-

a) Solvent Extraction b) Solution Extraction but techniques such as supercritical fluid extraction are also in vogue.

In the present study the most economical extraction method i.e. aqueous extraction method was used.

3.2.3.1 Aqueous extraction. The plant material was cut to small size. These were then added into distilled water. The plant material containing water was boiled for different periods. After specific time of boiling the coloured solution was separated by filtration. Care was taken not to pass fine particles in the filtrate.

Finally three concentrations were tried out. The dye material to water ratio taken for extraction kept constant as 1:50. The quantity of dye material taken for extraction was dependent on the depth of colour of treated fabric as obtained in the pilot study.

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This experiment was common for all the three sources used for dye extraction. The quantity of water added to the plant material was decided by intensity of coloured solution obtained.

3.3 Pilot Study (Pre-Testing)

Pilot study was carried out to optimise the dyeing parameters separately by each dye source.

3.3.1 PREPARATION OF DYE EXTRACT FOR SELECTED DYES

3.3.1.1 Aqueous extraction of Carissa carandas fruits. The carandas fruits were crushed and seeds were removed. Water was then added to it and boiled.

The material to water ratio was maintained to 1:50 throughout the extraction. Extraction was continued to get optimum colour in the solution. The solution was filtered to remove unwanted residue. Care was taken not to pass any fine particle in the filtrate.

3.3.1.2 Aqueous extraction of Holarrhena antidysenterica leaves. The Holarrhena antidysenterica leaves were cut into fine pieces, mixed with water and boiled. The material to liquor ratio was maintained to 1:50 throughout. Extraction was carried out to get optimum colour of the solution. Solution was filtered to get clear extract.

3.3.1.3 Aqueous extraction of Marigold flower petals. Extract of Marigold flower petals was obtained by boiling petals in the water. The material to liquor ratio was maintained to 1:50 during the complete extraction process. Extraction was carried out to get maximum colour in the solution. The solution was then filtered to get clear extract.

3.3.2 PRE-TREATMENT WITH ALUM

Test specimen of 1gm from scoured cotton and degummed tussar silk were taken for the pre-testing. Previous studies reveals that mordants are considered as an integral part of the natural dyeing procedures. It has also been proposed that alum and aluminium sulphate should be used as mordants in dyeing with natural dyes, as their
environmental toxicity is low. Therefore alum was selected as a sole mordant for the present study. To carry out the dyeing as pre-testing; pre-treatment with 10 percent alum (as a sole mordant) was carried out for the scoured cotton samples and degummed silk samples for 45 minutes at 90°C to increase an affinity of the fibre towards natural dye.

Both fresh and dried dye materials were tried. Each dye selected was optimised for dye material concentration, extraction time and dyeing pH using fresh and dried dye materials.

3.3.3. OPTIMISATION OF DYEING PARAMETERS FOR CARISSA CARANDAS

3.3.3.1 Optimisation of dye material concentration. Dye solutions of 20%, 40% and 60% dye material conc. (owf) were prepared by boiling keeping the M:L as 1:50. Maximum dye yield was observed after 30 minutes at 90°C with 60% dye material. Decrease in colour intensity was observed with 40% and 20% concentration.

3.3.3.2 Optimisation of extraction time. Three dye concentrations i.e. 20, 40 and 60% (owf) were used keeping the M:L as 1:50 for each. Optimisation was done at boiling temperature. The test time for extraction was determined as 30, 60 and 90 minutes. The optimum dye extraction was observed for 60% conc. (owf) at 90°C for 30 minutes extraction time. The minimum colour extraction was observed at 15 minutes time with 20% concentration.

3.3.3.3 Optimisation of pH of dye solution. Dye solution was prepared with 60% dye material (owf) keeping M: L as 1:50. Extraction was carried for 30 minutes at 90°C. The pH of the dye extract was tested. It was found to be 4. The pH of dye solution was adjusted to 5, 6, 7, 8, and 9 with 1% solution of sodium carbonate. Dyeing was carried out with extracts having original pH as well as adjusted pH values.

Test specimens were subjected for dyeing, pre-mordanted with 10% Alum (owf) at 90°C for 45 minutes. The solutions were allowed to cool for 15 minutes at
room temperature. Removed the dyed specimens; squeezed gently and rinsed thoroughly and shade dried.

Dyeing was carried out for cotton as well as silk.

For the above parameters both fresh as well as dried fruits were experimented and the dyed samples were evaluated visually.

It was observed that during and after the extraction of dried fruits the intensity and the depth of colour were much less for the same trial proportions. Therefore only fresh fruits were recommended for further experiment.

Table- 3.2 Optimum Conditions for Dyeing with Fresh Carissa carandas Fruits

<table>
<thead>
<tr>
<th>Variables</th>
<th>Trial Proportions</th>
<th>Optimum Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1  Dye material concentration</td>
<td>20, 40, 60 % (owf)</td>
<td>60% (owf)</td>
</tr>
<tr>
<td>2  Dye extraction time</td>
<td>30, 60, 90 minutes</td>
<td>30 minutes</td>
</tr>
<tr>
<td>3  PH of dye solution</td>
<td>4, 5, 6, 7, 8, 9</td>
<td>4 (Not adjusted)</td>
</tr>
</tbody>
</table>

3.3.4 OPTIMISATION OF DYEING PARAMETERS FOR HOLARRHENA ANTIDYSENTRICA
Since leaf material is available almost throughout the year, leaves were collected in September and in February (just before leaf fall). Both fresh and dried leaves were experimented.

3.3.4.1 Optimisation of dye material concentration. Dye solutions were prepared by boiling with 20%, 40% and 60% dye material (owf) keeping M: L as 1:50. Observations were made after every 15 minutes. No remarkable changes in dye extract of both fresh and dry leaves were marked up to 60 minutes. After 60 minutes maximum dye yield with reddish brown colour of extract in case of fresh leaves were found at 90°C with 40% dye material concentration, whereas it was yellow in terms of dried leaves. The solutions were decanted and used for dyeing.

3.3.4.2 Optimisation of extraction time. The dye material conc. i.e. 20, 40, and 60% (owf) were used to prepare the dye solutions keeping the M:L as 1:50 for each concentration. Optimisation was done at boiling temperature. The test time for extraction was determined as 60, 90 and 120 minutes.
3.3.4.3 Optimisation of pH of dye solution. Dye solution was prepared using 40% dye material (owf) keeping M: L as 1:50. Extraction was carried out for 90 minutes at 90\(^{0}\)C. Solution was decanted and the pH of the dye was found to be 7.

Dye solutions were adjusted for pH 4, 5, 6, 8 and 9 by adding 0.1 N acetic acid and 1% solution of sodium carbonate for acidic and alkaline pH respectively. Dyeing was carried out with extracts having original as well as adjusted pH values.

The samples pre-treated with 10% Alum (Control) were added to each of the prepared dye solutions initially at 50\(^{0}\)C and temperature was raised up to 90\(^{0}\)C and dyeing was carried out for 45 minutes. The dye solutions were allowed to cool for 15 minutes at room temperature. The samples were removed from the dye solutions squeezed gently, rinsed and dried.

The procedure was followed for dyeing of both Cotton and Silk fabric and the samples were evaluated visually.

The following observations were made during and after dyeing.

**Dye material concentration.**
- Colour depth in terms of fresh leaves was found to be almost same when dyed with 40% and 60% dye conc.
- Pinkish rust colour was produced when dyed with extract of fresh leaves for both cotton and silk.
- Pale yellow colour was produced on both cotton and silk when dyed with extract of dried leaves with the same concentration.

**Extraction time.**
- The dye concentrations increased with increase in the duration of extraction at high temperature.
- The shades of the samples dyed in extract of 90 minutes and 120 minutes produced were similar. As increased time would add the cost on fuel; a period of 90 minutes was taken as optimum.
- Results were same for both cotton and silk fabric.
- Best results were obtained with fresh leaves.

**pH of the dye solution.**
• In terms of cotton samples dyed with extract of fresh leaves it was investigated that there was an increase in depth of red colour with increase in pH i.e. it was optimum at pH 9. Where as cotton samples dyed with extract of dried leaves produced yellowish brown colour at pH 9. Muddy yellow colours were produced with lowering of pH.

• In terms of silk samples dyed in extract of fresh leaves with pH 6 and 7 produced similar shades of optimum red brown colour. Silk samples dyed with extract of dried leaves were lacking in reddish colour.

Table – 3.3 Optimum Conditions for Dyeing with Fresh H. antidysentrica Leaves

<table>
<thead>
<tr>
<th>Variables</th>
<th>Trial proportions</th>
<th>Optimum conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Dye material concentration</td>
<td>20, 40, 60% (owf)</td>
<td>40% (owf)</td>
</tr>
<tr>
<td>2 Dye extraction time</td>
<td>60,90,120 min.</td>
<td>90 minutes</td>
</tr>
<tr>
<td>3 PH of dye solution</td>
<td>4, 5, 6, 7, 8, 9</td>
<td>9 (Cotton) 7 (Silk)</td>
</tr>
</tbody>
</table>

3.3.5 OPTIMISATION OF DYEING PARAMETERS FOR TAGETES ERECTA LINN

To optimise the results both fresh and dried flower heads were tried.

3.3.5.1 Optimisation of dye material concentration. Dye solutions of 20, 40 and 60% dye material concentration (owf) were prepared by boiling keeping the M: L as 1:50.

3.3.5.2 Optimisation of extraction time. Three dye concentrations i.e. 20, 40 and 60% (owf) were used keeping the M:L as 1:50 for each concentration. Optimisation was done at boiling temperature.

The test time for extractions were determined as 30, 60 and 90 minutes.

3.3.5.3 Optimisation of pH of dye solution. Dye solution was prepared with 60% concentration (owf) keeping the M: L as 1:50. Extraction was carried for 60 minutes at 90°C. The pH of dye bath was tested and it was found to be 7. The pH of dye solution was adjusted to 4, 5 and 6 with dilute acetic acid 0.1 N where as 8 and 9 with 1% solution of sodium carbonate. Dyeing was carried out with extracts having original pH as well as adjusted pH values.
Test specimens were subjected for dyeing, pre-mordanted with 10% Alum (controlled) (owf) at 90°C for 45 minutes. The solutions were allowed to cool for 15 minutes at room temperature.

Dyed specimens were removed, squeezed gently and rinsed thoroughly and dried.

Dyeing was carried out for cotton as well as silk and the samples were evaluated visually.

The following observations were made during and after dyeing.

- **Dye material concentration.**
- Optimum dye yield was observed with 60% dye material concentration with decreased colour intensity for 40% and 20%.
- No significant difference was found in samples dyed with fresh and dried flower heads regarding depth of colour.
- **Extraction time.**
- 60 minutes was the optimum time for the extraction of dry flower heads.
- Yellow ochre shade was obtained when dyed with fresh and dry flower heads on cotton samples. Where as brighter yellow colour was produced on silk samples.
- **pH of the dye solution.**

The colour imparted was almost same for samples dyed with pH 6 and 7. pH 6 shows slightly brighter effect. To make the procedure more eco-friendly pH 7 was selected for the further experiment.

Table – 3.4 Optimum Conditions for Dyeing with Dried Marigold Flower Heads

<table>
<thead>
<tr>
<th>Variables</th>
<th>Trial proportions</th>
<th>Optimum conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Dye material concentration</td>
<td>20, 40, 60% (owf)</td>
<td>60% (owf)</td>
</tr>
<tr>
<td>2 Dye extraction time</td>
<td>30, 60, 90 minutes</td>
<td>60 minutes</td>
</tr>
<tr>
<td>3 PH of dye solution</td>
<td>4, 5, 6, 7, 8, 9</td>
<td>7</td>
</tr>
</tbody>
</table>
Fabrics:  
- C - Cotton  
- S - Silk  

Dye Sources:  
- C - Carissa carandas  
- H - Holarrhena antidysenterica  
- M - Tagetes erecta (Marigold)  

Mordants:  
- A - Alum  
- T - Tin  
- I - Iron  
- B - Babol bark  
- R - Pomegranate Rind  

Mordant Concentration:  
- 0 - (10:0)  
- 1 - (9:1)  
- 2 - (7:1)  
- 3 - (5:5)  

* NT - Control group - (No Tannin treatment)  
* TT - Experimental group - (Pretreatment with Tannin)  
* The first part of the ratio is Alum mordant and the second part is other mordant in the combination.

FIG-3.1 SCHEMATIC REPRESENTATION OF DYEING FOR CONTROL AND EXPERIMENTAL GROUP
The test samples were divided into control (i.e. no pre-treatment given with harda tannin) and experimental group. Dyeing was carried out for all the samples of control and experimental group with *Carissa carandas*, *Holarrhena antidysenterica* and *Tagetes erecta* pre-mordanted with alum as a single mordant and alum with two eco-friendly metal mordants i.e. Tin and Iron and Pomegranet rind, Babool bark as natural mordants.

Exhaust method of dyeing was used. Treatments given prior to dyeing were harda (tannin) treatment and premordanting.

In the present work an attempt has been made to use the most effective mordant alum as a major constituent. Other two metal mordants tin and iron which are not red listed according to eco standards and pomegranate rind, babool bark which are considered as a natural mordants were used. The sources of natural vegetable tannin were used along with alum with different ratios as binary mordant combination.

### 3.4.1 TANNIN TREATMENT (HARDA TREATMENT)

Test samples of experimental group were treated with harda powder. 10 percent harda powder (owf) was soaked overnight in a steel vessel at 1:20 M:L ratio. Next day the solution was filtered. Care was taken not to allow the fine particles of myrabolan (harda) in the filtrate. The test sample was put in this solution and was treated for 30 minutes at room temperature with constant handling of fabric. Samples were removed, padded and dried without washing for twenty-four hours. The treatment was carried out separately for each test sample of cotton and silk. Total 26 samples i.e. (13 of cotton and 13 of silk) were treated for each dye.

### 3.4.2 MORDANTING

Two eco-friendly metal mordants such as tin and iron and two natural vegetable mordants, pomegranate rind and babool bark were used in combination with alum to prepare (Alum + tin), (Alum + Iron), (Alum + Pomegranate rind) and (Alum + Babool bark) binary combinations. Taking alum as a single mordant and in binary
combination with other mordants the ratio of mordant was varied from (10:0) (9:1) (7:3) and (5:5) for each combination.

The mordanting bath was set for 10 percent mordant concentration on weight of the fabric keeping the material to liquor ratio at 1:50. Separate mordanting baths were prepared for cotton as well as for silk samples. The scoured cotton and degummed silk fabric samples were allowed for wetting out for 5 minutes in water and then were introduced into two separate mordanting bath initially at 50°C and treated for 45 minutes with adequate fibre liquor movement. The temperature was raised up to 90°C during mordanting time. The dye bath was allowed for cooling for 15 minutes. Fabric samples were then taken out of the mordanting bath, squeezed gently and then put immediately into the dye bath that was previously set at 50°C.

Mordanting was carried out for all the samples of control and experimental group. Samples were mordanted with alum as a single mordant and with each binary mordant combination for all the three rations. Total 52 samples i.e. (26 of cotton and 26 of silk) were mordanted for each dye.

3.4.3 DYEING CONDITIONS

Optimum conditions for dyeing were determined by visual analysis of the samples, in the pilot study.

Taking the optimised conditions into consideration dye extracts of Carissa carandas fruits, Holarrhena antidysenterica leaves and Tagetes erecta flower petals were prepared.

3.4.3.1 Preparation of dye extract for Carissa carandas. The dye extract was prepared with 60% dye material concentration (owf) keeping M: L as 1:50. Extraction was carried out for 30 minutes at 90°C; maintaining the level of solution in the container throughout. The dye extract was allowed to cool at room temperature. The extracted solution was then strained through a nylon filter to remove the residual part and made the dye solution clear. Care was taken not to allow fine particles of dye material in the filtration process. The solution was then transferred to open bath for
exhaust dyeing. The pH of the dye solution was scanned and it was found to be acidic i.e. 4. pH was kept as original and prepared the dye-bath for dyeing.

3.4.3.2 Preparation of dye extract for Holarrhena antidysentrica. The dye extract was prepared with 40% dye material concentration (owf) keeping the M:L as 1:50.

Extraction was carried out for 90 minutes at 90°C; maintaining the level of solution in the container same. The dye extract was allowed to cool at room temperature. The solution was then decanted to remove the unwanted residue. The decanted solution was then transferred to dye bath for dyeing. The pH of the dye solution was scanned and it was found to be 7. The pH of the dye solution was kept 7 as original pH for dyeing of silk where as it was adjusted to 9 by adding 1 percent solution of sodium carbonate for dyeing of cotton fabric and the dye baths were kept ready for dyeing.

3.4.3.3 Preparation of dye extract for Tagetes erecta (Marigold flower heads). Dried flower petals were taken for the extraction. Extraction of the dye was carried out for 60 minutes with 60 percent dye material concentration at 90°C keeping the M:L as 1:50. The extract was then filtered through a nylon filter to make the dye solution clear. The solution was then transferred to dye bath for dyeing. The pH of the dye bath was tested and it was found to be 7. pH was kept original as neutral for dyeing of cotton and silk samples.

3.4.4 DYEING PROCEDURE

Simultaneous dyeing of cotton and silk sample was carried out into separate dye baths by exhaust dyeing method. Dyeing was carried out for 45 minutes with adequate movement of dye liquor. Initially the temperature of the dye bath was 50 ±1°C then it was raised up to 90±1°C during dyeing time. The dye bath was allowed to cool at room temperature for 15 minutes. The dyed samples were removed squeezed gently and rinsed thoroughly in cold water.

The procedure was repeated for the samples of control and experimental group. Total 156 samples were dyed.
3.4.4.1 Soaping. Soaping of the dyed samples was done with non-ionic detergent (0.5gpl) at 60°C for 20 minutes.

3.4.4.2 Rinsing. Samples were rinsed thoroughly and dried in the shade.

3.5 Evaluation of Colour Values

Colour values of the samples were analysed on the basis of L* a* b* c* h* values using reflectance spectra through data colour international spectrophotometer.

3.6 Assessment of Fastness Properties

Coloured fabric is required to maintain its colour that is to possess an acceptable amount of colourfastness. There are various treatments that fabric may be subjected to during further processing and use, which may affect the colourfastness. These treatments are known as agencies.

In the present study colourfastness of dyed samples was assessed for four agencies. Viz., colourfastness to washing, perspiration, rubbing (crocking) and sunlight

3.6.1 COLOURFASTNESS TO WASHING (ISO2)

The composite specimen was treated in a solution containing 5g/l soap at 50°C for 45 minutes in Launder-O-meter at material to liquor ratio 1:50.

Then the samples were rinsed with water to make the samples free from soap and shade dried. The change in colour of the test specimen and the staining of the adjacent fabrics were assessed with the help of Grey scale.

3.6.2 FASTNESS TO PERSPIRATION (IS: 971-1983)

Composite specimen is prepared by placing the test specimen (5x5) cms between the two adjacent fabrics. The samples were soaked in the test liquor (acidity and alkaline) with M: L 1:50 for 30 minutes at room temperature. The treated samples were kept between two glass plates of perspirometer under force of 5 kg. The apparatus was kept in hot air oven for four hours at 37±2°C. Test samples were removed from the oven and were air dried at temperature not exceeding 60°C. The
numerical ratings for change in colour of the test piece and staining of the two adjacent pieces were done using Grey scale.

3.6.3 COLOUR FASTNESS TO RUBBING (IS : 766-1988)

The undyed test specimen of size 5x5 cm wrapped to the finger of rubbing device of crock meter and wrapped test specimen of size 14x5 cm was fixed at the base of rubbing device. The specimens were rubbed to and fro against undyed piece with a downward force of 900gms in a straight line along a track of 10cms for 10 times in 10 seconds. The test samples were graded for the colour staining using Grey scale. The test was carried for both wet and dry rubbing.

3.6.4 COLOUR FASTNESS TO SUNLIGHT (IS : 686 –1985)

Test specimens of 1x6 cms were wound closely on a card and were mounted in the exposure rack. The rack was placed at an angle of 45°. The rack was exposed everyday from 9 am to 3.00 p.m. (6 hours per day) for eight days. The samples were evaluated for colour change after 48 hours of exposure using Grey scale.

3.6.5 EVALUATION OF THE TESTED SAMPLES WITH GEOMETRIC GREY SCALE AS PER ((ISO 105-102:1818, BS 1006 – 1021990, Std. Method)

The tested samples were assessed for colour change and colour staining.

3.7 Cost Estimations for Dyeing per Kg of Fabric

Dyeing cost per kg of silk and cotton was evaluated for optimum conditions.

3.8 Printing of Cotton and Silk with Selected Natural Dyes

The printing of cotton with vegetable dyes is practised in different parts of the country. The vegetable dyes that are being widely used by the traditional dyers/printers of the country are Alizarin, Indigo, Harda, Dhawri flower, Pomegranate rind and Haldi etc.

In the present study, efforts were made to assess the viability of printing cotton and silk fabrics with three sources of natural dyes using the traditional hand block printing technique.
Fabrics: -
  C - Cotton
  S - Silk
Dye Sources: -
  C - Carissa carandas
  H - Holarrhena antidysenterica
  M - Tagetes erecta (Marigold)
Mordants: -
  A - Alum
  T - Tin
  I - Iron
  P - Pomegranate Rind
  B - Babool bark
Mordant Concentration: -
  0 - (10:0)
  1 - (9:1)

* Tannin treatment was given prior to printing with mordants.
* The first part of the ratio is Alum mordant and the second part is other mordant in the combination.
3.8.1 PRINTING PROCEDURE

Five samples for each cotton and silk fabrics were printed with 10% alum and its combination with Tin, Iron, Pomegranate rind and Babool bark as eco-friendly mordants with only one ratio i.e. (9:1) was studied due to limitations.

3.8.1.1 Pre-treatment with Harda tannin. Previously prepared cotton and silk samples of required amount was treated with vegetable tannin (Harda). 20% Harda powder (owf) was taken and it was soaked in water for two hours at 1:20 M: L. It was then boiled for 1 hour. The liquor ratio was maintained during boiling. The solution was decanted and transferred into a cold-water bath. The well-prepared cotton and silk fabric samples were put into two separate baths and were treated with adequate liquor movement at room temperature for 30 minutes. Then the samples were taken out, padded and dried.

3.8.1.2 Preparation of printing paste.

Preparation of gum. 1 kg. of gum Arabic was put into 1 litre of water and was soaked for one day. Just before the printing, the gum was mixed thoroughly and sieved.

Preparation of Black colour. By taking jaggary and iron fillings with 2:1 ratio, black colour was prepared. The material was added to 10 litres of water in the earthen pot. The mixture was allowed to ferment for 20 days. The liquor obtained from the above procedure was added to gum Arabic in 1:3 (Liquor gum ratio) at the time of printing.

Preparation of printing paste with mordants. Printing paste was prepared for 10% alum as single mordants. 20gms of alum was taken and it was mixed with 100ml of hot water and boiled till the solution became half. To this pre soaked and filtered 180gms of gum Arabic was added and stirred thoroughly.

Preparation of printing paste with alum and its combination with other mordants. Alum 18gms and other mordant 2gms (9:1) such as tin was taken and added to 100ml hot water and boiled till the solution became half. To this, pre soaked and filtered 180gms of gum arabic was added and stirred thoroughly.
The printing pastes with other three mordants that are Iron, Pomegranate rind and Babool bark in combination with alum were prepared separately.

3.8.2 PRINTING WITH PREPARED MORDANT PASTES

- Block printing method was adopted for the study.
- Three different blocks were selected for three different dye sources.
- Printing was carried out for both cotton and silk fabrics. Out line of the design was printed first with black printing paste. The filling was done with prepared mordant pastes.
- The printed samples were dried under direct sunlight for a day.

3.8.2.1 Steaming. Each printed sample of cotton fabric was steamed for 1.5 hour and each printed samples of silk fabric was steamed for 1 hour.

3.8.2.2 Washing and rinsing. Washing of printed samples was carried out in running water thoroughly.

3.8.2.3 Dyeing. Dyeing of the printed samples was carried out separately with three selected dyes.

Same dyeing procedure with optimised dyeing conditions was adopted.

Printed and dyed samples were rinsed thoroughly and dried.

3.8.2.4 Soaping. Soaping of the printed samples was done with non-ionic detergent (0.5 gpl) at 60°C for 20 minutes.

3.8.2.5 Rinsing. Samples were rinsed thoroughly and dried in the shade.

3.9 Assessment of Fastness Properties

Appraisal of the performance of a dye begins at the time of its synthesis and ends with tests designed to indicate the level of performance during its use. Obviously, consumer goods must have satisfactory resistance to domestic cleaning treatments and a reasonable resistance to fading under the action of daylight and other wet treatments.
The tests are specifically required because the efforts were made to print with new dye sources to assess whether they withstand various conditions associated with the wide range of end uses.

Washing, rubbing, perspiration and sunlight of fastness properties of printed samples were evaluated according to standard methods laid down by-

Wash fastness was determined using ISO Test 2. On paramount Launder-0-meter (IS: 3361-1979)

- Rubbing fastness was determined using (IS: 766-1988)
- Fastness to perspiration was determined using (IS: 971-1983)
- Sunlight fastness was determined using (IS: 686-1985)
- The tested printed samples were assessed for colour change and colour staining with Geometric Grey Scale.

3.10 Cost Estimation of Printed Samples

Printing cost was evaluated by taking into consideration the dyeing grade chemicals and 50% more labour cost.