CHAPTER III

MATERIAL AND METHODS

This was an experimental study on the effects of pre-treatments for the dyeing behavior with natural dyes on jute. The main aim of the study was aimed to develop relationship between effect of pre-treatments and the absorption of jute fabrics.

This chapter deals with materials and methods followed for fulfilling the objective of this study.

The experimental procedure has been explained under the following subtitles:

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3.2 Specification of the fabric

3.3 Preparation of fabric

3.4 Phase I: Pilot work

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3.4.2 Testing of parameters affecting colouration

3.4.2.1 Determination of wash fastness of dyed fabric

3.4.2.2 Determination of light fastness of dyed fabric

3.4.2.3 Determination of crock fastness of dyed fabric

3.4.2.4 Evaluation of degree of color yield

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3.5.4 Testing of Optical Indices

3.5.5 Testing of Fastness Properties

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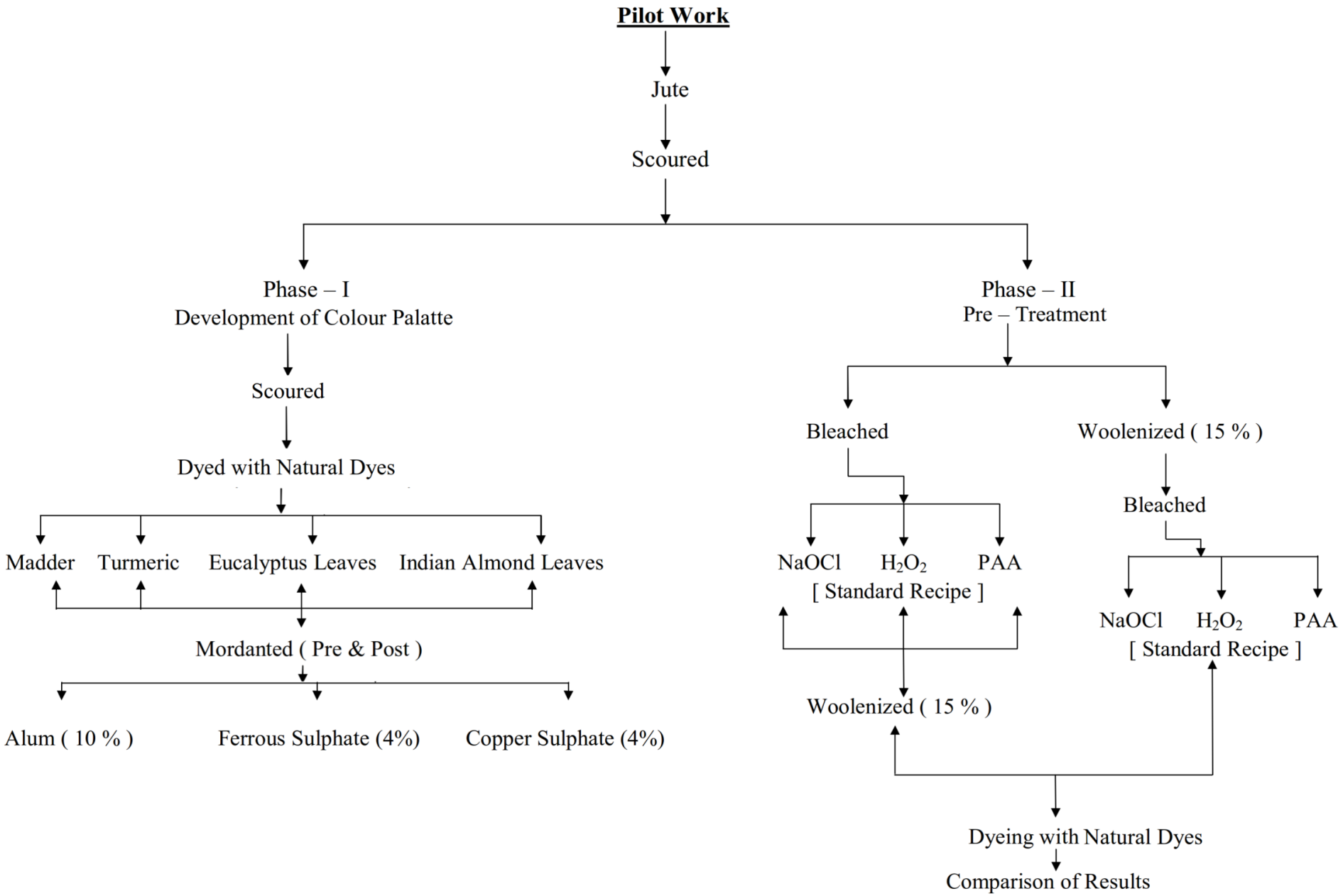

Pilot Work
Jute
Scoured
Phase – I
Development of Colour Palatte
Scoured
Dyed with Natural Dyes
Madder
Turmeric
Eucalyptus Leaves
Indian Almond Leaves
Mordanted (Pre & Post)
Alum (10 %)
Ferrous Sulphate (4%)
Copper Sulphate (4%)
Phase – II
Pre – Treatment
Bleached
Woolenized (15 %)
NaOCl
H_2O_2
PAA
[Standard Recipe]
Bleached
NaOCl
H_2O_2
PAA
[Standard Recipe]
Woolenized (15 %)
Dyeing with Natural Dyes
Comparison of Results

3.1 Selection of fabric

For the study, 100% jute fabric was used. Grey fabric obtained from Reliance Jute Mills (International Ltd.), Kolkata, West Bengal was used for pilot work. For final work fabric was procured from Department of Jute and Fibre Technology, Kolkata, West Bengal.

3.2 Preliminary data of fabrics

3.2.1 Determination of Fabric Count:

The fabric count was determined by counting the number of threads per inch in warp and weft direction using pick glass. An average of five readings were taken randomly and represented as number of thread per inch in warp and weft direction.

3.2.2 Determination of weight per unit area:

The samples of 5cms × 5cms were cut. Care was taken to keep samples free from any kind of fabric irregularity such as knots and distortions. The samples were then conditioned for 16hrs. At room temperature over saturated salt solution in a desiccator. Each sample was weighed accurately using an analytical balance. An average of five readings was obtained and weight was calculated using the formula:-[71]

$$\text{Weight in gm/m}^2 = \frac{W \times 100 \times 100}{5 \times 5 \text{ (area of sample)}}$$

3.2.3 Determination of thickness of the fabric:

For determination of fabric thickness five samples of size 5 cms × 5 cms were cut and conditioned for 16hrs. in a dessicator. The specimens were checked for any kind of irregularity. Each sample was placed on the anvil of “Thickness guage” and the reading on dial was recorded which was then converted to millimeters.

3.2.4 Fabric Analysis:

3.2.4.1 Fiber Identification-

The final fabric was analysed using standard microscope tests. Samples were taken randomly from different parts of the material. The grey jute fabric had jute fibre in both warp and weft directions.

3.2.4.2 Weave Analysis-

The final fabric was checked using the pick glass to identify the weave structure. It was observed that the fabric had 1×1 plain weave structure.

3.2.4.3 Quantitative Analysis-

Samples of size 5 cms ×5 cms were cut from the fabrics. Care was taken to see that test specimens were free from any irregularities. Samples were conditioned over saturated salt solution in a desiccator. Each sample was weighted separately in analytical balance. Warp and weft threads were then weighed separately and analysis was done using the formula:-

$$\% \text{ Weight of warp threads} = \frac{W_1 - W_3}{W_1} \times 100$$

$$\% \text{ Weight of weft threads} = \frac{W_1 - W_2}{W_1} \times 100$$

Where: W_1 = Weight of the fabric

W_2 = Weight of warp threads

W_3 = Weight of weft threads

3.3 Preparation of fabric

For any treatment, the fabric should be free from impurities like dust, wax, etc otherwise it hampers the properties of the treated fabric. Hence, grey jute (100%) fabric was scoured by treating with 2 gm/lt of detergent and 2 gm/lt of soda ash (owf) maintaining the material to liquor ratio (M:L) as 1:40 at 60-65^{0}C for one hour. The

fabric was then washed thoroughly under running water and air dried at room temperature.

3.4 Phase I: Pilot work

3.4.1 Development of color palatte

3.4.1.1 Dyes Used:

Experimentation of natural dyes on cotton has been done. Jute and cotton both are natural cellulosic fibres so the dyes were selected on the basis of their performance.

Table 3.1:- Specification of the dyes used

Sr. no.	Name of Dye	Botanical Name	Parts Used	Class of dye	Colour Component
1	Madder	Rubia cordifolia	Roots	Quinones	Alizarin
2	Turmeric	Curcuma longa	Roots	Curcuminoids	Curcumin
3	Eucalyptus leaves*	Eucalyptus globulus	Leaves	Flavonoids	Tannins and Flavonol
4	Indian Almond leaves*	Terminalia catappa	Leaves	Flavonoids	Tannins

*Fallen leaves were collected and used as dye source

3.4.1.2 Extraction of dyes:

As a natural dye source, Madder, Turmeric, Eucalyptus leaves and Indian Almond leaves were used. The madder and turmeric were obtained in the powdered form. For extraction of dye from it, 2% concentration of dye solution was taken and boiled for 30 minutes. In case of Eucalyptus leaves and Indian Almond leaves, the leaves were dried in shade and then powdered. In order to extract the dye, 3% concentration of dye was taken and was boiled for 30 minutes. The dye extract thus obtained was directly used for dyeing of the bleached samples.

3.4.1.3 Mordanting of the fabrics:

Alum (10% concentration), copper sulphate (4% concentration) and ferrous sulphate (4% concentration) were used as mordants. Both pre and post mordanting was carried out. For which mordant was mixed with water keeping the material to liquor ratio 1:40. The samples were kept in the mordant solution for 30 minutes and then kept in the dye bath for dyeing.

3.4.1.4 Dyeing procedure for the fabrics:

The exhaust dyeing method was used. The dye baths were prepared with the requisite amount of dye, which was calculated on the basis of material to liquor ratio of 1:40. The pre mordanted samples and samples without mordanting were kept in separate dye bath at room temperature. The temperature of the dye bath was increased to boil gradually and the dyeing continued for 1 hour. After dyeing, the pre mordanted samples were thoroughly rinsed and dried. For the post mordanting, samples were kept for mordanting immediately after dyeing, then rinsed and dried.

Pre- mordanting= Mordanting → Dyeing →Rinsing

Post- mordanting= Dyeing → Mordanting →Rinsing

3.4.2 Testing of parameters

3.4.2.1 Determination of wash fastness of Dyed fabrics

An evaluation of wash fastness of dyed fabrics was done using the launder-o-meter as per the A.A.T.C.C. standard test method IA61-1962. The specimen samples were cut and put in a glass jars of 800 ml capacity. The M:L ratio was maintained at 1:40 and the temperature at 40^0C. The test method comprised of three cycles of laundering, each of 45 minutes, which is equivalent to 15 hand washes.

3.4.2.2 Determination of light fastness of Dyed fabrics

Atlas Fade-o-meter was used for evaluating light fastness as per A.A.T.C.C. standard test method 16A-1963. Test specimens were cut of size 3” × 3” and were mounted on a card board having its face covered with black paper. The test specimens were exposed to 5, 10, 15, 20 standard fading hours.

3.4.2.3 Determination of crock fastness of Dyed fabrics

The crocking test to evaluate the rubbing fastness (dry and wet) of dyed fabrics was conducted using the crock-o-meter as per A.A.T.C.C. standard test method 8-1961. The test specimen was prepared by cutting 2" × 5" and fastened to the base of the crock-o-meter so that it rests flat on the abrasive cloth with its long dimension in the direction of rubbing. A square white bleached cotton cloth of size 2" × 2" was mounted over the end of the finger which projects downward from weighed sliding arm. The white testing cloth was allowed to slide, onto the test specimen back and 42 times by making 10 complete turns of the crank at the rate of one turn per second.

For wet crocking test, white testing square were thoroughly wet out in distilled water and were squeezed between filter paper with hands. Wet pick up was brought to 65% $\pm$ 5%. Care should be taken to prevent evaporation from reducing the moisture content. The sample was mounted over the end of the finger which projects downward from weighed sliding arm. The white testing cloth was allowed to slide, onto the test specimen back and 42 times by making 10 complete turns of the crank at the rate of one turn per second.

The staining grey scale by A.A.T.C.C. was used for visual assessment to evaluate crock fastness to dry rubbing and wet rubbing. The rate of fading to every level of laundering and exposure to light was also determined by using geometric grey scale.

The geometric grey scale consists of five pairs of standard grey chips, each pair representing a difference in color corresponding to a numerical fastness rating. The results of color fastness tests are rated by comparing the difference in color of the tested specimen and original textile with the differences represented by the scale. Five classes are provided for most other properties ranging from 5-1 (high to low).

The staining grey scale consists of one pair of white and four pairs of grey and white color chips each representing a visual difference in color or contrast corresponding to a numerical rating for staining. Class 5, negligible or no staining to class 1, heavily stained.

3.4.2.4 Evaluation of degree of color yield

The color strength (k/s) values were computed to study the degree of color yield of treated dyed fabrics. The "Spectro flash SS5100 A" based on CIE laboratory was used for determining the color strength (k/s) values. The k/s values of dyed fabrics were measured three times across the visible spectrum (400-700nm). The wavelength at which maximum k/s value was obtained is indicative of color strength and is termed as maxima, every dye having its own maxima. Thus every dye had its own percent transmission curves. The L*a*b* readings shows the value 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), DL* showed (lightness & darkness of colour), k/s gave (colour strength) and a* and b* values indicated how redder or bluer a colour was.

3.4.3 Pretreatments of the fabric

For the pilot work the pre-treatments based on the studies previously conducted were followed.

3.4.3.1 Bleaching of fabric:

3.4.3.1(a) Hydrogen Peroxide (H_2O_2):

100% grey Jute fabric was bleached with 1% (v/v) of hydrogen peroxide keeping the material liquor ratio 1:40 and pH maintained between 9-10 by using 2gm/l of sodium silicate as stabiliser. The samples were kept into this solution for 30 minutes at 50^0C and then neutralized with 1% (v/v) acetic acid for 5 minutes. The samples were given a final rinse with cold water and air dried at room temperature.

3.4.3.1(b) Sodium Hypochlorite (NaOCl):

15ml/l of sodium hypochlorite was taken and the grey Jute samples were kept into this solution for 45 minutes at room temperature. The samples were given a final rinse with cold water to remove the excess of bleaching agent and air dried at room temperature.

3.4.3.1(c) Peracetic Acid (PAA):

Peracetic acid solution was prepared using 1ml of hydrogen peroxide mixed with 2ml of glacial acetic acid and this mixture was rested for 24 hr. After 24 hr the grey Jute samples were treated with this mixture of peracetic acid for 1hr at room temperature. The samples were given a final rinse with cold water and air dried at room temperature.

The above bleached samples were tested for weight loss, thickness, tensile strength and whiteness index.

3.4.3.2 Woolenization of the fabric:

Woolenization of jute is a process to treat the jute fiber with strong alkali to improve severely the appearance and handle of jute fibers & its blends. Profound changes such as lateral swelling occurs, together with considerable shrinkage in lengths, as a result of which the fiber is softened to the touch and develops a high degree of crimp or waviness. The crimp gives a wool-like appearance to the fiber & the resultant fiber is termed as woolenized jute fibre.[126] The solution of 15% Sodium hydroxide was prepared and the samples were kept into it for 30 minutes at room temperature, then these samples were neutralized with 1% acetic acid for 1 hour. The woollenized samples were tested for weight loss, tensile strength and whiteness index.

3.4.4 Determination of physical properties of fabrics

3.4.4.1 Determination of weight loss:

Samples of size 11.5×11.5 cm from the control fabric were conditioned for 16 hours. Then they were weighed and treated with various pre-treatments. The treated samples were air- dried and again conditioned in the desiccators for 16 hours. Then the samples were weighed accurately and the weight loss was calculated using the following formula:

$$\text{Percent weight loss} = \frac{\text{Initial weight loss}}{\text{Initial weight}} \times 100$$

Initial weight loss = Initial weight loss – weight after the treatment

3.4.4.2 Determination of Tensile Strength:

The tensile strength and elongation of the fabrics with varying treatments were determined on Instron, Model 1121, 10KN Testing Instrument using ASTM Test method D5035. The instrument was based on the principle of CRE (Constant Rate of Extension).

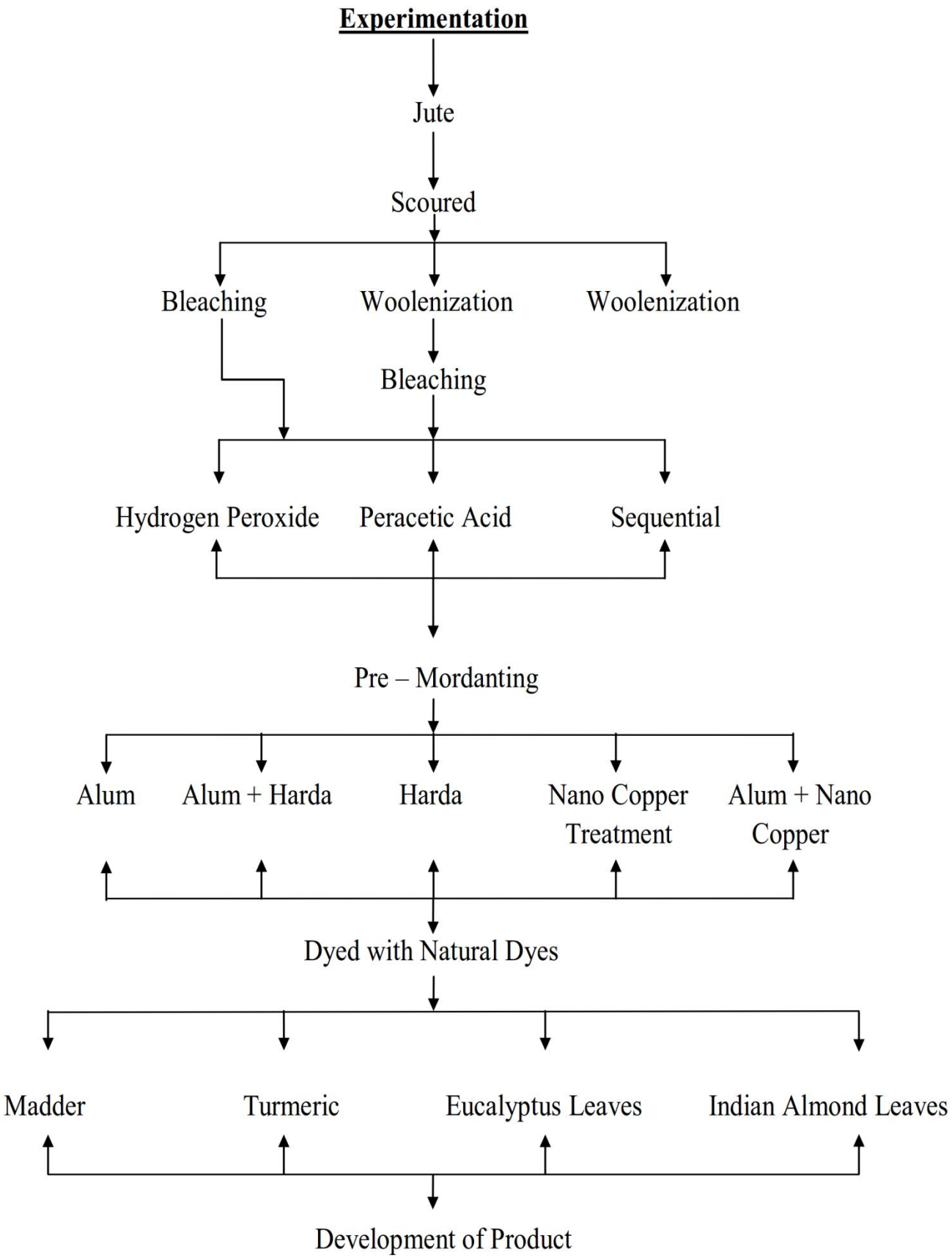
3.4.4.3 Determination of Stiffness:

Stiffness or bending length was measured using “Bending Cantilever Test Method” on “Shirley” Stiffness Tester. The cantilever method was used for the measurement of stiffness of treated fabrics according to ASTM D1388-55T, 1955 standards.

3.4.4.4 Determination of Whiteness Index:

Bleaching of jute was carried out for the removal of natural colour of jute and to make them more whiter. The most common method for estimating the bleached jute was by determination of whiteness index.

Using Spectrascan 5100 spectrometer, manufactured by Premier Colourscan, the whiteness-index of the fabrics were assessed by Hunter Whiteness Index under illuminate D65, 10 degree observer. Whiteness index measurements were performed according to AATCC test method 110. Three measurements were obtained for each sample and average value was calculated and recorded.


Experimentation
Jute
Scoured
Bleaching
Woolenization
Woolenization
Bleaching
Hydrogen Peroxide
Peracetic Acid
Sequential
Pre – Mordanting
Alum
Alum + Harda
Harda
Nano Copper Treatment
Alum + Nano Copper
Dyed with Natural Dyes
Madder
Turmeric
Eucalyptus Leaves
Indian Almond Leaves
Development of Product

3.5 Phase II: Experimentation

Experimentation was carried out as per the results obtained from the pilot work. Grey jute fabric procured from Department of Jute and Fibre Technology, Kolkata, West Bengal was used as the base fabric. This grey fabric was scoured with the standard recipe followed in pilot work. It was observed from the pilot work that the samples which were woollenised first and then bleached gave a better result compared to the one first bleached and then woollenised. Hence, after scouring the fabric, woolenization of the samples was carried out, and then these samples were taken for bleaching.

3.5.1 Pre-treatments of the fabric using standardized procedure

3.5.1.1 Woollenization of the fabric:

The scoured jute fabric was first woollenized with the standard recipe. The solution of 15% Sodium hydroxide was prepared and the fabric was kept into it for 30 minutes at room temperature, then it was neutralized with 1% acetic acid for 1 hour.

3.5.1.2 Bleaching of woollenised samples:

The results of the pilot work revealed that hydrogen peroxide and peracetic acid gave better result compared to sodium hypochloride in terms of whiteness and strength of the fabric. Hence, further bleaching was done with hydrogen peroxide, peracetic acid and sequential bleaching was also added.

The woollenised samples were bleached using the following recipe for three different concentration of hydrogen peroxide ie 6%, 8% and 10%.The recipe used for hydrogen peroxide was as follows:

30% (w/v) Hydrogen Peroxide:- 6%/ 8%/ 10%

Sodium Metasilicate:- 10%

Sodium Carbonate:- 2.5%

Sodium Hydroxide:- 10%

Wetting Agent:- 0.5%

M:L :- 1:20

Temperature: 95^0C

Time:- 1 hr

pH :- 10.8

After the operation the samples were washed thoroughly and neutralized with acetic acid

In case of peracetic acid, the recipe was same as that of pilot work but here pH was varied into three ie self pH (2), pH 6 and pH 7.

In case of sequential bleaching, the scoured jute sample was subjected to two consecutive bleaching treatments with a particular bleaching agent under the same condition as described in pilot study. The jute sample was first treated with Peracetic acid and then treated with Hydrogen peroxide.

All the bleached samples were tested for various parameters like tensile strength, whiteness index, and SEM. The samples which showed the best result were taken up for further work.

3.5.2 Pre-mordanting of the bleached samples:

From the pilot work, it was observed that pre mordanting was better compared to post mordanting. Also from the pilot work, alum was considered further for the study. The best bleached samples were pre mordanted with three different mordants and its combination was carried out. The three mordants and its combination were alum, harda, alum + harda, Nano Copper treatment, alum + Nano copper treatment.

3.5.2.1 Alum, Harda and their combination:

The bleached jute fabric sample was first pre-mordanted using 10% concentration of alum (wof) and harda individually for 30 minutes at room temperature keeping the material to liquor ratio 1:40.For the combination of alum and harda, first the samples were dipped into 10% alum for 1hour then the sample was dipped into 10% harda for another 1 hour.

3.5.2.2 Copper Nano Treatment and its combination:

The 100ml solution of copper sulphate in distilled water was kept on constant shaking water bath, was slowly reduced by drop wise addition of very dilute cold (2 degree C) solution of sodium borohydride in presence of nitrogen atmosphere. As the color of the solution was turned to light yellow, the addition of solution of sodium borohydride was ceased. The bleached jute fabric sample was pretreated with synthesized copper

nano colloid at 40^0C keeping material- to -liquor ratio 1: 40. After 15 minutes, the temperature was gradually raised to 80^0C and the treatment continued for a further period of 30 minutes. The samples were thoroughly washed, neutralized and were directly dyed.

For combination of copper nano and alum, the samples were first pretreated in a bath containing copper nano colloid as discussed above and then the pretreated samples were further mordanted using 10% concentration of alum (wof) and then dyed.

3.5.3 Dyeing of the bleached samples:

As a natural dye source; madder, turmeric, eucalyptus leaves and Indian Almond leaves were used. So, a total of four dyes were selected for the study. Mordants selected for the study were alum, harda and copper nano and their combination were tried out. Hence, the total of mordants was six variations. Bleaching agents which were finalized were three in number. Thus, 18 shades per dye were developed out of the bleaching agent, mordant variations. Thus, overall 72 shades were developed for dyeing of jute with natural dyes.

Madder and turmeric were obtained in the powdered form whereas Eucalyptus leaves and Indian almond leaves were dried under shade and then powdered. In order to extract the dye, 10% of each dye source was taken and boiled for 30 minutes. The dye extract thus obtained was used for dyeing of the samples. The exhaust dyeing method was employed in this study. Dyebaths were prepared with the required amount of dye, keeping material to liquor ratio 1:40. The pre mordanted samples were kept into the dyebath at room temperature. The temperature of the dyebath was increased to boil gradually and dyeing continued for 1 hour. After dyeing, the pre mordanted samples were thoroughly rinsed and dried.

3.5.4 Testing of Optical Indices:

Using Spectrascan 5100 spectrometer, manufactured by Premier Colourscan, the whiteness, brightness and yellowness indices of the fabrics were assessed by Hunter Whiteness Index under illuminate D65, 10 degree observer. Three measurements were obtained for each sample and average value was calculated and recorded.

3.5.5 Testing for Fastness properties:

The dyed samples were evaluated for the following fastness properties.

3.5.5.1. Light Fastness:

The dyed samples were tested for their fastness to light. A fade-o-meter was used to carry out the testing using the test method IS 2454:1985 based on ISO/B-1984. 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.

3.5.5. 2. Rub/ Crock Fastness:

Crock-o-meter was used to assess the rub fastness (dry and wet) of the dyed samples using test method IS 766: 1988 based on ISO 105/X-1984. 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 9 Newton. 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.

3.5.5.3. Wash Fastness:

Lauder-o-meter was used to evaluate the wash fastness of the dyed sample using test method IS 764:1979, based on ISO 105/C-1982. A mechanical washing device with a water-bath containing a rotor with which containers of stainless steel of 500 ml capacity are rotated at speed of 40 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 60^0 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 10cm x 4cm 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. 2gm/l of liquid soap was taken to make the soap solution and it was heated to 60^0 C. The samples were then put along with the soap solution into the jars of the launter-o-meter. A 45 minutes 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 samples. For the evaluation of the rate of staining of un-dyed cloth, was done by comparing the difference in the colour of stained and unstained cloth with the difference represented by the scale. For the evaluation in 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.

3.6 Phase III: Development of product

To test the applicability of jute fabric other than sacks and packing material, the treated fabric was explored for its use in apparels. For that ten jackets were designed and best four jackets were purposely selected. Two natural dyes namely Turmeric (root source) and Indian almond leaves (leaf source) were selected for dyeing these finalized jackets. Two jackets in each dye were pre mordanted with alum and dyed. The construction of these jackets was carried out following the specifications of the designs.

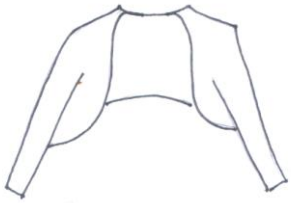
 Design 1	 Design 2
 Design 3	 Design 4
 Design 5	 Design 6
 Design 7	 Design 8
 Design 9	 Design 10

Plate 3.1 Sketches of jackets for final application