CHAPTER 3

MATERIALS

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METHODS
100% cotton fabric was used for studying the selected natural dyes. The amount of fabric required for the experimental procedure was calculated and purchased from the local market.

The experimental procedure used for the present study was as follows:

1. Materials used
2. Preparation of fabric
3. Dye extraction procedure
4. Dyeing of unmordanted fabric
5. Mordanting
6. Dyeing of fabric
7. Printing of fabric
8. Colour fastness test

Materials used

(A) Fabric:

(a) Fiber Content: THE fiber was insoluble in dil HCl, acetic acid and phenol. It showed swelling in 70% H$_2$SO$_4$, acetone and conc. HCl, while in conc. H$_2$SO$_4$ it dissolved completely. The
fabric was 100% cotton was confirmed by microscopic appearance and burning test. Under microscope the fiber showed broken lumen and on burning the fiber gave a golden yellow flame with a burning paper odour and gray paper ash.

(b) Determination of fabric count: The number of ends and picks per centimeter were determined using the Alfred Suter counter. Average of five readings was recorded as fabric count.

(c) Determination of weight per unit area of the fabric.

Five specimens of 5 cm X 5 cm were cut at random from the fabric. The specimen were conditioned over a saturated common salt solution at room temperature in a dessicator. Each specimen was weighed separately on a balance. An average of five readings was obtained and the weight per unit area in grams/square meter was calculated using the formula:

\[
\text{Weight in gms/sq.mt} = \frac{W \times 100 \times 100}{5 \times 5}
\]

Where "W" is the average weight in gms of the specimens.

(B) Dyes

Three dyes were used:

1. Majeetha
2. Turmeric
3. Onion peels
Maieetha (Rubia cordifolia): This is a root of the plant Rubia cordifolia. It is deep red in colour and is locally available in the market in dry form, because of its medicinal importance. The root was broken into small pieces and ground in a grinder to make a fine powder.

Turmeric (Curcuma longa): This is stem under the soil. According to the laymen it is also called root. It is yellow in colour. It is available in the local market. For the present study the dry form was purchased and powdered in the laboratory.

Onion peels (Allium cepa): This is a stem under the soil. The outer most dried skin of the onion was taken. Two varieties of onion are locally available the one with red peels and the other with white peels. The red variety was only taken for the study. The peels were dried in sunlight for 2 hours and then grounded into a powder form.

(C) Mordants:

The following six mordants were used.

1. Aluminium potassium sulphate salt
2. Aluminium acetate
3. Cupric sulphate
4. Lead Acetate
5. Stannous chloride
6. Tannic acid
Preparation of fabric:

The fabric was soaked in water over night for shrinkage. The fabric was then scoured in a soap solution containing 2 gms/liter of soap and 2 grams/litre of soda ash at 80°C for one hour. The material to liquor ratio was maintained to 1:30. After which the fabric was washed in running tap water, dried and ironed.

Dye Extraction Procedure:

In 100 ml 1 gm of dye was taken in a beaker. The beaker was kept for boiling in water bath for half an hour. Then the solution was filtered through filter paper, and the loss in water by evaporation was made up by adding the required amount of plain water.

Dyeing of unmordanted fabric:

A fabric sample of the required size was taken and weighed according to the weight of the fabric the required amount of dye solution was taken, so as to maintain 1:20 material to liquor ratio. Dyeing was carried out in a water bath at boiling temperature of 30 minutes. After which the sample was rinsed in plain water, dried and ironed.
Mordanting:

Use of natural dyes requires mordanting of fabric for improved fastness. For the study pre-mordanting was done for 2 different time periods:

- 30 minutes
- 8 hours (over night)

Mordanting and dyeing together was also studied.

For pre-mordanting of fabric 1 gm mordant was taken in a beaker to which 100ml water was added to prepare a stock solution. The fabric to be mordanted was weighed and from the stock solution the required amount of mordant was taken for 1:20 material to liquor ratio. The fabric sample was soaked in plain water, squeezed lightly and then soaked in the mordant solution for the required time, at room temperature with occasional stirring.

For mordanting and dyeing together, in the extracted dye solution, 1 gm of mordant was added. The fabric sample was weighed and the required stock solution was taken for 1:20 material to liquor ratio. Dyeing and mordanting together was carried out for 30 minutes in a water bath at boiling temperature with occasional stirring. The fabric sample was taken out rinsed in plain water, dried and ironed.
Dyeing of fabric:

The pre-mordanted fabric sample was taken and according to the weight of the fabric the required amount of dye solution was taken from the stock solution. The fabric from the mordant solution was squeezed and put in the dye solution. Dyeing was carried out in a water bath and boiled for 30 minutes. After which the sample was rinsed in plain water, dried and ironed.

Printing of fabric:

(a) Print paste formulation:

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Binder SNL</td>
<td>13 ml</td>
</tr>
<tr>
<td>CMC</td>
<td>300 mg</td>
</tr>
<tr>
<td>Diammonium hydrogen phosphate</td>
<td>1 gm</td>
</tr>
<tr>
<td>Dye solution</td>
<td>12 ml</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>25 ml</strong></td>
</tr>
</tbody>
</table>

In 25 ml of water, 1.250 gm of dye (5%) was boiled for half an hour in water bath. The extracted dye was filtered through filter paper. In a beaker 1 gm diammonium hydrogen phosphate, 300 mg of carboxy methyl cellulose sodium salt (CMC), 13 ml binder and 12 ml of dye solution was made into a fine paste by mixing with a glass rod.
Actual Printing with block:

A printing ink pad was made by spreading layers of squoered mulmul fabric in a petri dish on the mulmul fabric the print paste was spread. The fabric to be printed was stretched on a padded table. The block was placed lightly on the printing ink pad, lifted and placed on the fabric, little pressure was applied on the block so as to transfer the print past from the block on to the fabric. The printed fabric was allowed to dry flat and then cured dried in oven at 130°C for 15 minutes.

Colour fastness test:

(a) Washing: The dyed samples were tested for their wash fastness by AATCC Test method for colour fastness 61-1968. Test No.1 [A]. The samples (5 X 5 cm) were put in glass jar containing soap solution 5 gm/liter concentration. Mild washing was carried out for 45 minutes at room temperature with occasional stirring. After that samples were rinsed in cold water and ironed. The washed samples were compared with unwashed samples for their wash fastness. 3 samples for each dye were taken. Similarly the block printed samples were also tested for their colourfastness.
(b) **Ironing:**

1. **Dry Ironing fastness:**

Iron was set at ironing temperature for cotton. A white scoured fabric sample (5X5cm) was placed on the dyed fabric sample (5X5 cm). The sample was ironed by placing the hot iron for 30 seconds. The iron was removed and the white fabric sample was examined for transfer of dye from dyed fabric to white fabric. Three samples for each dye were taken.

2. **Wet Ironing fastness:**

The method followed was same as for testing dry ironing fastness except the dyed fabric sample of 5 X 5 cm was dipped in distilled water for few seconds then the fabric was removed and placed between two layers of blotting paper to remove excess water and the procedure followed was same as dry ironing test.

(c) **Crocking:**

Flat materials size was 5.1 X 12.7 cm and the dyed fabric was 20 cm X 1.3 cm.

(a) **Dry crocking test:**

The test specimen was placed on the base of the crockmeter so that it rested flat on the abrasive (20 cm X 13 cm) cloth with its long dimension in the direction of rubbing. A 5 X
5 cm square of white testing cloth was mounted with the weave oblique to the direction of rubbing down-wards from the weighed sliding arm. The covered finger was lowered on to the test specimen and caused it to slide back and forth twenty times by making ten complete turns of the crank at the rate of one turn/second. The white test square was removed for evaluation.

(b) **Wet Crocking Test:**

The white testing square was thoroughly wetted in distilled water. Then it was pressed between filter paper to remove excess water. The sample was mounted on the crockmeter and the procedure followed was same as for dry crocking test.