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CHAPTER III

EXPERIMENTAL PROCEDURE

The study had an experimental and exploratory format on dyeing of minor protein and cellulosic fibres with natural dyes. The main aim of the study was to develop a colour palette by varying pH, mordants and composite dyeing and then applying the total range of shades obtained through experimentation, onto products made out of minor fibres for use in home décor. The study was also planned to analyze the acceptance of the natural dyed value added products by means of consumer responses. This chapter deals with materials and methods followed for fulfilling the objective of this study.

The experimental procedure of the study undertaken has been subdivided into the following subsections:

3.1. PHASE I: Pilot study on cellulose substrate.

- **3.1.1.** Experimentation with ten natural dyes and four mordants and testing for fastness parameters.
- 3.1.2. Standardization of recipes for extraction
- 3.1.3. Product design, preparation and exhibition

3.2. Sample selection and variables of the study

- 3.2.1. Selection of substrate, dyes and mordants for development of colour palette.
- 3.2.1a. Selection of substrate
- 3.2.1b.Selection of dyes based on colour yield.
- 3.2.1c. Selection of metal mordants based on Eco parameters
- **3.2.2.** Variables used for the study
- 3.2.2.1. Variation in pH: acidic, alkaline and self pH.
- 3.2.2.2. Mordants: Metal and Natural
- 3.2.2.3. Composite dyes: Three combinations of two dyes
- 3.2.2.3a. Recipe for composite dyes

3.2.3. Standardization of recipes.

3.3. PHASE II: Dye application to develop a colour palette.

3.3.1 Application of the natural dyes on Eri silk and Sisal fibers.

3.3.1a. Pretreatment with myrobolan

3.3.1b. Mordanting with metal and natural mordants

3.3.1c. Method of Dye Application

3.3.2. Spectrophotometeric analysis.

3.4. Testing for parameters.

3.4.1. Tests of Optical density of spent dye bath solution.

3.4.2. Testing of fastness properties

3.4.2a. Light fastness

3.4.2b. Rub fastness

3.4.2c. Wash fastness

3.5. PHASE III: Product designing and selection of material

3.5.1. Designing of products from minor fibres.

3.5.1.1. Selection of product design categories

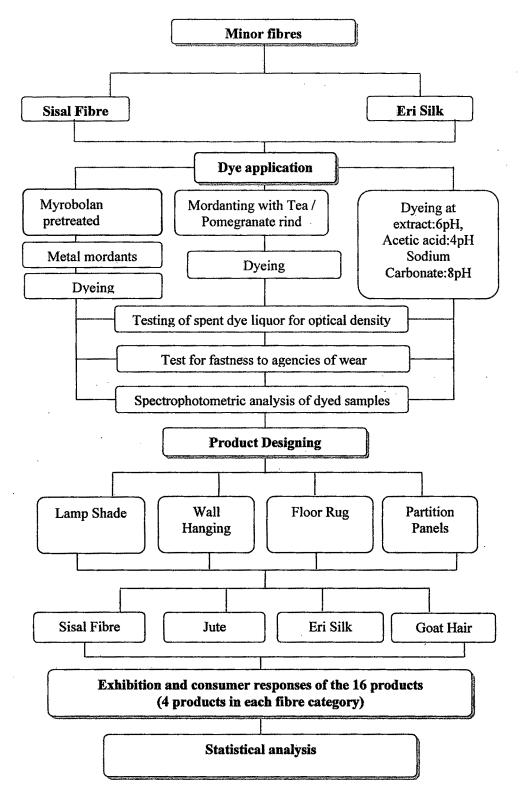
3.5.2. Techniques used for products.

3.5.3. Colour palette for the products.

3.5.4. Preparation of questionnaire.

3.5.5. Exhibition of products.

3.5.6. Analysis of data.



RESEARCH DESIGN

Figure 3.1: Flow chart of Research Design.

3.1. PHASE I: Pilot study on cellulosic substrate:

3.1.1. Experimentation with ten natural dyes and four mordants and testing for fastness parameters.

A pilot study was an essential step for this research since it was exploratory in nature. This was done on a cotton substrate with four metal mordants namely, alum, copper sulphate, potassium dichromate and ferrous sulphate and ten natural dyes used for the pilot study were:

٠	İndian madder	Earthy red
٠	Marigold	Olive green and yellow
٠	Henna	Light greens
•	Flame of forest	Yellow
٠	Ratanjot	Grey
٠	Catechu	Brown
٠	Ravenchi wood	Brown
٠	Red Sandalwood	Red to Maroon
٠	Ferrous Acetate	Black Dye
٠	Lac	Red Dye

The pilot study formed a basis for the selection of dyes and also for the various mordants and their concentration to be used and whether some additional variation was to be worked out. The selection of dyes for the final study was based on the pilot study and hence, was purposive in nature. The dyes were selected on the basis of the colour yield; so that a large colour palette for the minor/underutilized fibre products may be developed.

3.1.2. Standardization of recipes for extraction.

Sr. No.	Dye Source	Obtained as	Procedure of extract	Time and M:L ratio for extraction	Time and M:L ratio for premordanti ng	Time and M:L ratio at
1.	Indian Madder	Root	Powdered and soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	dyeing 45 min. at 1:40 M:L
2.	Marigold	Flower petals	soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
3.	Henna	Leaves .	soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
4.	Flame of forest	Flowers	soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
5.	Ratanjot	Wood	Powdered and soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
6.	Acacia Catechu	Resin	Powdered and soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
7.	Ravenchi wood	wood	Powdered and soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
8.	Red Sandalwoo d	wood	Powdered and soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
9.	Lac	Crystallyze d resin matter	Powdered and soaked for 15 minutes	30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L
10.	Ferrous Acetate	liquid		30 mins. At 1:40 M:L	30 min. at 1:30 M:L	45 min. at 1:40 M:L

Table:3.1 Standardized recipes for extraction of dyes

3.1.3. Product design, preparation and exhibition.

A range of soft furnishing were designed which included cushion covers, wall hangings, lampshade, rugs, dupattas, table mats and coasters. Techniques used for

ornamentation were block printing, stencil printing, screen printing and patchwork. It was observed that there was an overall positive response towards the products displayed during the exhibition. It was also observed that there was greater inclination towards items like lampshade, wall hanging and rug. Hence, this exhibition formed a basis for the category of articles to be designed out of minor fibre products to be value added with natural dyes.

3.2 Sample selection and variables of the study

3.2.1. Selection of substrate, dyes and mordants for development of colour palette.

There is an innumerable variety of natural fibres available across the country. According to Ghosh,G.K ⁽²⁰⁾ developing even some of the fibres for textile use will help meet the textile crisis of future. With the increase in the number of nuclear families and an exponential increase in the textile consumption of the Hospitality and Hotel industry, there is a need to explore the hidden potential of the natural minor fibres available throughout the country. The study aimed to explore value addition of minor fibre by dyeing the products with natural dyes and also to make them cent per cent eco friendly.

3.2.1a. Selection of substrate

There are two major categories of the availability of minor fibres. The protein minor fibres available predominantly from animals reared as cattle; where the fibre is available as wool or hair. Another source of protein fibres are the wild silks available from the non reared silk worms where cocoons are collected from the forests. The cellulose minor fibres are distributed in categories like bast fibre, leaf fibre, nut husk or seed fibre.

Eri silk fabric in the protein minor fibres and Sisal fibres in the cellulose category of minor fibres were selected for development of colour palette with the chosen dye and mordant variables for the study.

3.2.1b.Selection of dyes based on colour yield.

The selection of dyes for this study was purposive in nature. A pilot study was done using cotton as substrate in Phase I of the present study. Ten dyes with metal mordants formed the basis of study and the colour yield of the dyes and their fastness properties formed the basis for the dye selection. Six natural dyes were selected for the study, depending on the colour yield of the dye. Experimental Procedure | 57

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Table: 3.2 Classification of the Natural dyes the study

	Hindi name	English name	Botanical name	Dye	Basic molecule	Dye class	Colour obtained
Natural dyes selected for the	Majith	Indian Madder	Rubia tinctorium L.	Munjistin, purpurin	Anthraquinone	Acid/ Mordant/Disperse	Earthy Red
study	Genda	Marigold	Tagetes petula	Lutein, Zeaxanthin	Carotenoid	Mordant/ Vat	Olive green to Yellow
	Mehendi	Henna	Lawsonia innermis	Lawsone	Alpha- Napthaquinone	Mordant/ Acid	Light Green
	Tesu	Flame of Forest	Butea frondosa	Butein	Flavonoid	Mordant/Disperse	Yellow
	Laljori/Ratanjot	Ratanjot	Onosma echioides	Alkannin	Napthoquinone	Mordant/Disperse	Grey
	Katha	Catechu	Acacia catechu	Catechin	Flavonoid	Acid/Mordant /Disperse	Brown

3.2.1c. Selection of metal mordants based on Eco parameters

"Natural dyes that are not substantive in nature dye a surface by forming metal complexes and thereby there is tendency to use all types of metal salts for the purpose of dyeing and better fastness properties. Restrictions to the use of metal salts have been put by the famous "German Ban". Accordingly, the indicative maximum permissible quantities of different metals in the ultimate product are as follows:

Metal salts of chrome, iron, tin, copper and aluminum are the common mordants generally used. The residual metal mordants which belong to the 'banned' list of chemicals, like chromium and tin remain as residue in the bath and lead to contamination of the substrate. Any permenant or temporary fixed metal salt on the substrate is of great importance when eco-parameters of the metal mordants are inspested. Hence the determination of the residual metal or amount of incorporated metal salts was estimated and it was done by Atomic Absorption Spectroscopy (AAS) at ATIRA for samples treated with copper sulphate and potassium dichromate.

Sample evaluated for heavy metals was fabric for which acidic perspiration solution was prepared. The solution was prepared as follows:

- 0.5 gm of 1-histidine monohydrochloride monohydrate
- 5 gm of sodium chloride
- 2.2 gm of sodium dihydrogen orthophosphate dehydrate, all the ingredients were added to 1 litre of water and brought to pH 5.5 with 1N acetic acid solution

Five gram sample of finely cut fabric was soaked in 100 ml of acidic perspiration solution, which was kept in a water bath for 60 minutes at 40°C. The solution was then filtered and taken for reading on Atomic Absorption Spectrophotometer (AAS). A blank (only acidic perspiration solution) was first taken for the control reading. And then the extracted filterate solutions were run on the AAS for the reading. AAS is a single element method. One element is determined at a time and the instrumental parameters are optimized for the next element and the series is repeated⁽³⁴⁾.

3.2.2. Variables for the study

The investigator had undergone training in the process of natural dyeing at the Weavers Service Centre, Ahmedabad, to get hands on experience of the process. This training process also sensitized the investigator to the traditional practices of natural dyeing. It was observed that most traditional natural dyeing process incorporate the use of metal mordants in dyeing and printing with natural dyes. The use of metal mordants is the only practice followed for variation in the colour obtained and for better dye fastness. Hence, the researcher wanted to inspect methods of extending the colour palette by varying pH or by composite dyeing and by experimenting with natural mordants.

In order to increase the applicability of natural dyes, a larger colour palette would definitely imply more and varied use of the dye. Hence, certain variables were inspected in order to achieve higher applicability. This was done by studying:

3.2.2.1. Variation in pH: acidic alkaline and self pH

Variation of pH was explored in order to assess if there are new or variable shades of dye obtained. The pH was adjusted to 4, 6, and 8 for the samples that were to be dyed at that pH respectively. 4pH was obtained by 1% Acetic Acid, 6pH was the pH of the extract and 8pH was obtained by 1% w/v solution of Sodium carbonate.

3.3.1.2. Mordants: Metal and Natural.

Today, the main thrust aspect in the selection of products is safety. The best product selection must take into account non-toxic and safer starting materials and finished products. Use of alum, copper sulphate, potassium dichromate and ferrous sulphate was proposed, out of these copper sulphate and potassium dichromate were test for permissible residual matter on the substrate. Residual copper was well under the permissible limits of <50ppm and potassium dichromate which exceed the permissible limits with 5% concentration included in the study after lower concentration to 2.5%. Tea and pomegranate rind are rich sources of tannins. Hence, tea and pomegranate rind were chosen as natural mordant to explore their suitability as mordants and also to get different colours.

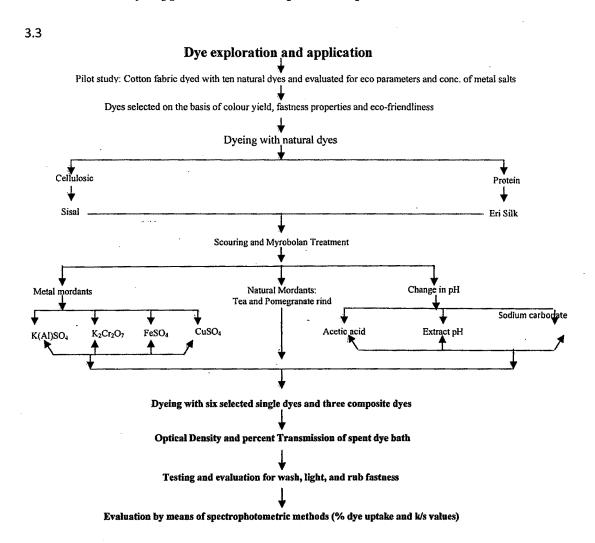
3.2.2.3. Composite dyes: Three combinations of two dyes

Composite dyeing method was adopted in order to explore possibilities of evolving new shades and consequently a large colour palette. Two dyes of a similar chemical composition or nature but giving different colours were combined in order to make a composite shade. The dyes that had larger colour variation and similarity of behavior with various pretreatments were combined; for composite dyeing. Hence, three combinations were worked out. They were:

Majith and Ratanjot	Madder and Ratanjot	Earthy red + Grey
Majith and Tesu	Madder and Flame of forest	Earthy red + Yellow
Majith and Genda	Madder and Marigold	Earthy red + Olive
~		green + yellow

3.2.2.3a. Recipe for composite dyes

Each of these dyes were weighed individually and kept for aqueous extraction separately. Concentration of madder, ratanjot was 2% and that of marigold and tesu flowers was 4%. After 30 minutes of extraction the dye solution was filtered and kept aside for cooling and reuse later on. The extracts were taken in 1:1 proportion according to the material liquor ratio and the dyeing was carried out.



3.3. PHASE II: Dye application to develop a colour palette.

Figure 3.2: Flow chart of dye exploration and application.

3.3.1. Application of the natural dyes on Eri Silk and Sisal Fibre.

Dyeing with natural dyes requires a systemic investigation into the process of building up of colour on the fabric. Hence, it will be observed that unlike synthetic dyes; where colour in applied in one or two steps, in natural dyes the colour builds up in a series of steps starting from the preparation of the fabric to final output. Eri silk and sisal fibre were chosen for the development of a colour palette. The fabrics were first scoured and then taken for pretreatment and dyeing.

Preparation of selected substrate

Scouring:

Any material to be dyed should be clean before dyeing. The scouring of Eri silk was done with hot water at 65°C and liquid soap. Eri silk was scoured to remove the natural gummy substance *sericin* so that the surface of the fabric is in good condition to receive the dye.

Scouring of Sisal fibre was scoured with 2% soap and 2% soda ash at 85 °C with a material liquor ration of 1:40. The scouring procedure helps to make the fibre lustrous but slightly pale in appearance. This yellowness disappears and a light creamy white coloured fibre is obtained on drying.

The sequential procedure of dyeing with natural dyes is as follows:

3.3.1a. Myrobolan Treatment:

A pretreatment with myrobolan was done taking 4% myrobolan powder on the liquor. The material liquor ratio was kept as 1:30. Myrobolan powder was weighed according to the proportions with respect to the weight of the fabric and liquor and then the powder was soaked for 15 minutes in the required quantity of water. The water was then filtered and this solution was used to give the myrobolan treatment to the scoured fabric.

3.3.1b. Mordanted with metal mordants / natural mordants:

It was observed from the AAS study that residual Chorme mordant on the fabric exceeded the permissible limits of 2.0ppm. Hence, various lowered concentrations of chrome mordant were worked out.

	•
i. Alum K(AI)SO ₄ :	10% on the weight of material
ii. Copper sulphate CuSO ₄ :	5% on the weight of material
iii. Potassium dichromate K ₂ Cr ₂ O ₇ :	2.5% on the weight of material
iv. Ferrous sulphate FeSO ₄ :	1% on the weight of material
v. Natural mordant: Tea:	5% on the weight of material
vi. Natural mordant: Pomegranate rind:	5% on the weight of material

Myrobolan pretreated fabrics were taken and then cut into samples as required for the metal premordanting. The fabric weight was taken and the mordants were calculated according to their respective percentages and fabric weight. The treatment was given at 85°C for 30 minutes.

A natural mordant treatment was given to another set of fabric at the second step; i.e. at the metal mordant stage. The samples were treated with 5% tea extract or 5% pomegranate rind extract in order to assess the dyeing properties of the dyes and the fastness properties were also assessed. This was done in order to test if the natural mordant could be used instead of the metal mordant. In all 10 pretreatments were worked out they were:

Sr. No.	Treatment	Code for Eri Silk Samples	Code for Sisal Fibre Samples
1.	Dyed only with dye.	ED	SD
2.	Myrobolan pretreated and mordanted with Alum.	EMAD	SMAD
3.	Myrobolan pretreated and mordanted with Copper sulphate.	EMCuD	SMCuD
4.	Myrobolan pretreated and mordanted with Potassium dichromate	EMCrD	SMCrD
5.	Myrobolan pretreated and mordanted with Ferrous sulphate	EMFeD	SMFeD
6.	Myrobolan pretreated and dyed at 4 pH.	EMD _{4pH}	SMD _{4pH}
7.	Myrobolan pretreated and dyed at 6 pH.	EMD _{6pH}	SMD _{6pH}
8.	Myrobolan pretreated and dyed at 8 pH.	EMD _{8pH}	SMD _{8pH}
9.	Myrobolan pretreated and mordanted with Tea as a mordant.	EMTD	SMTD
10.	Myrobolan pretreated and mordanted with Pomegranate rind as a mordant.	EMPRD	SMPRD

Table 3.3: List of different treatments of the study

Where: E = Eri, S = Sisal, M = pretreated with Myrobolan, A = mordanted with Alum, Cu = mordant with Coppersulphate, Cr = Mordanted with Potassium dichromate, Fe = mordanted with Ferrous sulphate, T = premordanted with Tea, PR = premordanted with pomegranate rind and D = Dyed at extract pH.

The material liquor ratio for myrobolan treatment was 1:30 for 30 minutes at room temperature. The samples were then squeezed and dried and the excess myrobolan powder dusted out by gently hitting the fabric on a wall. The metal mordant and natural mordant treatment was given for 30 minutes at 85°C. The fabric after the mordant bath was entered into the dye liquor in the wet state.

3.3.1c. Method of Dye Application with natural dyes:

A total of six natural dyes were selected for the study based on the colour obtained through the dye and their fastness properties on cotton substrate. The selection of six natural dyes from the pilot study was hence; purposive in nature. Three composite dye mixtures were also experimented. Hence, a total of nine dyes were selected for the study. Dyeing at extract pH, acidic and alkaline pH and use of tea and pomegranate rind as natural mordant were the five factors which contributed to a total of 10 pretreatments worked for Sisal and Eri silk fabric. Thus, 90 shades per substrate were developed out of the dye, mordant, fibre and pH combinations explored in the study.

The extracted dye was applied on minor fibres/minor fibre fabrics. The application of natural dyes was done by premordanting technique and standardized extract of natural dyes. This was essential to obtain reproducibility. The dye was calculated on the amount of liquor required for dyeing. It is taken as 2% in case of root, wood and barks dye source and 4% in case of leaves and petals of flowers; as the roots, bark and wood yield more colour than the petals. The material liquor ratio was 1:40. The dye source powder was weighed according to the weight of the material and the powder was steeped in water according to the liquor ratio and the dye was extracted for 30 minutes at boil. The liquor was maintained by occasionally adding distilled water to the extract in measured quantities. The extract was then allowed to cool and filtered. This liquor was again adjusted to the measured liquor ratio and the extract was taken for spectrophotometric analysis; where the optical density of the extract was recorded.

The dye application was carried out my means of exhaust dyeing method. The extract was distributed in 10 beakers according to the material liquor ratio for the fabric to be dyed. The samples according to the treatment they were to be given were put into

the respective beakers. The pH was adjusted to 4, 6, and 8 for the samples that were to be dyed at that pH respectively. 4pH was obtained by Acetic Acid, 6pH was the pH of the extract and 8pH was obtained by Sodium Carbonate.

The material liquor ratio for extraction of dyes was 1:40 at boil for 30 minutes. Dyeing took place at 85°C, at 1:40 M:L ratio for 45 minutes.

3.3.2. Spectrophotometer analysis.

Spectrophotometer analysis was done by using Premier 5100 Spectrophotometer to measure the k/s and C.I.E. L*a*b* values of the dyed samples. D65 illuminate having a colour temperature of 65000k equivalent to average day light and 10° visual angle were selected to obtain the values. The k/s values of the dyed samples were measured at three different points across the visual spectrum (400-700nm). The percent transmission curve of control and dyes with six individual dyes and three composite dyes used in the study were obtained. The wavelength at which the maximum transmission value indicate the colour present in the dyes. Thus every dye had its own percent transmission curves. The L* a* b* show the values of samples in comparison to the standard sample. The reflectance curves of control and dyed samples with each dye used in the study were obtained. The values DE* gave (maximum colour change), k/s gave (strength of colour), DL* indicated (lightness of the colour) and a* and b* values how redder or bluer a colour was.

3.4. Testing for parameters.

3.4.1. Tests of Optical density of spent dye bath solution.

Traditional recipe and past researchers have employed the use of metal mordant in order to dye a substrate with natural dyes. It was mostly established from the past experience that metal mordants lend a certain colour and also contributed positively towards fastness properties to the dye. All these factors worked as constant parameters for the present study and change in pH and natural mordants were the variables of the present study. It was hence important to establish a relation between the colour obtained from a particular dye, the optical density and pH Transmission of the dye solution and the dye behaviour at different pH. Hence, extract of the dyes were used to establish the characteristic wavelength of the dye and optical density of the spent dye bath and its percent transmission were recorded in order to study the amount of exhaustion of the dye.

This instrument comprised of a monochromator to supply monochromatic light and a photometer to detect it. The method is to note on photometric scale each wavelength in turn of intensities of incident and transmitted light from specimen. Thus, the spectrophotometer measured the reflectance of spent dye sample separately at each wavelength, for colour measurement visible spectral region from 300nm to 720 nm at an interval of 20 nm. The light source in the spectrophotometer is a low voltage tungsten lamp. Distilled water was filled in one cuvette and the rest of the cuvettes held spent dye liquor from the dyeing experiments filtered through Watsons filter paper. The optical density is adjusted to zero with the help of the water cuvette and transmission readings of the coloured solutions at zero optical density for a range of light spectrum from 400nm – 720nm at an interval of 20nm. Readings indicate that when the incident light and the colour of the dye solution is of the same colour then we get a point of maximum transmittance which was called the λ maxima of the dye. This indicated the wavelength of the dye solution and hence the colour in the visible spectrum. After obtaining the λ maxima from the extracted dye solution the spent dye baths were read at that wavelength and the optical density versus transmittance were recorded across the visible spectrum for spent dye liquor after dyeing at 4pH, 6pH, 8pH, tea-premordanted and pomegranate rind premordanted samples. Percent dye absorption was computed out of each of these readings.

3.4.2. Testing for Fastness properties.

The dyed samples were evaluated for the following fastness properties:

3.4.2a. Light Fastness : Test method IS 2454:1985 based on ISO 105/B-1984. (34)

The dyed samples were tested for their fastness to light. A fade-o-meter was used to carry out the testing. The testing was carried out by exposing the dyed samples for 10 and 20 hours and the fading under light emitted from calibrated carbon-arc lamp. The geometric grey scale by ICI (as specified by the Society of Dyers and Colorists) was used for visual assessment to evaluate the degree of fading of dyed samples after 10 and 20 hours of exposure to light.

3.4.2b. Rubbing/crocking fastness: Test method IS 766: 1988 based on ISO 105/X-1984.⁽³⁴⁾

To assess the rub fastness (dry and wet) of the dyed samples a Crock-o-meter was used. The crock-o-meter has one of two alternative sizes of rubbing finger, depending upon the type of textile to be tested as follows. The rubbing finger should comprise of a cylinder of 1.6cm diameter moving to and fro in a straight line along a 10.0 cm track on the specimen with a downward force of 9N.

The samples sized 14cm x 5 cm, were cut and mounted on the crock meter. The experiment was conducted by the dry and wet rubbing of scoured white cotton fabric with 10 strokes in 10 seconds with a force of 9Newton. For wet crocking fastness test, scoured white cotton fabric was dipped into distilled water and the wet pickup was adjusted to 100%. Care was taken to maintain the moisture of the wet rub fastness fabric. The dry white cotton fabric was mounted first on one of the fingers of the rubbing arm of the crock-o-meter and then the wet white was mounted and the test strokes were immediately taken. These samples were evaluated for staining on white.

The geometric grey scale by ICI (as specified by the Society of Dyers and Colorists) was used for visual assessment to evaluate the degree of crocking by comparing the difference in colour of stained and unstained cloth with the difference represented by the scale.

3.4.2c. Laundering/washing fastness: Test method IS 764:1979, based on ISO, 105/C-1982⁽³⁴⁾

To evaluate the wash fastness of the dyed sample a launder-o-meter was used. A mechanical washing device with a water-bath containing a rotor with which containers of glass or of stainless steel of 500 ml capacity are rotated at speed of 40 ± 2 revolution per minute and a thermostat to control the temperature of water bath so as to maintain the

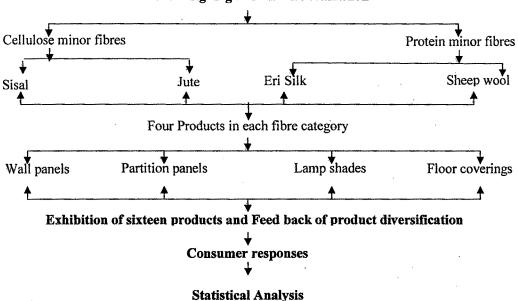
temperature of test solution in the containers at 60 ± 2 °C. This instrument works on the principle that samples washed under suitable condition of temperature, alkalinity and abrasive action such that the desired loss of colour is obtained in a short time.

The samples sized $10 \text{cm} \times 4 \text{cm}$ were first sandwiched between two layers scoured white cotton fabric by means of hand stitching and the combined weight of this sample was taken for the material liquor ratio. 2 gms/litre of liquid soap was taken to make the soap solution and it was heated to 60° C. The samples were then put along with the soap solution into the jars of the launder-o-meter. A 45 minute wash cycle was executed. The samples were then taken out, rinsed dried and then evaluated for change in colour and staining on white.

The geometric grey scale by ICI (As specified by the Society of Dyers and Colorists) was used for visual assessment to evaluate the rate of staining and colour change of the dyed sample. For the evaluation of the rate of staining of un-dyed cloth, was done by comparing the difference in colour of stained and unstained cloth with the difference represented by the scale. For the evaluation of the change in colour, the rating was done by comparing the difference in colour of tested specimen and the original textile with the difference represented by the scale.

3.5. PHASE III: Product designing and selection of material

An important phase in order to implement the colours obtained from the various experiments done to obtain a large colour palette. Consumer responses were elicited in the form of a structured questionnaire administered to the respondents who were invited to the exhibition of the products designed. The flow of work in this phase was as follows:



Product designing and market evaluation

Figure 3.3: Flow chart of product designing and market evaluation.

3.5.1. Designing of products from minor fibres.

3.5.1.1. Selection of product design categories

It was observed from the pilot study that great interest was generated by the products displayed for home décor. Especially the category lampshade, wall hanging and floor rug made out of jute and mulberry silk fibres received great appreciation. Hence, it was concluded that minor fibre products would be designed for utilization in home décor.

Design ideology: By definition minor fibres in textiles mean those fibres that are limited in production and also not available on the economies of scale of a mass market. The fibres have a limited production and application. This research aimed to utilize the exclusivity factor of these minor fibres and also diversify the form of their usage in order to add value and make them applicable for niche market consumption.

Design concept: Four minor fibres chosen for the study. Two minor fibre in cellulose were Sisal fibre and Jute fabric and Eri silk fabric and Patanwadi goat hair were the two

selected fibres in protein category. It was observed from the review of literature that most of these fibres were utilized in a limited capacity for conventional usage of cordage material or structural material for handicrafts of local regions of produce. But the aim of this research was to explore the potential of minor fibres in order to make them suitable for use as home textile material. It was also observed that most of the fibres in this category have a harsh tactile quality. It was kept in mind that the products designed for use should not be in constant contact of the skin of the user. Hence, the product design categories were:

- Lampshades
- Partition panels
- Wall hangings and
- Floor coverings
- **3.5.2.** Techniques used for products.

A total of four minor/underutilized fibres were selected to design a co-ordinated product range for home décor out of each fibre.

Two cellulose fibres selected were Jute, which was obtained from Champdany Jute mills, Kolkatta, and Sisal in fibre collected by the efforts of Mr. Girimath from the villages surrounding Bangalore. Experiments were carried out to hand spin the fibre and generate a yarn. Methods like end-to-end joining, twisting, wet twisting were tried out by the researcher and a test fabric length in plain weave was generated by the researcher. This knowledge was then shared with rehabilitation patients at Sevasi village, Baroda. In the protein fibre category two fibres were sourced. Eri silk fabric was obtained from Ribhoi, Assam, through Dr. Dingdoh and Goat Hair fibre was obtained from Vankar VishramValji of the Bhujodi village, in Gujarat.

A wide variety of techniques were employed for the designing of products. Care was taken that each product would have some variation in techniques so as to imply that many variations were possible with these fibres. Techniques with none/low use of machines were employed, so that they could be easily learnt or practiced by an artisan with very little investment and with their existing skill set (as most of the traditional crafts in India particularly deal with skills like embroidery, appliqué, quilting, couching, hand block printing, etc). Direct application of print was done by means of stencil and block printing methods. Several variants in block printing were also tried out. The first five techniques detail the use of direct method of print application i.e. stencil printing and block printing. Resist printing techniques were also employed for surface ornamentation of some products. Batik and tie and dye were employed. Two variations; pleating and hand tritik and pleating and twisting were used for the products. Surface ornamentation by means of using embroidery was also done in the form of machine and hand patchwork, metal embroidery, white on white embroidery, braiding and couching and draw-thread methods. Non-woven mesh making technique and tapestry weaving were also employed. Details of the techniques used for the designing of products were as follows:

Technique 1: Stencil and Block printing was combined for printing the Jute rug. The bold outline was done by using stencil cut outs and application of mordants was done through the stencil. Central rectangular patch was developed by applying alum metal mordant by means of a brush and a border print was made by means of block printing. The size of the jute floor rug was 3.5 feet by 5.0 feet. Ratanjot dye was used for colour development on the borders and the central piece was dyed in tea pretreated flame of forest dye.

Technique 2: An all over print surface was developed by means of block printing for the partition panels made out of eri silk fabric. Different blocks of similar square layout were chosen and were arranged in regular or checker board pattern and printing was done with the help of metal mordants and ferrous acetate solution. The samples were allowed to dry and then washed in a tank filled with water. Then each face of the samples was dyed with four different dyes. The dyes used were Flame of Forest, Marigold, Henna and Madder. Two fabrics were stitched together to form the front and back face of the panels and ring rivets were driven on the edges of each panel. The panels were then mounted on a wooden frame of size $6' \times 2'$, two such panels were made.

Technique 3: Spot prints were created on the centre of a jute panel of size 1.5' x 1'; six double sided panels were made. Each of them was pretreated with alum as a mordant and colour was developed with the six individual dyes selected for the study. These paired samples were stitched together leaving an opening at the top and bottom for the samples to slide into a rod in the frame. The partition panel frame size was six feet by four feet.

Technique 4: Eri silk lamp shade had a structure that was organic in nature. A placement print technique was used for imparting print to the surface of the lamp made out of eri silk fabric. A border motif was selected and the print was imparted in such a way that the creeper would run along the curved edges of the lampshade. The outline was printed with ferrous acetate solution and mordants were filled in the leaves and flowers of the creeper.

Technique 5: An experimental method of block printing was chosen for printing of wall hanging made out of Eri silk fabric. Blocks of assorted shapes and sizes were arranged in a way that it would make a composition out of the print. Brush was used where necessary and the printing was done. The sample was developed with madder dye.

Technique 6: Sisal rug was ornamented by batik technique. The paraffin was to bees wax ration was taken as $60:40^{(21)}$ as it was the optimum ratio for batik using natural dyes. A fish motif with chevron pattern was selected and the wax application was done according to the print desired. Colours were developed by dyeing with Marigold at 4pH for the fishes, Acacia Catechu with Copper sulphate for the chevron pattern and at 8pH for border, edge finishing with bias binding done with cotton fabric dyed with ferrous acetate. The size of the rug was 3' x 4'.

Technique 7: Tie and Dye method with pleating and hand tritik technique was employed for creating a wall hanging. Alum pretreated jute fabric was dyed with Ratanjot dye. The bottom of the wall hanging was created by pleating and tying, where 2.5 inch pleat fold were made in the vertical and horizontal directions and then tied by means of a cotton thread. The top of the wall hanging was created by three rows of hand tritik to form a circular motif and also three rows of hand tritik for the two straight lines. Edge finish of the wall hanging was done by brocade fabric.

Technique 8: Accordion pleats of size 1.5 inch were taken throughout the length of the wool fabric. The pleated strip was then highly twisted in order to form a ball and held in place by cotton threads. Two such panels were created and dyed in different dyes. An advantage of this technique is that the front and reverse sides of the fabrics were identical in nature and hence only one layer of fabric was needed for the design of partition panel.

Technique 9: Patch work and white on white embroidery were used for the designing of floor rug made out of Eri silk fabric. Multi-coloured dyed patches were sewn together by a simple sewing machine and the central yellow plain fabric was dyed with Flame of Forest dye with myrobolan mordant. White on white embroidery was used on un-dyed Eri silk fabric and the outer border was dyed with madder dye and potassium dichromate mordant. The size of the rug was 4' x 3.25'.

Technique 10: Wall hanging made out of sisal fibres was done using embroidery machine. Where patches of fabrics were cut out and stitched with a broad zigzag setting. The thread used for the patchwork was white in colour and the back ground fabric was dyed with Madder and Ratanjot dye mixture and the fore ground had mixed colour in Ratanjot and Acacia Catechu. The size of the wall hanging was 2' x 2'.

Technique 11: The wall hanging made out of Goat Hair was designed in such way that metal (*zardozi*) embroidered white goat hair fabric was as an accent to the subdued natural dyed patches of wool dyed with Ratanjot and Madder dye. In order to add an element of interest natural dye printed strips of cotton were matted and a part of the frame was created in order to impart visual interest to the frame. Care was taken that the cotton fibre material would contribute less than 25% of the total material share of the article made. Ratanjot Dye at neutral pH, Madder dyed with Alum, natural dye printed cotton strips of mixed dyed like madder, Flame of forest, Ratanjot, etc, were used. 3' x 1.75' was the size of the wall hanging.

Technique 12: Braiding and couching method was employed to design a lampshade out of sisal fibre. Base fabric was dyed with Marigold dye at 6pH, braids were made out of mixed palette from all the dyeing experiments and they were couched on to a base with golden thread. By this method the whole colour palette developed on sisal fibre appeared

on the surface of the lamp and it lends a colourful look to the lamp shade during day time when there is ample natural light falling on the surface and when the lamp is lit from within then the surface shows up an interesting dark patterning against light background.

Technique 13: Most of the wool fabric available had a chambray like weaving pattern with alternating black and white yarn in weaving. Hence surface ornamentation by print method was avoided since the print outlines would not show clearly. A draw thread method of ornamentation was used in order to create a play of light from the lampshade when it is lit from within. The fabric was dyed with tea pretreated madder dye.

Technique 14: In this technique a textured surface was created by means of stitching pleats in alternate directions, this method is called rippling. Acacia catechu dye was used for dyeing one panel at 6pH. Two other panels were pretreated with copper sulphate and ferrous sulphate and the dyed with Acacia Catechu.

Technique 15: Multi-coloured sisal fibre web simulating a non-woven fabric was laid with all the experimental colours. The web was then sandwiched between two glass sheets of 6' x 2' size, this was then mounted on a wooden base and the top edges were clamped, two such panels were made.

Technique 16: Goat hair is available in abundance in its natural colour black and in limited quantities in white. Hence, it was kept in mind that maximum use of the naturally available colour was to be made with value addition of the white fibres with natural dyes. Hence the Floor Rug was woven in the "Kharad" (Kutch style door mat), with a dominant use of natural black fibre and with fibres dyed with madder dye. The rug was dyed with Madder with Myrobolan as pretreatment.

3.5.3. Colour palette for the products.

The chief design concern during the designing of the products from minor fibres was also that the colour palette that was experimented in the earlier stage was to be incorporated into the final designed products and also that the whole shade card is depicted into the products. Hence, the products were designed using multiple techniques of surface ornamentation; ranging from dyed patches to block printed to embroidered samples. Results of the fastness properties of the dyes were also kept in mind while selecting a particular dye fibre combination and their fastness properties. The specification sheets of the designed products are discussed in the results chapter.

3.5.4. Preparation of questionnaire.

A questionnaire was prepared keeping in mind the components of concept testing. The questionnaire consisted of a mix of open and close ended statements and also some statements that elicited multiple responses. It was validated by five experts of the product design department and then it was pretested taking a sample of 20 students. The products obtained from the study belonged to an underutilized category of fibres and hence the products had a relatively new appearance i.e. it was assumed that the respondents had almost none or very little past reference to a previous product they might have used. Hence, it was important to first establish whether the respondents were aware of the minor fibres, and also if they were aware of the environmental concerns of present times. It is also understood that awareness would lead to a need and in relation to the need of a particular product there would be a perceived gap in supply and perceived value of the product. Purchase intention and awareness about non-availability of a particular type of product would also serve as having a positive co-relation to the need, liking and acceptance of the products. Hence, the questionnaire was made keeping the above parameters as the key points of study and a mix of positive and negative statements were formulated. The final questionnaire was administered to the respondents during the exhibition of natural dyed products design with minor fibres.

3.5.5. Exhibition of products.

Market evaluation of natural dye value added minor fibre products, through consumer responses elicited by a structured questionnaire. A **target market** in marketing is the market segment to which a particular product is marketed. It's often defined by age, gender, geography, and/or socio-economic grouping.

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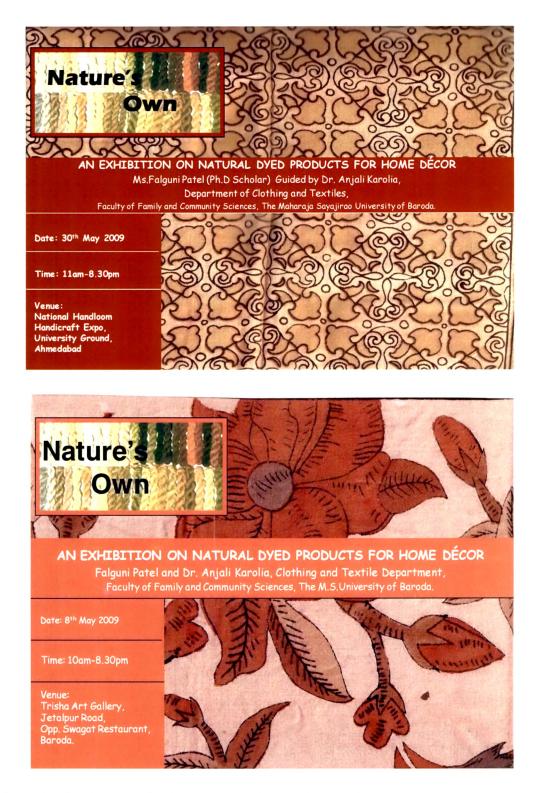


Plate 3.1: Invitation cards designed for the promotion of exhibition

A **niche market** is a focused, target able portion (subset) of a market sector. By definition, then, a business that focuses on a niche market is addressing a need for a product or service that is not being addressed by mainstream providers. A niche market may be thought of as a narrowly defined group of potential customers. A distinct niche market usually evolves out of a market niche, where potential demand is not met by any supply.⁽⁸⁴⁾

Suitability of the products to a market and its acceptance by the target consumer group would lend leverage to the natural dye value added minor fibre products. Exhibitions of the products in the cities of Baroda and Ahmedabad were planned to showcase the products to a large population. The exhibitions were advertised by putting a note in the news paper, also by sending invitation cards to acquaintances and also to regular gallery visitors (650) and by promotion through short message services (2000 sms through mobile networks). A banner titled "Nature's Own" was designed and put up at the venue for visibility to a passer by and on entering the venue of exhibition a poster informing about the natural dyes and minor fibres was kept for the visitor's benefit. Also, the shade card of 180 shades 90 in each fibre category i.e. Eri silk and Sisal fibre was kept at the venue. Samples of the four minor fibre fabrics (untreated/original form) used for the designing of the products were also kept.

A brief introduction to the work was given to the visitor or groups of visitors they were then lead to the products. A questionnaire was administered to the visiting respondents. The questionnaire included multiple choice statements and also statements consisting of responses according to five point Likert-type scales where the respondents were asked to state their responses under categories like strongly agree, agree, neutral, disagree and strongly disagree. Some of the statements also elicited close ended positive and negative responses. These responses were recorded to generate a raw data which was further subjected to correlation analysis.

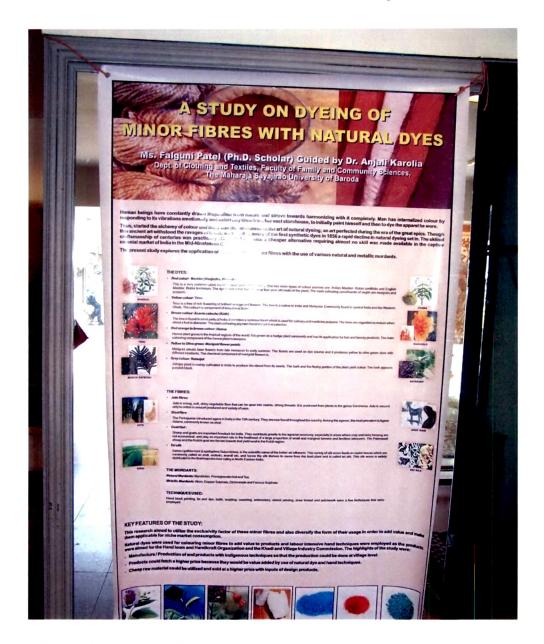


Plate 3.2: Design of poster exhibited at the venues

Two exhibitions were held, the first one at the Trisha Art Gallery, Baroda. A second exhibition was held at National Handloom and Handicraft Fair at University Ground, Ahmedabad. A total of 16 products 4 belonging to each fibre category; value added by means of natural dyeing were displayed at the exhibition. Out of the total

promotional activities a random sample of 500 respondents was expected by the researcher. Total 560 questionnaires were administered and 485 complete responses were obtained. Hence, the sample for the present study is 485 respondents.



Plate 3.3: Shade card displayed at the exhibition venues for the respondants

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Plate 3.4: Photographs of exhibitions held at Trisha Art Gallery, Baroda and Handloom Fair at University Ground, Ahmedabad.

3.5.6. Statistical analysis of data.

It was of importance to examine the *relationship* of one variable of the tool made, to another than to just measure the preference of products by individual or a group alone. The concept testing tool that was made for this study set to answer certain questions like:

• Does that awareness towards natural dye and eco friendly products imply that there is a need generated for this category of products?

- Was there a relationship between the product need level and the gap in availability of the products?
- Likewise, it was also necessary to probe whether there was a correlation was also studied between products need and perceived value of the products, perceived value versus purchase intention and also between purchase intention and availability of the products through handloom and handicraft outlets?

The collected responses were coded in the form of frequency scores on a coding sheet. The responses were summed and multiplied with the weights assigned according to the nature of each statement. Analysis of the data was done by Pearson's correlation analysis method. Correlation coefficients were computed between categories like awareness towards natural dye and eco friendly products with the need level for the products. Correlation was also studied between product need level and gap in availability of the products, need and perceived value of the products, perceived value versus purchase intention and also between purchase intention and availability of the products through KVIC outlets. "A coefficient of correlation falling between .00 and 1.00 always implies some degree of positive association, the degree of correspondence depending upon the size of the coefficient." Coefficients of correlation are indices ranging over a scale which extends from -1.00 through .00 to 1.00. A positive correlation indicates that large amounts of the one variable tend to accompany large amounts of the other; a negative correlation indicates that *small* amounts of the one variable tend to accompany large amounts of the other. A zero correlation indicates no consistent relationship. The results of the product testing are evaluated in the results and discussion chapter.⁽¹⁸⁾