CHAPTER III

RESEARCH DESIGN

III. RESEARCH DESIGN

The methodology pertaining to the study on **"IMPROVING THE EFFICIENCY OF SELECTED NATURAL DYES ON COTTON FABRIC"** is discussed under the following headings :

3.1 Conduct of Survey
- 3.1.1. Market Survey
- 3.1.2. Household Survey

3.2 Selection of the Fabric

3.3 Pretreatment of the Fabric

3.4 Preparation of Fabric for Dyeing

3.5 Conduct of Pilot Study
- 3.5.1. Selection of Dye Sources
- 3.5.2. Selection of Mordants and Mordanting Techniques
- 3.5.3. Modifying Metallic Mordants into Processed Form of Mordants
- 3.5.4. Extraction of the Dye Solution

3.6. Optimization of Dyeing Parameters
- 3.6.1. Concentration of Dye
- 3.6.2. Concentration of Mordants
- 3.6.3. Mordanting Time
- 3.6.4. Dyeing Time
- 3.6.5. Selection of Optimized Proportions of Dyeing Variables for the Study
- 3.6.6. Procedure used for Actual Dyeing

3.7. Evaluation of the Dyed Samples
- 3.7.1. Subjective Evaluation
  - 3.7.1a. Visual Inspection
- 3.7.2. Objective Evaluation
  - 3.7.2a. Fabric Weight and Fabric Thickness
  - 3.7.2b. Breaking Strength and Elongation of the Fabric

3.1. CONDUCT OF SURVEY

3.1.1. Market Survey

A survey is a process of collecting data from the existing population units (Kothari, 2004). Market survey is very essential to gather information regarding the availability of natural dyes, extent of availability of natural dyed fabrics, purchase and sale details, type of consumers and problems faced by the shopkeepers in their routine trade. The survey included the following steps,

a. Selection of Area, Target Groups and Shops
b. Selection and Formulation of the Tools
c. Collection, Analysis and Interpretation of Data

a. Selection of Area, Target Groups and Shops

Four major textile cities in Tamilnadu namely Coimbatore, Salem, Karur and Madurai where majority of the dyeing units are situated, were identified as the areas for conduct of the market survey. These cities also housed most of the shops which sold natural as well as synthetic dyes. For conducting survey, the investigator selected 360 shops among which 120 shops were selected to collect information regarding natural dyes, 120 shops for information on synthetic dyes and another 120 textile shops to find out the information related to the availability of natural dyed fabrics. The ease of approach by the investigator and cooperation extended by the shop keepers were also taken into consideration in locating the shops for the collection of information regarding natural dyes.

b. Selection and Formulation of the Tools

The tool selected for conduct of survey was an interview schedule. An interview is one, where a number of questions or statements relating to the investigation is prepared and these questions are asked face to face and

answers recorded by the interviewer, thus information obtained is first hand and original in character as stated by Gupta (2004).

The Schedule-I comprised of questions pertaining to the availability of synthetic dyes and chemicals, purchase and sale of the product, demand for the product, category of the consumers and the problems faced in their trade.

Schedule-II which was used to collect information from the shop owners consisted of questions relating to the availability of natural dyes, cost of the dyes, fast moving dyes, type of consumers, demand for the product and other details on purchase as well as problems faced and the solutions in their trade. The Schedule-III included the availability of naturally dyed fabrics, details on purchase, sales and the problems faced by the traders. Pretesting of the survey forms was carried out by the investigator in 10 shops in each category by using the prepared interview schedules. Based on the results of the sample survey the necessary modifications were made in the interview schedules and were restructured to elicit best possible information. The finalized schedules used for the different market surveys are presented in Annexures – I, II and III.

c. Collection, Analysis and Interpretation of Data

The investigator approached the shopkeepers at a time convenient for them and explained about the purpose of the study and the necessary information was gathered by using the already prepared interview schedule. The collected data was systematically consolidated, analysed and interpreted. The results of the survey is presented in Chapter-IV : Results and Discussions.

3.1.2. Household Survey

Kothari (2004) points out that surveys are concerned with conditions or relationships that exist, opinions that are held, processes that are going on, effects that are evident or trends that are developing. The household survey

was conducted among 200 families to find out the awareness and preference of naturally dyed fabrics by the homemakers. The survey included the following steps.

a. Selection of area and sampling technique
b. Selection and formulation of the tool
c. Conducting the survey and analysis of data

a. Selection of Area and Sampling Technique

The household survey was conducted in the selected areas of the four textile cities already mentioned. Kothari (2004) and Saravanavel (1999) state that in the purposive sampling technique a researcher deliberately selects certain units for the study from the Universe, with the intension of finding out something about the population from which they are taken. In the purposive sampling method, the investigator uses his or her own judgement about the respondent to choose and pick the best to meet the purpose of the study. Hence the investigator selected purposive sampling technique for the study.

According to Housing Urban Development Corporation (HUDCO, 2002) the total family income per month for low income groups range from Rs.2,500 to Rs.4,500, for middle income Rs.4,501 to Rs. 7,500 and for high income Rs.7,501 and above. These income levels were kept in mind while selecting the 200 households from middle and high-income families because the investigator considered that these income groups were aware of the naturally dyed fabrics.

b. Selection and Formulation of the Tool

The interview schedule offers consistency and is helpful in gathering accurate and additional information about the respondents. Many researchers use this technique since it provides fairly reliable results, state Gupta (2004).

The interview Schedule-IV had questions on socio-economic status of the family, budgeting practices, awareness and extent of use of naturally dyed fabrics, factors influencing purchase of clothing items, type of dyed fabrics preferred and care of those fabrics by the homemakers. Information about the preferred dyed materials for furnishing items and type of detergents used for naturally dyed fabrics were also collected. The questions were framed in clear and simple language so that the respondent can answer with ease.

Pre testing of the prepared form was done among ten homemakers, which helped in enriching and finalizing the design of the interview schedule. All aspects including lay out, question sequence and instructions and so on should be the part of the pre test, according to Gupta (2004). Based on the pretesting, the questions were modified and reorganized. The finalized interview schedule is given in Annexure-IV.

c. Conducting the Survey and Analysis of Data

After developing the necessary rapport, the investigator collected responses from the homemakers. The importance of the survey was explained to the homemakers in order to elicit accurate response. The questions were asked one by one and information recorded instantly. The investigator analysed the survey forms carefully for completeness, accuracy and uniformity. Each aspect of the response was consolidated, tabulated and given in the Chapter-IV : Results and Discussions.

3.2. SELECTION OF THE FABRIC

Cotton fibre is a bounty given by nature widely accepted by consumers and products made of cotton have been used by man for a long time. Cotton is the purest form of cellulose that can be found in nature and exceeds physical and chemical homogeneity of any other fibre (Ramachandralu and Subramanian, 2003).

Cotton fibre posses many useful characteristics such as comfort, softness, good absorbency, colour retention, good strength, machine washable, easy to handle and sew. According to Janhan et al. (2004) cotton enjoys an extremely positive image due to its naturalness and gentleness to the human skin. Shivaleela et al. (2004) point out that the fibre absorbs and releases perspiration quickly thus allowing the fabric to 'breathe'. Cotton can stand high temperatures and takes dyes easily. Cotton will retain 24-27 times its own weight of water and is stronger when wet than dry and does not offer the typical slippery touch of synthetic fibre. This is specially favourable for health care applications requiring skin contact. Cotton offers excellent resistance to heat as well as providing dimensional stability and strength.

Cotton material with plain weave is the most commonly used fabric for various apparel purposes because of its even dye uptake and good appearance (Kumar et al., 2002 and Anitha and Jacob, 2003). Hence the investigator selected 30s count, plain weave cent per cent cotton grey material for the study.

3.3. PRETREATMENT OF THE FABRIC

The aim of the preparatory process is improving the quality, by removing impurities and foreign matters thoroughly and uniformly from the fabric and make the fabric suitable for follow up processing, points out Anthappan et al. (2006). The impurities present in grey cotton fabric are sizing ingredients, fat, waxes, pectins and natural colouring matter. Efficient removal of these impurities during grey preparation is essential to guarantee proper dyeing process (Prabaharan and Rao, 2003).

Grey cotton fabrics show a hydrophobic character due to the presence of cuticle around the fibre. Besides hydrophobic wax material, cuticle contains cellulosic and non-cellulosic polysaccharides, proteins and mineral matters which interferes with the processing of cotton in the water based dyeing and finishing processes (Pardeshi et al., 2002).

Nakamura (2000) remark that pretreatment of cotton includes desizing, scouring, bleaching and mercerizing. Desizing is to remove sizing materials, scouring is to remove the non-cellulosic cuticle constituents prior to bleaching and dyeing. This improves wettability of the fibres thereby facilitates uniform dyeing and finishing.

Bleaching is to impart perfect whiteness to the fabric by removing the natural colouring matter from the fabric, explains Pardeshi (2006). Mercerizing is the treatment given to cotton with or without tension in a strong caustic alkali like sodium hydroxide in order to improve the lustre, hand, ability to absorb dye, tensile strength and elongation (Patel et al., 2006 and Nair and Pandian, 2005).

Shenai (2003) reports that boiling in sodium hydroxide is the most widely practised conventional processing to remove such impurities and to improve the wetting and penetration of aqueous dyeing and finishing solution. It is a most effective process in removing impurities and leaves the fabric with 99 per cent cellulose content on a dry basis and also enhances the accessibility and reactivity of cotton cellulose. Hence the selected cotton grey material was mercerized as per the procedure given below :

Scouring

Material weight	:	100 g
MLR	:	1:10
Desizing agent (Lenotol)	:	1.5% (OWF)
Wetting agent (Lissopol)	:	0.5% (OWF)
Scouring agent :		
Caustic soda	:	4% (OWF)
Soda Ash	:	1%
Temperature	:	50-60°C
Time	:	15 minutes

Procedure

100 g of cotton material was scoured in a bath containing 1.5 g of linotol and 0.25 g of lissopol at 60°C for 15 minutes. The material was taken out, rinsed thoroughly by changing water three times, squeezed well and dried.

Bleaching

Weight of the material	:	100 g
MLR	:	1:10
Wetting agent alpox 200	:	1% OWF
Caustic soda	:	4% OWF
Soda ash	:	1% OWF
Hydrogen peroxide	:	2.3 % OWF
Temperature	:	60°C
Time	:	30 minutes
Neutralizing solution	:	few drops of acetic acid

Procedure

100 g of cotton material was bleached in a bath containing 3 g of hydrogen peroxide, 1 g of soda ash in 1000 ml of water MLR 1:10 at 60°C for 30 minutes. After bleaching the material washed thoroughly with hot water followed by cold. To neutralize the material was treated with mild acetic acid.

Mercerizing

Weight of the material	:	100 g
MLR	:	1:10
Sodium hydroxide	:	22% OWF

Wetting agent

Heptalic liquid	:	0.5%
Neutralizing solution (Acetic acid)	:	few drops
Temperature	:	60 -70° C
Time	:	30 minutes

Procedure

100 g of material was immersed in a bath containing 2 g of sodium hydroxide, 0.5 g of wetting agent in 1000 ml of water at 70°C for 30 minutes. The material was given hot water wash twice. To neutralize, the material was immersed in a solution containing mild acetic acid.

3.4. PREPARATION OF FABRIC FOR DYEING

Cotton has no affinity for most of the dyes from natural sources remark Gulrajani (2002). Sivakumar et al. (2003) report that natural dyes may require some mordanting agents in order to produce affinity between the fibre and the dye. Mathur (2002) defines a mordant as a mineral salt used to help the dye to adhere to the material being dyed and act as a colour fixing agent. Sharadadevi et al. (2001) suggest that cotton fabrics need to be coated with tannin which modify the affinity of fibres towards different dyes. Myrobalan (Plate – I) has ample natural tannin, which is the most important ingredient in dyeing with natural dyes producing yellow, brown, grey and black colours (Gulrajani, 1999 and Anitha and Jacob 2003).

Kumar et al. (2002) and Alikhan et al. (2005) state that myrobalan treated fabrics have more affinity to dyes and have better dye fastness and also makes pores at the surface of the fibre to which the dye can fix. The myrobalan solution was prepared by soaking 15 grams of myrobalan powder in 100 ml of soft water (pH 6-7) as suggested by Sunanda et al. (2001). The material was soaked in myrobalan solution and left overnight at room temperature. The next day the material was removed from the myrobalan

solution, squeezed thoroughly and dried in the shade without washing. When dried the unfixed myrobalan was removed by shaking which otherwise produce hindrance while dyeing. Therefore the mercerized cotton materials were treated with myrobalan solution for better dye fixation and colour fastness.

3.5. CONDUCT OF PILOT STUDY

Pilot study is a preliminary study conducted in a limited scale before the large scale are carried out in order to gain some primary information, on the basis of which the main project would be planned and formulated (Saravanavel, 1999). A pilot study was carried out in order to select dye sources, mordants, mordanting techniques and dyeing procedure are as follows :

3.5.1. Selection of Dye Sources

Based on availability, easy application, affordable cost and appealing colours, about 12 natural dyes derived from different sources like seeds, leaves, barks, tubers, roots and rinds were chosen for the study. Annatto seed, amaranth seed, jambo seed, neem leaf, henna leaf, babool bark, karingali bark, poovarasan bark, vembadam bark, turmeric tuber, madder root, red sandal wood were selected for the pilot study. The dyes were procured from a popular and standard shop and forest department in Coimbatore city.

3.5.2. Selection of Mordants and Mordanting Techniques

Sivakumar et al. (2003) report that natural dyes may require some mordanting agents in order to produce affinity between the fiber and the dye. Mathur (2002) defines a mordant as a mineral salt used to help the dye to adhere to the material being dyed and act as a colour fixing agent.

Battacharya (2002) points out that the most commonly used mordants with natural dyes are alum, chrome, copper sulphate, ferrous sulphate and

tannic acid. The chemicals used for mordanting are generally not environmentally acceptable. Gulrajani (1999) reports that metallic mordants produce bright and fast colours but they are not eco-friendly and also observed that the future of the natural dyes lies in its ability to produce fast and bright colours with the combination of non-chemical mordants.

The metallic mordants may also have a deteriorating effect on fibres. Considering the drawbacks of metallic mordants there is an imperative need to explore processed / bio mordants to minimize the use of metallic mordants (Gill and Singh 2003). In order to produce totally eco-friendly natural dyeing, it is necessary to modify the metallic mordants into processed form (Deo and Paul, 2000).

Keeping the above points in mind the investigator selected mordants such as alum, copper sulphate and ferrous sulphate and bio mordants such as pomegranate rind powder and amla powder were selected for the pilot study. The metallic mordants were modified into processed form of alum, copper sulphate and ferrous sulphate as follows :

3.5.3. Modifying Metallic Mordants into Processed Form of Mordants

a. Alum $Al_2(SO_4)_3$

One hundred grams of alum was boiled in a flat thava until the content changed to liquid state, then removed from the fire and dried overnight so as to get a powdered form of the product.

b. Copper Sulphate ($CuSO_4$)

Few drops of ghee was smeared all over the deep frying pan. One hundred grams of Copper Sulphate was spread and roasted until it turned to a white powdered form.

c. Ferrous Sulphate ($FeSO_4$)

One hundred grams of ferrous sulphate was soaked in 25 ml of stale cows urine (komeyam) in a glass beaker and left for 24 hours, the urine gets evaporated and the dried form of the product was obtained.

Mordanting Technique :

Tiwari et al. (2000) state that mordanting is a process of impregnating textiles with a mordant usually salt or acid to fasten the dyestuff which is applied before or after mordanting, sometimes a mordant is applied along with the dyestuff itself.

Kumar et al. (2002) observes that in the pre mordanting technique mordanting is the first step after which dyeing is done, while in simultaneous technique mordanting and dyeing are done simultaneously and in post mordanting technique, dyeing is the first step following which mordanting is done. In the without mordanting technique, dyeing is done without the aid of a mordant (Kadolph, 2002). The investigator selected all the four techniques for the present study.

3.5.4. Extraction of the Dye Solution

Seerangarajan (1999) points out that dye from natural sources can be extracted in alkaline, acidic and aqueous medium. As aqueous medium does not involve the use of harmful chemicals to extract the dye, the investigator selected aqueous medium for the study.

In order to find out the optimum extraction time, the dye bath was prepared by dissolving two grams of dye powder in 100 ml of water as recommended by Teli et al. (2004) in four cups and allowed to stand for 24, 36, 48 and 60 hrs respectively in cold condition. Two grams each of four different cotton samples were introduced in the respective concentrations. Then the dye solutions were boiled for ten minutes by maintaining the temperature at 100°C. The optical density was recorded using a

spectrophotometer at 530 (nm) just before and after dyeing at every 24, 36 48 and 60 hrs by taking 0.2 ml of dye solution from each dye bath and diluted 10 times. The spectrophotometer used was an ultra violet (uv) visible spectrophotometer DU series 500.

The spectrophotometer is a scanning laboratory instrument capable of analyzing in the near infra-red, visible and ultra violet spectrum. The DU series 500 uses very low to high wavelengths to measure the constituents. It measures a sample at multiple wavelengths in rapid succession, with up to four different wavelength measurements in one operation. It can scan across a range of 190 to 1100 nm, identifying the optimum wavelength, monitoring the changes in the coloured complex and then establishing the calibration curve. The instrument identifies peaks and valleys of optimum absorbance or transmittance.

The dye absorbed by the individual samples were calculated using the formula suggested by Agarwal and Gupta (2003) as given below :

$$\text{Per cent absorption} = \frac{\text{Optical density before dyeing} - \text{Optical density after dyeing}}{\text{Optical density before dyeing}} \times 100$$

The optimum time for extraction of dye was selected as 48 hours based on the maximum dye absorption.

The dye bath was prepared by dissolving 2.5 g of dye powder in 100 ml soft water. After 48 hours of extraction of dye, 0.01 g of sodium carbonate was added to the dye solution to increase the exhaustion of the dye (Breeni, 2006 and Neetu and Shahnaz, 2003).

In pre mordanting two grams of cotton materials were soaked in the respective 80 ml of mordanting solution for an hour at room temperature. The mordant bath was heated and the temperature was gradually raised to boil for 10 minutes. After mordanting the materials were taken out, squeezed

thoroughly and introduced into the respective dye solution without intermediate washing. The materials were taken out, rinsed thoroughly and soaping was done by boiling the materials with 2 g / lt of non-ionic detergent powder at 60°C for 2 minutes. Finally the materials were thoroughly washed and dried in the shade.

In simultaneous mordanting each of the specified quantity of mordant was mixed well with the prepared dye solution. Each of material was dipped into this mixture and soaked for an hour. The bath was heated to boil and continued for 30 minutes. Finally it was taken out, rinsed thoroughly and soap boiling was done, finally the materials were thoroughly washed and dried in the shade.

In post mordanting technique, each of the dyed material was squeezed well and dipped in the respective mordanting solution for an hour and the bath was heated to boil and continued for 10 minutes. Finally it was taken out, rinsed thoroughly and soap boiling was done. The samples were thoroughly washed and dried in the shade.

The dyed samples produced from the pilot study (150 Nos.) were visually evaluated by a panel of judges consisted of 25 members, who were specialists in the field of Textiles and Clothing in Avinashilingam University for Women. Panel members evaluated the dyed samples in terms of evenness in dyeing, brilliancy of colour, lustre and general appearance. The proforma used for the evaluation is given in Annexure – VI. Based on their recommendations, the following dye sources, mordants and mordanting techniques (Plates I and II) were selected for the final study which are as follows :

Dye Sources

Local Name		Botanical Name
Annatto seed dye	:	*Bixaorellana*
Babool bark dye	:	*Acacia nilotica*
Karingali bark dye	:	*Acacia catechu*
Madder root dye	:	*Rubia cardifolia*
Red Sandalwood dye	:	*Petrocarpus santalinus*
Vembadam bark dye	:	*Ventilago maderaspatana*

Annatto Seed

Annatto (*Bixaorellana*) seed is an age old source for food colour. It is a fast growing evergreen tropical tree. The leaves are simple with pink flowers and red pods. Each seed is coated with a yellowish orange substance which ultimately is the dye. The seeds are distributed mainly in South America and Brazil.

Babool Bark

Babool (*Acacia nilotica*) is an umbrella shaped tree 4-15 m tall. Two types of thorns around long, straight, white and small, hooked and brownish ones. The bark gives light brown colour. Native of Africa and the mid eastern countries. *Acacia nilotica* tree is found everywhere in India. It grows faster in the Rajasthan desert.

Karingali

Karingali (*Acacia catechu*) bark is a moderate sized tree, 9-12 m high, with a dark coloured bark and short dark brown or purple, glabrous leaves bi-pinnate, 10-15 cm long main rhachis pubescent with glands between many of the pairs of pinnae. Catechu is extracted from the heartwood of a medium sized Indian tree. It is produced in India and South East Asia.

Madder Root

Madder (*Rubia cardifolia*) is a prickly climbing tree. The plant is of commercial importance because of the valuable dye present in the roots and lower twig. The pigment contained in the red moss between the outer skin and the wood. *Rubia cordifolila* is found mainly in North – West – Himalayas, the Nilgiris and other districts of India.

Red Sandal Wood

Red Sandal Wood (*Petrocarpus santalinus*) a very pretty, moderate sized deciduous, up to 11 m in height with blackish brown bark, deeply cleft into rectangular plates and dark purple heartwood, gradually narrowed into a short stalk, winged, the central hard portion containing the seed, (reddish brown and smooth). Distributed in Cuddapah district in Andra Pradesh and adjoining areas of Tamil Nadu and Karnataka.

Vembadam

Vembadam (*Ventilago maderaspatana*) a large much branched, woody climber with branches hanging down and dark grey bark having vertical cracks exposing the inner vermilion surface. Leaves simple alternate oblong – lanceolate or elliptic ovate, obtuse or acute, entire or crenate base, rounded or acute, main nerves 4-5 pairs. Flowers greenish with an offensive odour in drooping pubescent terminal panicles, petals much smaller than sepals. Fruits yellowish globular nuts, supported by the persistent calyx, wing linear oblong, one nerved rounded at the apex. It grows throughout India in forests.

Mordants and Mordanting Techniques

Metallic mordants	:	Alum, Copper sulphate and Ferrous sulphate
Processed Mordants	:	Processed form of Alum, Copper sulphate and Ferrous sulphate
Bio Mordants	:	Pomegranate rind powder and Amla powder
Mordanting Techniques	:	Pre, simultaneous, post and without mordanting.

Based on the visual evaluation the best mordanting and mordant techniques namely pre-mordanting for alum, post mordanting technique for copper sulphate, and simultaneous mordanting technique for ferrous sulphate, were selected for dyeing the cotton materials for the study.

3.6. OPTIMIZATION OF DYEING PARAMETERS

Nair and Pandian (2005) warns that eco-friendliness does not mean that natural dyes do not have azo chromospheres group / carcinogenic amines, but the selection of proper mordants and optimization of dyeing conditions may reduce the effluent load, thus making it more eco-friendly.

A series of experiments were carried out by the investigator as per procedures suggested by Rani and Singh (2003) to determine optimum values for dyeing parameters, namely dye concentration, dyeing time, mordant concentration and mordanting time. In each optimization, the values of the parameters being optimized were varied while keeping the following parameters constant. The optimized value from previous experiment was used as constant for next parameter. The constant parameters used in all the experiments were :

Material liquor ratio	-	1 : 40
Dye soaking time	-	48 hrs
Dye extraction temperature	-	100°C
Dyeing temperature	-	100°C
Mordant soaking time	-	1 hr
Mordanting temperature	-	100°C
pH for dyeing and mordanting	-	7.00
Dyeing extraction medium	-	aqueous

3.6.1. Concentration of Dye

The concentration of the dye was optimized by taking four dye concentrations starting from minimum as one, two, three and four grams of

dye powder per 40 ml of water in four cups and allowed to stand for 48 hours in cold condition. Four different cotton samples were immersed into the respective concentrations. Then the dye solutions were boiled for 60 minutes by maintaining the temperature at 100°C. The optical density was recorded after 15, 30, 45 and 60 minutes. The maximum dye absorption was taken as the optimum concentration.

3.6.2. Concentration of Mordants

Alum, Copper sulphate and Ferrous sulphate with four different concentrations were taken and optimum concentrations of mordants were determined on the basis of highest per cent of dye absorption. As suggested by Rakshi and Vankar (2005) mordanting was carried out by using 2-4 per cent concentration of mordants. The four different concentrations adopted for alum were one, two, three and four per cent and that of copper sulphate and ferrous sulphate were 0.5, 1.0, 1.5 and 2.0 per cent.

3.6.3. Mordanting Time

To optimize the mordanting time the cotton materials were mordanted with optimized percentage of mordants and the temperature was raised to 100°C. The optical density was recorded after 15, 30, 45 and 60 minutes. Optimum mordanting time for cotton material was decided based on the time required to get the highest optical density.

3.6.4. Dyeing Time

To optimize the dyeing time the cotton materials were dyed individually with optimized percentage of dye powder for 15, 30, 45 and 60 minutes. The optical density was recorded based on the maximum time taken for absorption was taken as the optimum dyeing time.

3.6.5. Selection of Optimized Proportions of Dyeing Variables for the Study

Based on the spectrometer reading the optimized values for dyeing parameters were selected. The details of optimized dye concentration variables for dyeing cotton material are given in Table I.

TABLE I

OPTIMIZATION OF DYE CONCENTRATION

S.No.	Name of the dye	Dye Concentration g / 100 ml of water	Per cent Absorption
a.	Annatto seed	1	26.1
		2	**26.5**
		3	25.8
		4	25.2
b.	Babool bark	1	19.7
		2	**20.0**
		3	19.2
		4	18.7
c.	Karingali bark	1	15.7
		2	16.7
		3	15.8
		4	15.8
d.	Madder root	1	21.7
		2	**22.6**
		3	22.0
		4	21.8
e.	Red sandal wood	1	23.5
		2	**24.4**
		3	23.6
		4	23.4
f.	Vembadam bark	1	20.9
		2	**21.4**
		3	20.8
		4	20.7

It is evident from the Table I that two grams of dye powder in 100 ml of water (2 per cent) showed the maximum per cent absorption value

irrespective of dye selected for the study. The details of optimized mordant concentration variables are given in Table II.

TABLE II

OPTIMIZATION OF MORDANT CONCENTRATION

S.No.	Name of the Mordant	Mordant Concentration g /100 ml of water	Per cent Absorption
a.	Alum	1%	14.7
		2%	**15.6**
		3%	15.2
		4%	14.9
b.	Copper sulphate	0.5%	11.9
		1.0%	**12.5**
		1.5%	12.1
		2.0%	11.7
c.	Ferrous sulphate	0.5%	23.4
		1.0%	**25.0**
		1.5%	24.4
		2.0%	22.1
d.	Pomegranate rind powder	1%	21.3
		2%	**22.0**
		3%	21.7
		4%	21.5
e.	Amla powder	1%	20.5
		2%	**21.2**
		3%	21.0
		4%	20.8

Table II revealed that the rate of absorption for alum and biomordants was high at a concentration of 2 grams per 100 ml of water while for the other mordants copper sulphate and ferrous sulphate, the optimum absorption was found as one per cent.

Table III gives the time taken for optimization of dye extraction, mordanting and dyeing.

TABLE III

OPTIMIZATION OF DYE EXTRACTION, MORDANTING AND DYEING TIME

S.No.	Parameters	Time	Per cent extraction
a.	Dye Extraction Time (hrs)	12	24.3
		24	25.8
		36	26.7
		48	**27.8**
		60	26.4
b.	Mordanting Time (min)	15	24.3
		30	25.4
		45	**26.3**
		60	24.9
c.	Dyeing Time (min)	15	22.9
		30	24.2
		45	**24.5**
		60	23.8

It is evident from Table III that the optimum time for extraction of dye was found as 48 hours. The dyeing and mordanting time was found as 45 minutes.

The optimized parameters selected for the final study of dyeing the cotton materials are given in Table IV.

TABLE IV

OPTIMIZED PARAMETERS SELECTED FOR THE STUDY

S.No.	Dyeing Variables	Trial Proportions	Selected Proportions
1.	Dye Concentration	1,2,3,4,g / 100 ml	2 g / 100 ml
2.	Dye extraction time	12,24,36,48,60 hrs	48 hrs
3.	Mordant concentration		
	a) Alum	1%, 2%,3%, 4%	2.0%
	b) Copper sulphate and Ferrous sulphate	0.5%, 1.0%, 1.5%,2%	1.0%
	d) Pomegranate rind powder	1%, 2%,3%, 4%	2%
	e) Amla powder	1%, 2%,3%, 4%	2%
4.	Mordanting time	15,30,45,60 min	45 min
5.	Dyeing time	15,30,45,60 min	45 min

It is evident from the Table IV that two grams of dye powder in 100 ml of water (2 per cent) showed the maximum per cent absorption value irrespective of all dyes selected for the study. The optimum time for extraction of dye was found as 48 hrs and the dyeing time was as 45 minutes. It was found for the concentration of mordants that the absorption for alum, pomegranate rind and amla were high at a concentration of two grams per 100 ml of water, while the other mordants like copper sulphate, ferrous sulphate, the optimum was one per cent to yield a good shade of colour. Hence two per cent mordant concentration for pomegranate rind, amla and alum and one per cent each of copper sulphate and ferrous sulphate were used for the study. The optimum time for mordanting and dyeing time was found as 45 minutes hence 45 minutes was used for dyeing.

3.6.6. Procedure Used for Actual Dyeing

The myrobalan treated mercerized cotton samples were soaked in water to remove air and soften the material to facilitate dye penetration as stated by Gill and Singh (2003). The cotton samples were dyed following the optimized parameters selected for the study. The dyed samples were taken out, rinsed thoroughly and soaping was done by boiling the samples with 2 g / lit of non-ionic detergent powder at 60°C for two minutes. Finally the samples were thoroughly rinsed and dried in the shade. The nomenclature of the dyed samples are given in Table V.

TABLE V

THE NOMENCLATURE OF THE DYED SAMPLES

S.No.	Sample
1.	ADS
2.	BDS
3.	KDS
4.	MDS
5.	RDS
6.	VDS
7.	AMAP
8.	AMFS
9.	AMCPT
10.	BMAP
11.	BMFS
12.	BMCPT
13.	KMAP
14.	KMFS
15.	KMCPT
16.	MMAP
17.	MMFS
18.	MMCPT
19.	RMAP
20.	RMFS

S.No.	Sample
21.	RMCPT
22.	VMAP
23.	VMFS
24.	VMCPT
25.	APAP
26.	APFS
27.	APCPT
28.	BPAP
29.	BPFS
30.	BPCPT
31.	KPAP
32.	KPFS
33.	KPCPT
34.	MPAP
35.	MPFS
36.	MPCPT
37.	RPAP
38.	RPFS
39.	RPCPT
40.	VPAP

S.No.	Sample
41.	VPFS
42.	VPCPT
43.	APP
44.	APS
45.	APPT
46.	BPP
47.	BPS
48.	BPPT
49.	MPP
50.	MPS
51.	MPPT
52.	AAP
53.	AAS
54.	AAPT
55.	BAP
56.	BAS
57.	BAPT
58.	MAP
59.	MAS
60.	MAPT

Dyes

A – Annatto
B – Babool
K – Karingali
M – Madder
R – Red Sandal
V – Vembadam
DS – Dyed Sample

Metallic Mordants

MA – Metallic Alum
MF – Metallic Ferrous sulphate
MC – Metallic Copper sulphate

Processed Mordants

PA – Processed Alum
PF – Processed Ferrous sulphate
PC – Processed Copper sulphate

Bio-Mordants

P – Pomegranate Rind
A – Amla

Mordanting Technique

P - Pre
S – Simultaneous
PT – Post

3.7. EVALUATION OF THE DYED SAMPLES

The dyed samples were evaluated by subjective and objective evaluation.

3.7.1. Subjective Evaluation

3.7.1a. Visual Inspection

The shade of the fabric creates the first impression in the mind of an observer. Feel and handling of fabric determine the acceptance of fabric (Sule and Bardhan, 1999). To find out the acceptance of the dyed samples, visual evaluation was carried out by the panel members. The panel which consisted of 25 judges comprising of P.G. students and staff members specialized in textiles and clothing, evaluated the dyed samples using an evaluation sheet. The criteria considered for evaluation were evenness in dyeing, brilliancy in colour, luster and overall appearance of the samples. The scores were consolidated and the data were considered for further indepth study.

3.7.2. Objective Evaluation

Textile testing is the process of inspecting, measuring and evaluating the characteristics and properties of textile materials. The mercerized original and the dyed samples were tested for their fabric weight, fabric thickness, breaking strength and elongation, wettability and absorbency tests.

3.7.2a. Fabric Weight and Fabric Thickness

Fabric Weight

Fabric weight is an important component for comparing the two similar fabric constructions. Saini (2004) describes fabric weight as the relative weight of the fabric and expressed as the weight of a particular size of piece, such as grams per square meter or ounces per square yard.

Weight of the fabric is determined as weight per unit area, states Stoker et al. (2005). A sample (10 cm x 25 cm) was cut using a square

template and electronic weighing balance was used to find out the weight of the samples. The inference obtained was calculated using the formula :

$$\text{Grams per Square meter (GSM)} = \frac{\text{Weight of the fabric x Square metre}}{\text{Area of square}}$$

Weight of the fabric = x (g)
Square meter = 100 cm x 100 cm = 10,000 cm^2
Area of square = length x breadth square units

The same procedure was followed to find out the fabric weight of original and dyed samples used in the present study. Fabric weights were carefully recorded and the mean value was calculated.

Fabric Thickness

Fabric thickness is defined as the distance between the upper and lower surface of the material measured under a standard pressure, using the Baty Thickness Tester with an accuracy of 0.01 mm, as explained by Stoker et al. (2005) and Paul (2005). The fabric was placed between the pressure foot and anvil, the reading was noted from the dial. Ten readings were taken from different places of the original and dyed samples and the mean value calculated. Fabric thickness was noted in ten different points of the original and dyed samples and mean value was calculated and considered as the fabric thickness.

3.7.2b. Breaking Strength and Elongation of the Fabric

The breaking strength is the ability of a fabric to withstand a tearing force where yarns are broken (one or a few yarns at a time). It is the resistance of the fabric to a tensile load or stress in either the warp or filling directions, as described by Osayuande(1990).

According to Vaishnav and Joshi(2000), breaking strength is the force required to break the fabric when it is under tension. Elongation is the extent

to which the fabric under tension extends, till it cut off, defines Nakamura (2000). The per cent strength loss and per cent change in elongation at break were determined by the Tensile Testing Method using Tensile Strength Tester, according to standard procedures suggested by Prabaharan and Rao (2003) and Paul (2005).

The Eureka Model Tensile Strength Tester was used for the study. The rate of traverse and capacity of the machine was 45 cm per minute and 150 kg respectively. The gauge length was kept as 25 cm, the dial of the machine was calibrated in pounds and kilograms. Ravelled Strip Method was followed and the test samples from each of the dyed materials with the length of 33 cm and a width of 7 cm were cut from the warp and weft directions separately. Each sample was ravelled from both the edges until the width measured five centimeters. Each sample was clamped between the two jaws. Care was taken to see that the sample was perpendicular to the load. The load was applied until the sample was torn. The dial reading in kilograms for breaking strength and elongation in centimeters were noted. Ten readings for the original and dyed samples both in warp and weft direction were recorded.

3.8. CONDUCTING WETTABILITY AND ABSORBENCY TEST

The wettability and absorbency tests included drop test, sinking test and capillary rise test.

3.8.1. Drop Test

The ability of a fibre to take up moisture is termed as absorbency. According to Skinkle (1872) wettability is the time taken in seconds for a drop of water to sink into the fabric. If any fabric takes more than 200 seconds to absorb water the same is considered as unwettable.

A burette filled with distilled water was clamped in a stand. The sample was mounted in an embroidery frame and was placed at the base of the stand. The distance between the sample and burette nozzle was kept

constant as shown in Plant - IV. The nozzle of the burette was opened just to allow a drop of water to fall on the sample. The stop watch was started simultaneously and it was stopped when the drop of water fully sank into the material. The time taken for this was noted. The same procedure was repeated for ten times for the original and dyed sample and the mean value was calculated and recorded.

3.8.2. Sinking Test

Sinking test is a simple test that helps to measure the wettability of a fabric (Booth, 1996). As suggested by Skinkle (1979) ten samples were cut into the size 5 cm x 5 cm square from the mercerized original and the dyed samples. A 1000 ml beaker was filled with distilled water and few drops of wetting agent was added into the distilled water. The sample was dropped on the surface of the water from a standard height. The stop watch was started when the fabric struck the surface of water and stopped when the last corner sank below the water surface as shown in Plate – V and the time required for the sample to sink was noted. The same procedure was repeated for ten samples. The mean value was calculated for the above samples. Similarly, the mean values of the mercerized original and the dyed materials were calculated and the sinking time of each material was recorded separately.

3.8.3. Capillary Rise Test

Skinkle (1979) and Paul (2005) pointed out that the capillary travel method measures the rapidity of absorption. Ten samples were cut into sizes of 15 cm length and 2.5 cm width from the mercerized original and the dyed samples. One end of the sample strip was pasted with a glass rod which was placed on heavy wooden blocks and at the other end, two grams weight was attached to keep the sample straight. At the weighed end 2 cm of the sample was allowed to immerse in a tray of distilled water as shown in Plate - VI. The rise of the water level in the strip was noted by keeping length of the fabric as 5 cm constant. The same procedure was repeated for ten samples from the

same material and the mean value was calculated and the capillary rise of each material was recorded carefully.

3.9. COLOUR FASTNESS TEST

The colour applied to the textile material should be retained as long as the fabric lasts. Different dyes have different degrees of fastness to various conditions as perspiration, exposure to sunlight, crocking and washing (Suneetha and Mahale, 2003).

The American Association of Textile colourants and chemists (AATCC, 1995) have established Standard Terminology for rating colour fastness properties of fabric through different tests and also evaluating colour staining and colour transfer in fabrics. The investigator followed the AATCC grey scales and true view colour matching cabinet for the assessment of colour fastness of samples.

3.9.1. Effect of Sunlight and Washing on the Fabric

Gupta (1999) opines that fastness to light is one of the most important properties of dyed fabric needed to fulfil its utilization purpose over a period of time. According to Lyle (1982) a majority of colours fade when exposed to sunlight and a few become dark. Gupta (1990) remarks that the fastness to sunlight depends on the number, nature and position of constituent groups on the dye chromosphore. Ingamell (1993) states that humidity, air, temperature, surface tension of the sample, presence of atmospheric impurities, spectral quality and intensity of light source are variables that affect the influence of light on the fading of dyes.

AATCC (1995) defines colour fastness to light as the resistance of a material to a change in its colour characteristics as a result of exposure of the material to sunlight or an artificial light source. The light fastness of all the dyed samples were tested, according to ISO recommendation No : 2 – R102 day light method – 16-1995.

A sample piece of 16 cm x 5 cm was cut from each of the dyed materials. The length was divided exactly into eight equal divisions of 2.5 cm width each, by drawing lines. A black paper of the same size with equal number of divisions marked was attached width-wise to each of the samples at the top. The first division on the paper was removed on the first day and exposed to sunlight from 9.00 AM to 4.30 PM. Consecutively the second division was cut on the second day and so on. Finally, the first division after exposing it for seven days was assessed for colour change using AATCC greyscale (1995). The same procedure was repeated for all the samples of the dyed material.

Gohl and Vilensky (1987) explain that the loss in colour during laundering is referred to as lack of wash fastness or bleeding. Adjacent white materials become coloured due to the transfer of dye from the coloured material and referred as staining.

The wash fastness of all the dyed samples were tested, according to ISO 105-C06 : (1994) Raul (2005). The laundrometer consists of a central axis with four rods with eight stainless steel jars (two jars of each rod) with tight fitting lids which rotates inside the hot water bath in clockwise direction when the machine is in function. Test sample, of 5 cm x10 cm size was cut from each of the dyed samples, were sandwiched between undyed desized muslin cloth one at a time. Composite specimens were washed with 4 g of phosphate ECE-B detergent powder (composition is given in Annexure - VIII) in 1000 ml of water for 45 minutes at 40°C for the wash test, after which the samples were rinsed in cold water thoroughly, squeezed well and dried. The colour change and staining of the samples were assessed using AATCC grey scales.

3.9.2. Effect of Wet and Dry Crocking and Wet and Dry Pressing

a. Wet and Dry Crocking

According to AATCC (1995) crocking is defined as the surface of a coloured yarn or fabric to another surface or to an adjacent area of the same fabric principally by rubbing. Crocking test determines the extent to which colour is transferred from the surface of the dyed fabric to another by rubbing (Adanur 1995).

As suggested by AATCC (1995), Sasmira Crock Meter was used for ascertaining the fastness of dyed materials for wet and dry crocking. It consisted of two metal blocks. The base block was stationary, while the upper block had an arrangement to move to and fro on the base by means of a rotating handle. There was a finger knob attached to the upper block to hold the material with ring. Each test sample was cut to a size of 20 cm x 10 cm. The sample was mounted on the flat base of the Crock Meter. The white material (5 cm x 5 cm) was wrapped around the finger knob of the upper movable block with a ring. The number of rubs given was standardized and fixed as ten rubs. The white material was rubbed to and fro against the dyed samples along a track of 10 cms with pressure of 900g of the finger knob. The colour transfer from the dyed sample to the white material was assessed using AATCC grey scale. The same procedure was repeated for all the samples of the dyed materials.

For wet crocking, the damp white material was used and the same procedure was followed and repeated for all the dyed samples. The colour fastness of each dyed material to dry and wet crocking was carefully observed and recorded separately.

b. Wet and Dry Pressing

Colour fastness to wet and dry pressing was tested following the specification of Bureau of Indian Standards – BIS (1989). For dry and wet pressing test, a sample measuring 5 cm x 10 cm was cut from each of the

dyed materials. Each of these samples were sandwiched between a white cotton material of same size. For dry pressing a hot iron was placed on each of the composite specimens for 5 seconds at a temperature of 350 degree Farenheit.

The colour transferred from the dyed samples were assessed using AATCC grey scale. The same procedure was repeated for all the samples of the dyed materials. Thus the colour fastness of each dyed material to wet and dry pressing was carefully observed and recorded separately.

3.9.3. Effect of Perspiration on the Fabric – Colour Fastness to Perspiration

According to AATCC (1995) this test method is used to determine the fastness of the coloured textiles to the effect of perspiration. Perspiration is a saline fluid secreted by the sweat glands. It may be acidic or alkaline in nature. The samples were therefore tested for colour change in both acidic and alkaline solution. Two specimens (5 cm x 10 cm) were cut from each of the dyed samples and sandwiched between desized white cotton material, stitched on three sides and numbered. The instrument used for this test was the perspirometer.

Acetic acid and sodium bicarbonate was added to get the required pH value in acidic and alkaline solutions respectively. The prepared test samples were saturated with the respective solutions and kept for thirty minutes. The samples were then removed from the respective solutions, squeezed and mounted on the instrument placing each in between the plastic plate. The weight was thus replaced on this set up and the entire instrument was kept in the oven at 60° C for four hours. The samples were removed and evaluated for colour change and staining using the grey scale. The same procedure was adopted for all the samples of the dyed materials.

3.9.4. Colour Strength (K/S Values) of Dyed Samples

Colour strength (K/S values) of cotton was measured using x-rite colour difference meter (CDM). The x-rite CDM is a tristimulus reflection colorimeter, with spectral response closely matching the Commission International de E'clariage (CIE) standard observer under illuminant D65 and the 10° observer. The instrument operates as a colour difference meter. The CDM stores a single reference against which all subsequent measurements are compared.

The first significant advances made towards giving quantitative form to the human sensation of colour was made by the CIE (Commission International de E'clarirage). It is based on how eye perceives a mixture of the three primary colours (red, green and yellow) using the tristimulus values x, y, z, in the visible spectrum range (400 nm - 700 nm), so it is difficult to establish quantitative relationship of colour differences, where, (L) refers to the lightness or darkness of colour with values from (100-0), (a) values change from negative (green) to positive (red), and (b) values run from negative (blue) to positive (yellow) explains Michael et al. (2003). The relationship between the Hunter L, a, b, values and the CIE x, y, z values is as follows :

The total colour strength difference (?E) was calculated from the following equation :

$$?E = [(?L) + (?a) + (?b)]^{½}$$

where

$$?L = L_s - L_r,\ ?_a = a_s - a_r,\ ?_b = b_s - b_r$$

s, r represent sample and reference specimen respectively.

3.10. ASSESSING ALLERGIC REACTIONS DURING PHYSICAL CONTACT WITH HUMAN SKIN

To assess the allergic reactions during physical contact with human skin, the investigator randomly selected 54 samples (6 dyes x 8 mordants – 3 metallic, 3 processed and 2 bio mordants) from each of the dye group for the study. Wrist bands were made out of these samples with a dimension of 10" x 1" with adjustable Velcro tape so that the volunteers can easily wrap them around their upper arm. The investigator selected 54 members who were, sensitive to skin allergy and were asked to wear this band continuously for 12 hours up to 30 days and report even if they have any minute skin problem. The subjects were monitored by the researchers every day and allergies if any were recorded. Observations were made every 5^{th} day up to completion of 30 days of test period. The points for observation were red marks on the skin, swelling on the skin, rashes on the skin, itching of the skin and irritation on the part.

3.11. STUDYING THE PHYSICO-CHEMICAL CHARACTERISTICS OF NATURAL DYE EFFLUENT

The nature and chemical composition of textile wastewater will vary depending on the processes carried out in the textile process houses, Khandan et al. (2004). Textile wastewater contains substantial pollution loads in terms of COD, BOD, TDS and heavy metals. The values for these parameters are very high as compared to the values suggested set up by the environmental quality standards, as Shukla (2005).

In the present investigation, physico-chemical parameters like temperature, pH, TSS, TDS and COD were analysed by using standard methods suggested by Amercian Public Health Association (1995).

A portion of the effluent was collected in the suitable sterilized airtight containers and stored at 4°C to find out the physico-chemical analysis and used for watering the plants (Pot Culture Study).

Effluents contain dyes and chemicals in higher concentration, which impart colour to the receiving streams not only affecting their aesthetic nature but also interfering with the transmission of sunlight into streams and thereby reducing photosynthetic action. The colloidal and suspended impurities with colour cause an unsightly appearance as pointed out by Jajpura et al. (2004).

3.11.1 Potential Hydrogen (pH)

The pH of any dye effluent is a very important parameter, ranging between 5.5 and 9.0. It is a measurement of hydrogen ion concentration and indicates instantaneously the acidic or alkaline condition of a dye effluent. Electrometric methods are by far the most accurate and suffer with little or no interference. pH meter is widely employed instrument for the electrometric measurement of pH. pH values are useful to determine the type of treatment to be applied to the effluent and study the efficiency of treatment.

3.11.2. Total Suspended Solids (TSS) and Total Dissolved Solids (TDS)

a. Total Suspended Solids (TSS)

The undissolved substances present in wastewater is usually referred as the suspended solids. Determination of suspended solids is as important as that of BOD. It is valuable in judging the pollution potential of an effluent and also helps in deciding the efficiency of treatment. It is also useful in determining the load on biological treatments after the removal of settleable solids in primary setting tanks.

The suspended substance was determined by filtering or centrifuging two litres of natural dye effluent sample by following the filtration method as suggested by APHA (1998).

b. Total Dissolved Solids (TDS)

Total dissolved solids (TDS) comprises of inorganic salts and small amounts of organic matter that are dissolved in water. TDS included both dissolved organic and inorganic solids, the environmental problems are

mainly associated with the indiscriminate disposal of inorganic TDS bearing effluents. High hardeners in conjuction with high alkalinity causes scale formation. Abnormal high or low dissolved solids disturb osmotic balance of native species. Disposal of the salt laden effluents into the ground and earth surface, cause pollution and render them unfit for domestic, industrial and agricultural use. The change in the density of water cause trouble in floatation and sedimentation. The total dissolved solids in the natural dye effluent was analysed by following evaporation method suggested by APHA (1998).

3.11.3. Chemical Oxygen Demand (COD)

This test is highly useful to find out the pollutional strength of industrial effluent. COD is a measure of the oxygen required to oxidize unstable materials in a sample by means of dichromate in an acid solution. To measure the content of organic mater of an effluent, normally COD test is carried out. In COD test a strong chemical oxidizing agent is used in acid medium and the oxygen equivalent of the organic mater is determined. The test is normally carried out using $K_2Cr_2O_7$ at temperature in the presence of some catalyst like silver sulphate. The chemical oxygen demand was analysed following the method suggested by APHA (1998).

3.12. STUDYING THE EFFECTS OF EFFLUENTS ON PLANT GROWTH

3.12.1. Selection of Plants

The plants selected for assessing the growth characteristics include green gram, horse gram and cowpea as these were easily available, grow fast (within three months time), regularly consumed by majority of people and nutritionally rich.

3.12.2. Collection of Seeds and Soil

The seeds for the plants were collected from Tamil Nadu Agricultural University, Coimbatore. Red loamy soils and sand were collected from

Thondamuthur area of Coimbatore district, mixed thoroughly in 50 : 50 proportion and filled in 10 kg capacity pots.

3.12.3. Conduct of Pot Culture Study

The pots were segregated into three groups. The first set of three pots namely, T_1 was treated as control group and ordinary tap water was fed. The second set T_2 was fed with 50 per cent effluent + 50 per cent ordinary tap water while the third set T_3 was fed with 100 per cent effluent water. The seeds of green gram, horse gram and cowpea were selected at random and were sown in the three sets of pots. All the pots were kept under uniform conditions of sun light and air and were watered regularly (Plate – VII).

3.13. BIOMETRIC OBSERVATION OF PLANT SAMPLES

3.13.1. Analysis of Growth Parameters of Plant Samples

Three samples from each set were collected randomly at 30 days, 60 days and 90 days and their root length and shoot length were measured and recorded (Plate – VIII) as suggested by Sathya et al. (2004)

3.13.2. Germination Percentage

The germination percentage and vigour index were calculated at the first emergence of the seedlings following the standard procedure. After seven days of sowing germination percentage of the seedlings was calculated using the formula

$$\text{Germination Percentage} = \frac{\text{Number of seeds germinated}}{\text{Number of seeds sown}} \times 100$$

The protrusion of the radical through seed coat was taken as the criterion of germination.

3.14. ANALYSIS OF SOIL SAMPLES

Soil analysis was done prior to the day of sowing as well as at the end of the experimental period. The soil samples were air dried, gently ground with wooden mallet sieved through 2 mm sieve.

3.14.1. Soil pH and Electrical Conductivity

Soil reaction in a soil water suspension ratio 1 : 2 : 5 was estimated using a glass electrode (Jackson, 1973). The electrical conductivity was measured using a conductivity bridge and expressed as $d\delta m^{-1}$.

3.14.2. Estimation of Nitrogen

Available nitrogen in soil sample was estimated by the alkaline permanganate method as suggested by Subbiah and Asija (1956).

3.14.3. Estimation of Phosphorous and Potassium

The phosphorous content was estimated by extracting the soil with 0.03 N ammonia in 0.025 N. HCl extract (HCl extract Olssen method described by Jackson, 1973). Potassium was estimated by extracting with neutral normal ammonium acetate as per the procedure outlined by Standford and English (1949).

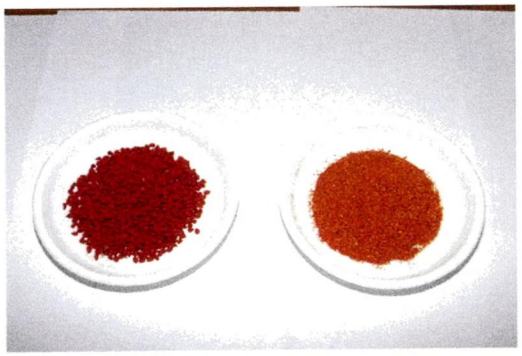
The equipments used for the entire study is given in Plates – IX and X.

3.15. ANALYSIS AND INTERPRETATION OF DATA

After the data collection, it was essential to organize the information in a systematic manner, in order to obtain the desired results and interpretation scientifically. The information thus collected were analysed statistically and findings are given in Chapter – IV : Results and Discussion.

PLATE - I

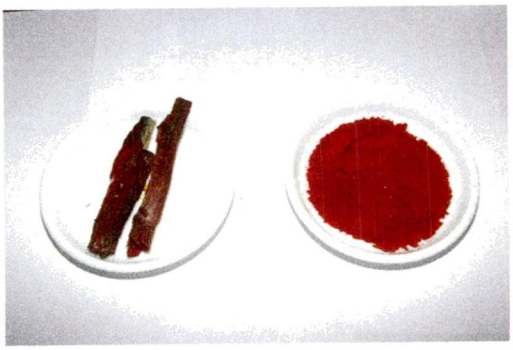
MYROBALAN USED FOR TREATING THE FABRIC



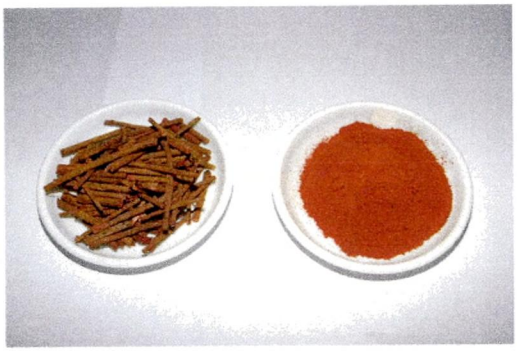
ANNATO

MADDER

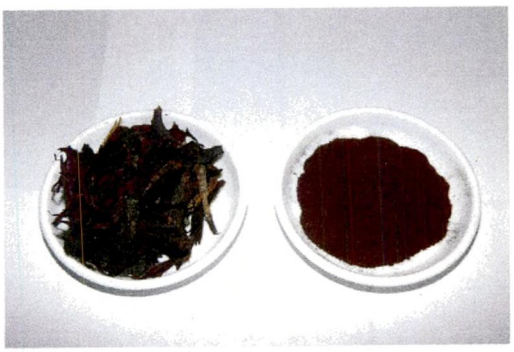
BABOOL



RED SANDAL



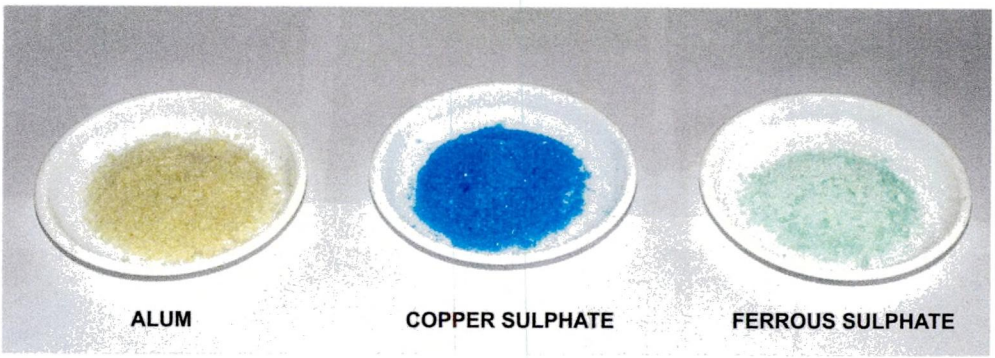
KARINGALI



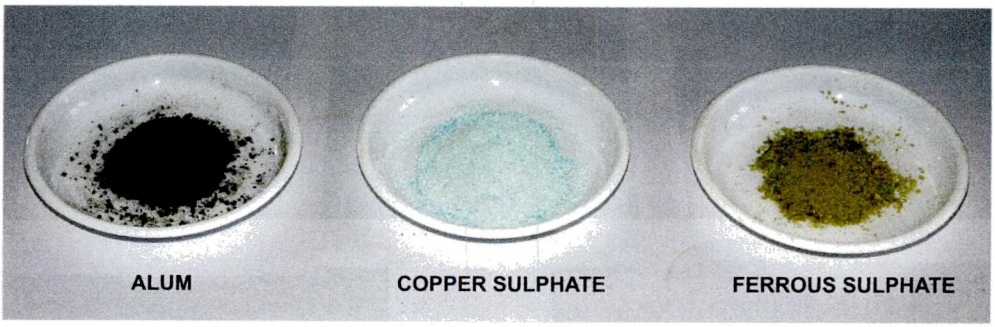
VEMBADAM

PLATE - II
SELECTED DYES

METALLIC MORDANTS



PROCESSED MORDANTS



BIOMORDANTS

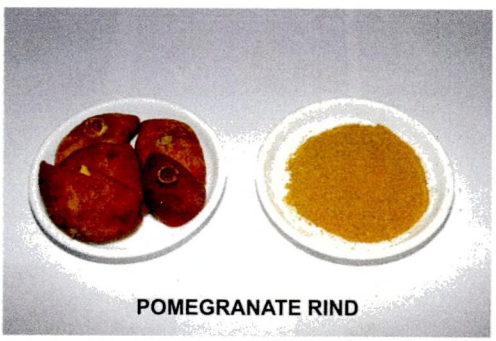


PLATE - III
SELECTED MORDANTS

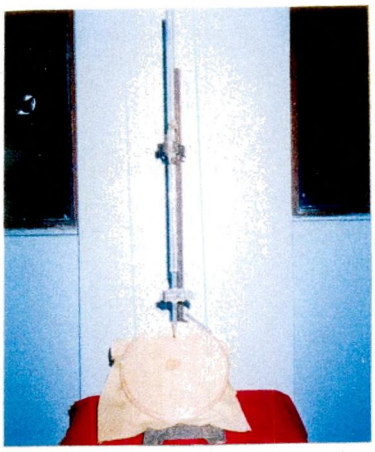


PLATE - IV
DROP TEST

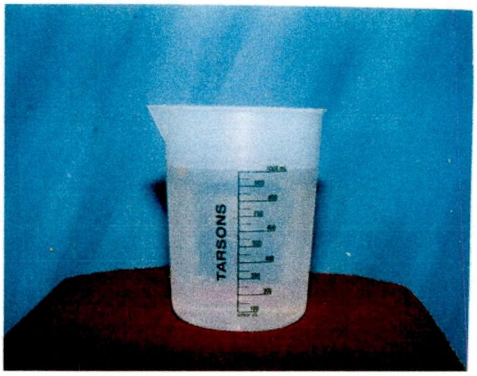


PLATE - V
SINKING TEST

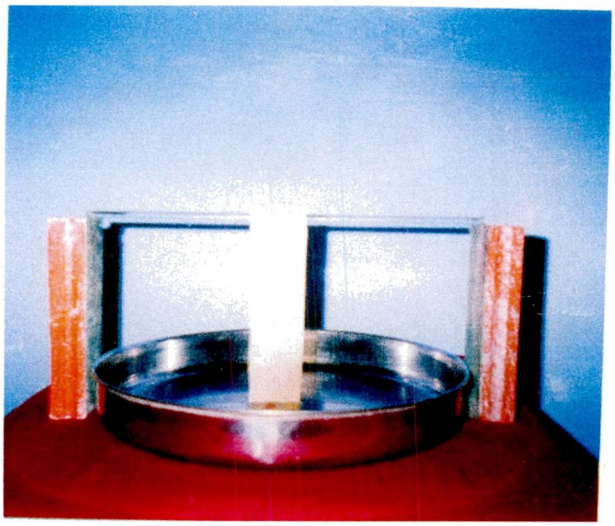


PLATE - VI
CAPILLARY RISE TEST

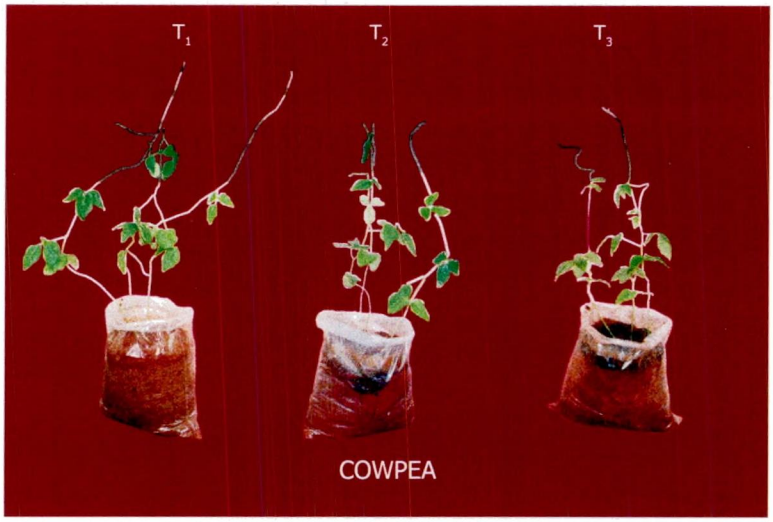
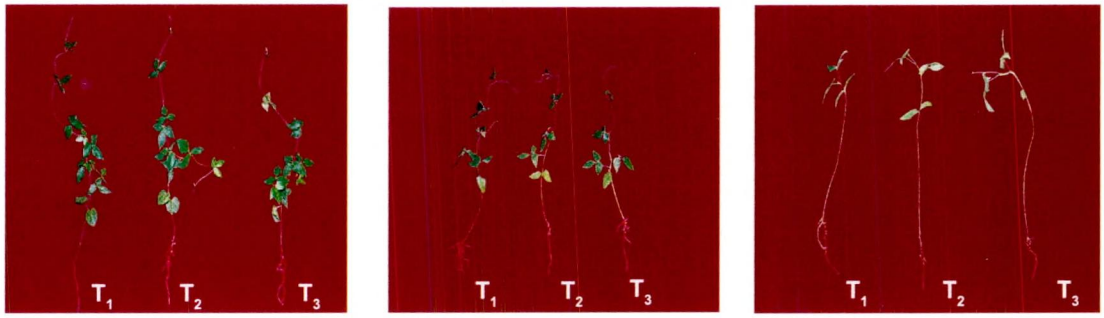
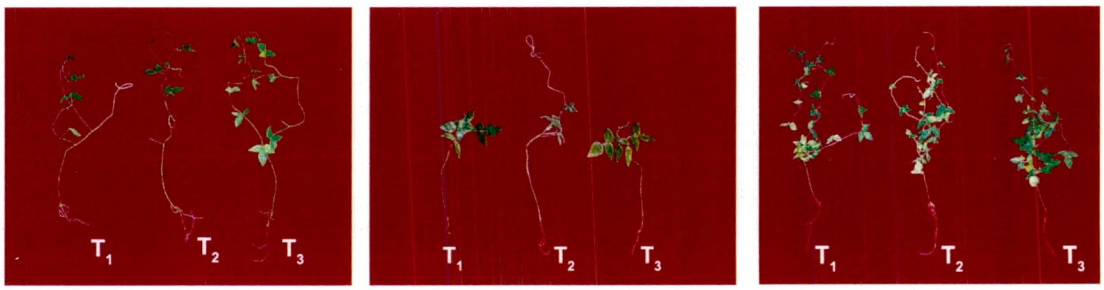


PLATE - VII
OVERALL VIEW OF POT CULTURE STUDY

COWPEA HORSE GRAM GREEN GRAM



30 DAYS



60 DAYS

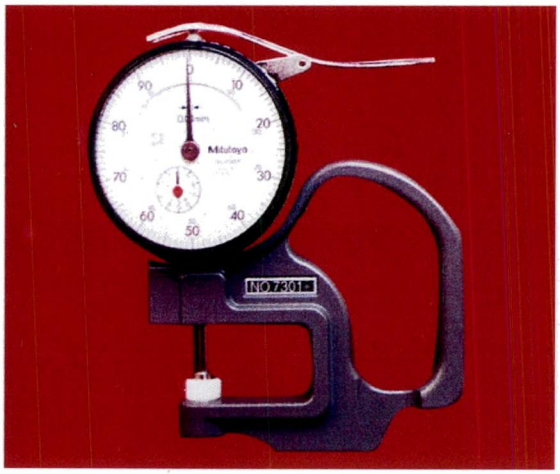
90 DAYS

T_1 - Control Group (Tap water)
T_2 - 50 : 50 (50% Effluent : 50% Tap water)
T_3 - Effluent (100% Effluent)

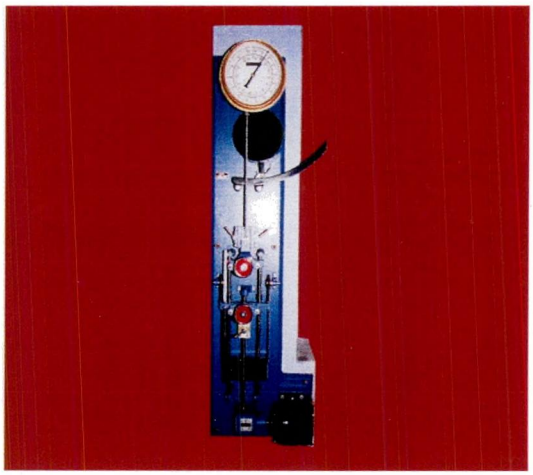
PLATE - VIII
ROOT LENGTH AND SHOOT LENGTH OF COWPEA, HORSE GRAM AND GREEN GRAM

SPECTROPHOTOMETER

ELECTRONIC WEIGHING BALANCE



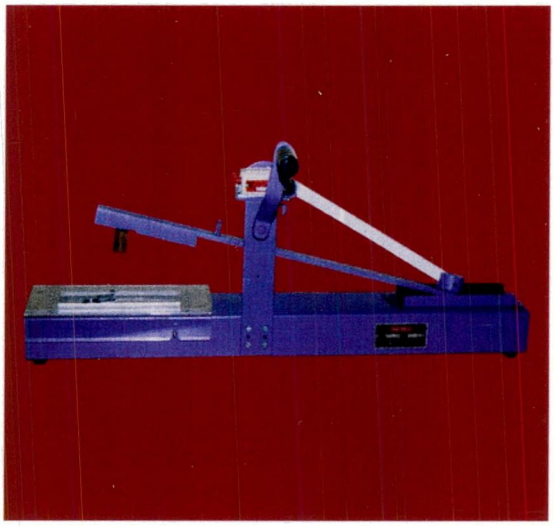
BATTY THICKNESS TESTER



TENSILE STRENGTH TESTER

PLATE - IX
EQUIPMENTS USED

LAUNDROMETER



CROCK METER

COLOUR DIFFERENCE METER (CDM)

pH METER

PLATE - X
EQUIPMENTS USED