CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS

The materials, dyes, dyeing recipes used in different technologies are given below.

4.1.1 Cloth Materials Used

- Cotton
- Polyester
- Blend (67% cotton and 33% polyester)

4.1.2 Dyes Used

**Reactive Dyes (Cotton)**
- C.I. Reactive Blue 3 (Procion Brilliant Blue)
- C.I. Reactive Red 29 (Procion Brilliant Red)
- C.I. Reactive Yellow 5 (Procion Supra Yellow)

**Disperse Dyes (Polyester)**
- C.I. Disperse Blue 2 (Acetoquinone Dark Blue)
- C.I. Disperse Red 15 (Resoform Red)
- C.I. Disperse Yellow 16 (Suden Yellow)
4.1.3 Dyeing Recipe

4.1.3.1 Cotton

Material: Liquor ratio - 1: 40
Dye concentration - 1%, 3%, 5%, 7% on weight of cloth
NaCl - 30 g/l
Na₂CO₃ - 20 g/l
Weight of the sample cloth - 4g

4.1.3.2 Polyester

Material: Liquor ratio - 1:30
Dye concentration - 1%, 3%, 5%, 7% of weight of cloth
Depth of shade: upto 0.5% = 6% carrier on weight of cloth
0.5 - 2% = 8% carrier on weight of cloth
2 - 4% = 10% carrier on weight of cloth
4 - 8% = 12% carrier on weight of cloth
Weight of the sample cloth - 3g

4.1.3.3 Blend

Material: Liquor ratio - 1: 30
Dye - 1%, 3%, 5%, 7% of weight of fabric
Weight of the sample cloth - 4g
Blend material - 33% Polyester and 67% Cotton

4.2 METHODS

Dyeing experiments were conducted with three different colours such as blue, red and yellow on different fabric materials by varying process parameters like time, temperature and concentration. Liquor ratio could not be
varied due to restriction in the size and space constraints of the individual experimental setup. Cotton, polyester and blend material with cotton (67%) and polyester (33%) were used for evaluation. In case of cotton, reactive dyes were used. In case of polyester, disperse dyes were used. In case of polyester, carrier dyeing method was adopted. In case of blend, 2-bath dying procedure was adopted. The power consumption in each experiment was measured using watt-hour meter. The dyed sample and remaining dye solution was analysed using spectrophotometer for absorbance measurements. The spectrophotometer figure is shown in Appendix F.2. In order to assess the final quality in each technology, dyed samples are tested for parameters like, colour matching, K/S values and fastness values. Conventional electric heater, ultrasonic equipment, microwave oven, infrared dyeing machine and magnetic stirred hot plate are the equipments used for different technologies respectively. The different process parameters for different technologies have been chosen in such a way that the maximum values do not exceed the conventional operating values. The dye concentrations were kept at 1%, 3%, 5% and 7% respectively.

Experiments on dyeing were conducted for three different colours such as blue, red and yellow for different fabric materials by varying process parameters like time, temperature and concentration of dye. Different processes are adopted and energy consumption figures were noted.

### 4.2.1 Procedure for Dyeing Cotton

The sample cotton cloth to be dyed is taken and weighed. The required amount of reactive dye, dye liquor and salts like (NaCl, Na$_2$CO$_3$) are taken according to the sample weight. The required amount of liquor is put in a beaker and kept in the heater and is slowly heated. After that, the sample cotton cloth and the dye, into the beaker and kept for 5 minutes. One third of the salt is to be added for fixation of the dye. After 10 minutes, half of the
remaining salt is added. Again after 10 minutes, the remaining salt is added. Now Na$_2$CO$_3$ is added and allowed for thirty minutes to react. The temperature is maintained at desired value till the process is completed. Then the sample is taken out rinsed and soaped for about 15 minutes and washed with cold water thoroughly and dried.

The dried sample is weighed. The liquor solution left in the beaker is put in spectrophotometer and absorbance is measured. The initial dye solutions absorbance value was also measured using the spectrophotometer. The reference absorbance graphs for cotton dyes are given in Appendix -A. The dyeing cycle graphs for cotton are given in Appendix – B.1.

4.2.2 Carrier Dyeing of Polyester

Four methods are normally used for dyeing polyester fabrics:

a. Conventional aqueous dyeing (batch dyeing)
b. Aqueous dyeing with a carrier (batch dyeing)
c. High-temperature (above 100°C usually 130°C) aqueous dyeing (batch dyeing)
d. “Thermo sol” dyeing (continuous dyeing)

When dyeing polyester with disperse dyes below 100°C without special auxiliaries known as carriers, only light shades can be obtained. This is because of the low dye absorption rate by this method; other techniques are required to obtain medium or heavy shades.

A carrier is an organic chemical that has an affinity for and will swell polyester fibers. Chemicals such as biphenyl, orthophenylphenol, benzyl benzoate, butyl benzoate, 1, 2, 4-trichlorobenzene and butyl phthalamide have been used. The use of a carrier at 100°C increases the
amount of dye absorbed and decreases staining of other fibers by disperse dyes.

It is important that as much carrier as possible be removed from the yarn or fabric after dyeing because fastness properties, particularly light fastness, can be reduced by traces of some carriers. Another reason for complete removal of carrier from dyed fabrics is to prevent skin irritations in wearers who might be sensitive to these chemicals. To effect complete removal of residual carrier and loose surface dye, carrier-dyed fabrics are normally given a reduction clear using sodium hydrosulphite and caustic soda at 60°C for 10 – 15 minutes. Sodium hydrosulphite is a reducing agent that destroys the surface deposited dyestuff that if not removed would result in dyeing of poor fastness to wet treatments as well as rubbing.

In this experiment, carrier dyeing is adopted due to low temperature use and low pressure requirements.

4.2.3 Procedure for Dyeing Polyester

The sample polyester cloth is weighed. The required amount of disperse dye and carrier as per the weight of the sample cloth are also taken. A beaker with a required amount of liquor is kept in a heater and the liquor is slowly heated to attain the desired temperature of 85°C. After the temperature is reached, the cloth is put and the temperature range is maintained at 85-95°C for 80 minutes. Throughout the process, the specified temperature range is maintained. Then the sample is taken out, reduction clearing on polyester is done before washing with cold water and squeezed.

The liquor solution left in the beaker is put in spectrophotometer and absorbance is measured. The initial dye solutions absorbance value was also measured using the spectrophotometer. The reference absorbance graphs
for polyester are given Appendix C. The dyeing cycle curve for carrier dyeing of polyester is given in Appendix B.2.

4.2.4 Dyeing of Blend Material

The blend material consists of 67% cotton and 33% polyester. Here the dyeing of the blend material is carried out under 2 bath procedure. First, the cotton is dyed as per the procedure given the earlier (paragraph 4.2.1) and then polyester is dyed with carrier as given in (paragraph 42.3).

4.2.5 Procedure for Dyeing Blend

The cloth which is to be dyed is weighed. The required amount of dye liquor, dye and salts like (NaCl, Na₂CO₃) are taken according to the sample weight. The required amount of liquor is taken in a beaker and kept in the heater until it slowly reaches a temperature of 40°C. Then the cloth and the reactive dye are added in the beaker. The same procedure used to dye cotton at the temperature of 80°C and kept for 1 hour is followed. After 1 hour, the disperse dye and the carrier solution should be added according to the recipe. The temperature is raised until it reaches 95°C. Then the sample is taken out and reduction cleared hot and cold water and squeezed and it is completely dried.

The liquor solution left in the beaker is put in spectrophotometer and absorbance of the spent solution is measured. The initial dye solutions’ absorbance value was also measured using the spectrophotometer. The standard values of absorbance are calculated based on the earlier graphs. Dyeing cycle for the blends is obtained by the addition of both individual cycles of cotton and polyester.
4.3 EQUIPMENTS

For different technologies, the following different equipments are used:

- Conventional heater
- Ultrasonic cleaner
- Microwave oven
- Infra red dyeing machine
- Magnetic hot plate stirrer

4.3.1 Conventional Heater

The following are the specifications of conventional heater. The setup figure is shown in Appendix – D.1. The specifications of conventional heater are given below.

Dimensions water bath - 41cm x 30 cm
Supply voltage and frequency - 230V, 50 Hz
Heater capacity - 1000 W

4.3.2 Ultrasonic Dyeing Equipment

Dyeing experiments were carried out with flat bottom vessel, clamped inside the ultrasonic vessel containing water. The photograph of experimental setup is shown in Appendix – D.2. A piezoelectric ultrasonic transducer fixed at the centre of the bottom wall of the ultrasonic cleaner generates ultrasound of 120W power at a frequency of 50 kHz. The diffuse sound field will be operative in the heterogeneous system comprising of dye solution and cloth. Experiments were carried out using a dyeing drum having 120 mm x 175 mm width and having temperature and speed control.
Experiments were carried out at 45 rpm with temperature control. Different temperatures, processing time, concentration of liquors are maintained in the ultrasonic vessel for studying the energy utilization. The following are the specifications of ultrasound dyeing vessel.

- **Tank size**: 250 x 175 x 90 mm
- **Power**: 120 W
- **Tank Capacity**: 3 Litres (Approx)
- **Tank Material**: SS 304, 18G
- **Frequency**: 34 ± 3 kHz
- **Generator**: Built in
- **Timer**: Digital
- **Power Supply**: 230 V Single Phase, 50 Hz
- **Heater capacity**: SS Strip type heater, 200 W

**4.3.3 Microwave Dyeing**

Microwave dyeing is carried out in a microwave oven consisting of the following.

- a magnetron
- a magnetron control circuit (usually with a microcontroller)
- a waveguide and
- a chamber

The photograph of microwave oven is shown in Appendix – E.1. The specifications are given below.

- **Power supply**: 230V, 50 Hz
- **Power**: 1400 W
- **Output power**: 900 W
- **Heater output power**: 1200 W
4.3.4 Infrared Dyeing Machine

This dyeing machine comprises multiple dyeing beakers, a rotating support assembly mounting the beakers and a frame mounting the support assembly for moving the beakers about an axis of rotation, wherein at least one beaker is provided with a dosing hose for supplying the beaker with a dye or other chemical during movement of the rotating support assembly. The figure of Infrared dyeing machine is given in Appendix F.1. The beaker carriage present in the machine will rotate in the infrared heated zone with the variable speed of 24 rpm to 45 rpm through A.C. frequency drive and geared motor. The temperature obtained in the heating chamber can be up to 145°C. Injection dosing system is provided for making time to time additions to the beakers without opening the beakers. A micro processor based programmer model DC4 F/R is provided for temperature control, rate of cooling control, rate of heating, alarm for indication of completion of process cycle and for the setting of hold times at different temperature as required. Because of its proper control of temperature, efficient heating occurs which minimizes energy losses. The specifications are given below.

<table>
<thead>
<tr>
<th>Beakers</th>
<th>Power</th>
</tr>
</thead>
<tbody>
<tr>
<td>12 x Beakers of 250 ml</td>
<td>4.5 kW</td>
</tr>
<tr>
<td>16 x Beakers of 100 ml</td>
<td>4.5 kW</td>
</tr>
<tr>
<td>8 x Beakers of 500 ml</td>
<td>4.5 kW</td>
</tr>
<tr>
<td>4 x Beakers of 1000 ml</td>
<td>4.5 kW</td>
</tr>
<tr>
<td>18 x Beakers of 250 ml</td>
<td>6 kW</td>
</tr>
<tr>
<td>12 x Beakers of 500 ml</td>
<td>6 kW</td>
</tr>
<tr>
<td>8 x Beakers of 1000 ml</td>
<td>6 kW</td>
</tr>
</tbody>
</table>
Microprocessor based programmer model: MC 10 F/R/ DC4-F/R

Gasket for Beaker - 48 Nos
Sensor with lid - 2 No
5 CC and 10 CC dosing syringe - 1 No. each
Needle set - 1 No
Lamps - 3 Nos
Beaker Locking Spring - 6 Nos
8 Pin Relay - 1 No
Fuses - 1 set
Heater 750 W - 2 Nos

4.3.5 **Magnetic Stirrer Dyeing**

It consists of a hotplate and a temperature controller and a stirrer which is shown in Appendix – E.2. The speed of the stirrer is controlled by a knob. The specifications are given below.

Features - MR 3000 D
Speed, Max. - 1250 rpm
Load max. - 25 kg
Power consumption - 30 W
Hot plate dia. - 145 mm
Dimension l x w x h - 240m x 155m x 120m
Weight - 2.6 kg
Supply Voltage - 230 V / 50 Hz

4.4 **PROCESS PARAMETERS**

The process parameters varied in the different methods of dyeing are temperature, time and concentration.
Three different temperatures and times are chosen to dye cotton and polyester with three colours based on the equipment capacity for experiments. The temperature and time values for different equipments and different materials are tabulated in Table 4.1, 4.2 and 4.3 respectively. The concentrations (1% indicates 1 g per 100 g of fabric) are varied as 1%, 3%, 5% and 7%. The different temperatures are represented as T1, T2 and T3 and the times are represented as t1, t2 and t3.

Table 4.1 Experiment parameters used in cotton dyeing

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Temperature (°C)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
</tr>
<tr>
<td>Conventional</td>
<td>60</td>
<td>80</td>
</tr>
<tr>
<td>Ultrasonicator</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>Microwave oven</td>
<td>60</td>
<td>80</td>
</tr>
<tr>
<td>Magnetic stirrer</td>
<td>60</td>
<td>80</td>
</tr>
<tr>
<td>Infrared dyeing</td>
<td>60</td>
<td>80</td>
</tr>
</tbody>
</table>

Table 4.2 Experiment parameters used in polyester dyeing

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Temperature (°C)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
</tr>
<tr>
<td>Conventional</td>
<td>80</td>
<td>85</td>
</tr>
<tr>
<td>Ultrasonicator</td>
<td>60</td>
<td>80</td>
</tr>
<tr>
<td>Microwave oven</td>
<td>75</td>
<td>85</td>
</tr>
<tr>
<td>Magnetic stirrer</td>
<td>80</td>
<td>85</td>
</tr>
<tr>
<td>Infrared dyeing</td>
<td>80</td>
<td>85</td>
</tr>
</tbody>
</table>
Table 4.3 Experiment parameters used in blend (67% cotton and 33% polyester) dyeing

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Temperature (°C)*</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
</tr>
<tr>
<td>Conventional</td>
<td>60-80</td>
<td>80-85</td>
</tr>
<tr>
<td>Ultrasonicator</td>
<td>40-60</td>
<td>60-80</td>
</tr>
<tr>
<td>Microwave oven</td>
<td>60-75</td>
<td>80-85</td>
</tr>
<tr>
<td>Magnetic stirrer</td>
<td>60-80</td>
<td>80-85</td>
</tr>
<tr>
<td>Infrared dyeing</td>
<td>60-80</td>
<td>80-85</td>
</tr>
</tbody>
</table>

Note: *The first value represents temperature for cotton and second value represents for polyester.

Different parameter values are kept for different processes due to process constraint. The values are so chosen that they represent minimum, maximum and likely optimum condition. For both temperature variation experiments and time variation experiments, the concentration is kept at 4%. When one of the parameter is changed, the other two parameters are kept constant. The temperature is kept below 100°C to avoid evaporation of the dye solution. Most of the non-conventional technologies are based on open systems.