EFFECT OF VARIOUS PRE- TREATMENTS ON THE DYEING BEHAVIOR OF JUTE WITH NATURAL DYES

Synopsis of Proposed Ph.D. Thesis

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Introduction

Jute is bio-degradable fibre which is 100% eco-friendly. Jute, the golden fibre is said to have derived its name from the word "jaht" a Sanskrit word. This golden fiber is used extensively in manufacturing sacks or bags which are used as packaging material for agriculture and industrial products. In India mostly jute is grown in Kolkatta and around it in warm and humid regions. The only country exporting jute on commercial scale today is India. Since jute is produced in India, the price is competitively less compared to other countries. Jute is considered the eco-friendly alternative due to its bio-degradability and recyclable property. It is vegetable bast fibre extracted from the stem of Corchorus plant. These stems are used as renewable energy resource and for other diversified uses. Its products are re-usable, sustainable and bio-degradable and deserve policy support towards environmental commitments.

Jute is a lignocellulosic fibre with hemicellulose, cellulose and lignin as the main constitution. It is available in inexhaustible quantities and at comparatively low prices and hence can be considered to replace several expensive fibres. It has inherent advantages like silky luster, high tensile strength, low extensibility, considerable heat and fire resistance, high moisture regain, good dyeability and long staple lengths. These advantages make it directly cater to technical and industrial requirements through its appropriate functional end-uses.

Jute is dyeable with all the dyes used for dyeing cotton. Jute also exhibits strong affinity towards basic dyes. This affinity is due to the presence of non-cellulosic constituents. Among these dyes, only direct, acid and reactive dyes are used extensively for dyeing of jute as they are cheap and easier in application. But the current world awareness about environmental pollution created by synthetic dyes has once again revived the use of environmentally friendly dyes less pollutant dyes. This has directed the researches towards natural dyes. Some of the advantages of using natural dyes:

- 1) It is obtained from renewable resources.
- 2) No health hazards, sometimes it acts as health care
- 3) Practically no or mild chemical reactions are involved in its preparations.
- 4) No residual problems.
- 5) It is harmonized with nature.

Jute in its natural form does not find many end-uses. Its utility can be enhanced by some modifications which include bleaching, softening and colouration. Substantial researches have been carried out on the effects of different bleaching agents on jute and dyeing of jute with natural dyes but the correlation between effects of bleaching agents on dyeing performance of jute using different natural dyes is not explored. Bleaching on jute brings out internal changes in the fibre and therefore it is expected that it will affect the physical properties also. Bleaching results in increasing whiteness index which gives the base for colouration. In the present study, an attempt has been made to study the effect of pre-treatments on the dyeing properties when dyed with natural dyes and its effect on the physical properties of the fibre.

STATEMENT OF THE PROBLEM

In India, jute is the second largest crop cultivated after cotton. Jute is mainly used in making the hessian cloth, sacking material, etc. with the technological advancement. Its use has been extended to various other fields like geo-textiles, furnishings, non-wovens, handmade articles, accessories though to a limited extend. The inherent properties of jute includes silky luster, good tensile strength, high moisture absorption, heat and fire resistant, dimensional stability, antistatic properties, highly dyeable and also good thermal conductivity property. It feels warm in winter and since it has good moisture absorption property it feels reasonably cooler in summer. But along with these advantages, there are a few disadvantages like harsh feel, stiffness, coarseness, poor wash ability and poor abrasion resistance. The stiffness of fabric has great impact on its bending length or drape co-efficient. These drawbacks restrict its use other than packing material. Its utility can be enhanced by overcoming these drawbacks.

With the increased awareness about the environment, the paradigm shift is towards the use of natural products which are bio degradable and cause minimum or no residual harm to the environment. Due to it natural origin and possession of many favourable properties jute has attracted many researchers. To reduce the chemicals in processing of jute, natural dyes are the alternative to produce colour palatte.

Jute has inherent colour which is a drawback to develop colour palatte on jute. Hence, to overcome this, bleaching is required. Bleaching holds the key for successful production of diversified coloured jute products. The primary object of jute bleaching is to get maximum whiteness with minimum loss in strength and retention of whiteness for a longer period. Woolenization, a typical process generally used for jute yarn for imparting crimps in its structure. The crimpness imparted offers wool-like appearance to the fibre.

Substantial research has been carried out on the effects of different bleaching agents on jute and dyeing of jute with natural dyes but the correlation between the effects of bleaching agents on dyeing performance of jute using different natural dyes is hardly available in the literature. In the present investigation, therefore an attempt has been made to study the effect of various

bleaching agents on the physical properties of jute fabric as well as its effect on the dyeing properties when dyed with natural dyes.

The objectives of the study:

- 1. To study the effect of bleaching agents on Jute fabric.
- 2. To study the effects of different bleaching systems on colour development of jute
- 3. To study the effect of nano copper particles on coloration of jute with natural dyes
- 4. To assess the effect of woolenization on Jute fabric.
- 5. To study the influence of natural dyes and mordants on pre-treated jute samples.
- 6. To produce a color palette with natural dyes.
- 7. To analyze the fastness properties of the dyed samples.

Delimitation of the study:

- 1. The study was limited to only two bleaching agents namely: Hydrogen Peroxide and Peracetic acid.
- 2. The study was limited to only four natural dyes namely: Madder, Turmeric, Eucalyptus leaves and Indian Almond leaves.
- 3. The study was limited to the use of two metal mordants; alum and copper sulphate and one natural mordant harda.
- 4. Developing commercial products to judge the applicability of the study.

Scope of the study:

- The study was aimed to develop relationship between effect of pre-treatments and the absorption of jute fabrics. This study will also help to understand the modification of fibre structure for better dye-uptake.
- The study aimed to optimize the chemical conditions to control the physical damage onto the fabrics.
- Also the study aimed to reduce the use of synthetic dye and increase the range of colors to be utilized onto jute fabric with the help of natural dyes.
- This study will give a new dimension in utility of these fabrics as they are eco-friendly and bio-degradable.

Review of Literature

Researches on pretreatments of jute

Jute is known as the golden fibre of India. It has a natural colour which is a disadvantage for the dyers to obtain a particular shade of colour onto it. Some other physical properties like rough texture, stiffness are restricts its use for various end-uses. Hence, to increase its use for various end- products pre-treatments were experimented.

1) Researches on bleaching of jute

Samanta A.K., and et al (2007) studied Hydrogen peroxide and potassium per-oxo-disulphate combined room temperature bleaching of jute, cotton and jute-cotton union fabrics—An energy-efficient eco friendly process. In this study a method for room temperature bleaching of jute, cotton and jute-cotton union fabrics using a combination of H_2O_2 and $K_2S_2O_8$ has been optimized. The measurement of physical properties, such as breaking tenacity, weight loss and surface appearance, including SEM study indicates that the proposed room temperature bleaching process is less damaging to the three types of fabrics studied as compared to conventional hot ($85^{\circ}C$) H_2O_2 bleaching. The whiteness index is within the acceptable range. The suggested reaction mechanism indicates that $K_2S_2O_8$ acts as a peroxide booster showing a synergistic action. This room temperature bleaching process is energy efficient, eco friendly and economical, which can be easily adopted by both small scale and large scale sectors.

Chattopadhyay D.P., Sharma J.K., & Chavan R.B. (2003) studied bleaching of jute with peracetic acid formed in-situ in bleach bath. It was observed that in an unbuffered system, the bleach bath pH drops from its initial set value. Excellent bleaching of jute with relatively higher loss in peroxide of in-situ peracetic acid bleach bath was obtained. A high degree of whiteness in jute with lesser damage was seen at low temperature.

Ghosh S.B., Bajaj P & Kothari V.K. (2003) studied the effect of different dyes and finishes on UV protection property of jute/cotton union fabrics. It was observed that bleaching with H_2O_2 makes the fabrics more permeable to UV rays and then the fabrics were dyed with reactive dyes. It was observed from spectral analysis that the monochlorotriazinyl reactive dye with cyanuric chloride nucleus, such as Cibacron Red FAL, is quite effective in UV protection. From the UV-visible spectral analysis, Cibatex UPF is found to be a suitable finishing agent for rendering sufficient UV protection to the jute/cotton fabric. Simultaneous dyeing and finishing with Cibacron Red FAL and Cibatex UPF provides higher UV protection. The treatment of jute/cotton fabric with titanium dioxide also provides satisfactory protection against UV rays.

Mondal I (2002) conducted a research on effect of hydrogen peroxide bleaching on sulphonated jute-cotton blended fabrics. Raw sulphonated jute, raw sulphonated jute-cotton and cotton fabrics were bleached under different conditions by varying pH, treatment time, temperature, hydrogen peroxide concentration and fabric-liquor ratio. It was observed that bleaching affected the

brightness and breaking strength. The optimum brightness (76.9%) and breaking strength (13.6 kg/l) for blended fabric were obtained with 6.5% hydrogen peroxide at pH 11, temperature 90° C, treatment time 100 min and fabric-liquor ratio 1:30. Brightness increased with the increase in bleaching variables upto certain value and then decreased. On the other hand the breaking strength decreased with the increase in the value of bleaching parameters. Also it was observed that the pH is the important factor which critically affects both breaking strength as well as the brightness. The thermal degradation of bleached sulphonated jute-cotton fabric was characterized by TG analysis and compared with the bleached cotton and bleached sulphonated jute fabrics. It had been observed that the thermal stability as well as brightness and breaking strength of the blended fabrics largely depend on the amount of lignin and hemi-cellulose present in the fabric sample.

2) Research on pretreatments :-

Chattopadhyay D.P., Samanta A.K., and et al. (1999) studied about the effect of caustic pretreatment at varying tension level on dyeing behaviour of jute, flax and ramie yarns has been studied using direct, reactive (cold and highly exhaustive brands) and basic dyes. It was observed that the higher the hemicelluloses content of the lignocellulosic fibre, the higher is the weight loss on caustic soda treatment. Weight loss increases for all the fibres with the increase concentration of caustic soda or decrease in tension level during the treatment. Results revealed that the maximum weight loss was observed in case of jute and minimum for ramie. Decrease in basic dye uptake and increase in direct and reactive dye uptake are observed with the increase in caustic soda concentration during the pretreatment for all the fibres. Dye uptake of reactive dyes increased for ramie and flax with the increase of caustic soda but in case of jute, dye uptake showed a drop. With the increase in tension level during treatment, the dye uptake decreases to some extent, irrespective of the fibre type or caustic soda concentration.

Sharma I.C., Chattopadhyay D.P, Mukhopadhyay A., Boruah R.K., & Vishwanath (1999) studied the effect of degumming followed by sequential oxidative and reductive bleaching on physical properties such as tenacity, thickness, fabric weight, bending length, crease recovery, flexural rigidity and air permeability of mulberry and tussar silk fabrics. It was observed that tenacity, thickness, fabric weight; bending length, crease recovery, flexural rigidity decreases whereas air permeability increases after degumming and bleaching.

3) Research on influence of pretreatments on the properties of jute

E. Sinha & S.K. Rout (2008) in their study used jute fibres which were degummed and then were chemically modified by treatment with 1.26 M (5 wt %) solution of NaOH solution at room temperature for 2,4and 8 hrs. The above samples were characterized by small angle X-ray scattering (SAXS) technique and XRD for macromolecular and microstructural parameters of the fibres before and after alkali treatment where as FTIR and SEM was used for fine structural

details and morphological studies of the fibres. Differential scanning calorimetry (DSC) and instron 1185 analyzed thermal and mechanical behavior of the fibres. Comparison analysis of results confirmed some changes in the macromolecular structure and microstructure of the fibres after chemical treatment due to swelling of macromolecules and removal of some non-crystalline constituents of the fibres. The findings conclude that change in crystalinity developed after alkali treatment resulting improvement in mechanical strength of the fibres. However, the removal of structural constituents after alkali treatment leads the thermal decomposition temperature of the cellulose reduced to 360.62° C after 8 hrs alkali treatment from 365.26° C for raw jute fibre.

Pan, N.and etal. (1999) studied infrared spectra of bleached jute. The Infrared spectra of jute fibre in raw, dewaxed and bleached condition have been observed over the frequency range of 500-4000 cm⁻¹ using KBr Pellet technique. The raw jute was bleached with sodium hypochlorite, sodium chlorite and hydrogen peroxide. Raw jute was dewaxed with ethyl alcohol benzene mixture (1:2 v/v) for 6 hrs. in a Soxhiet apparatus. The raw jute, dewaxed jute and bleached jute were evaluated in Fourier Transform Infrared (FTIR) spectrophotometer. The results revealed that there was no change observed in case of dewaxed jute when compared to raw jute. Sharp reduction was observed in the intensity of the bands at 1764cm⁻¹, 1725cm⁻¹ and 1709cm⁻¹ in the spectra of sodium hypochlorite and hydrogen peroxide bleached jute fibre. In sodium chlorite bleaching, the band intensity has been found to be same as raw jute.

George, E.(1994) studied the light and wash fastness characteristics of chemically pretreated jute based fabrics with reactive dyes, where in the measure of degree of fixation of the dichlorotriazinyl (cold brand), monochlorotriazinyl (hot brand) and vinyl sulphone based reactive dyes was a major objective. The results indicated that 100% jute fabrics has superior dye pick up and dye fixation property. Amongst the dyes procion yellow M-Gr had greatest affinity for the substrates and procion bril blue M-R had least affinity for the substrate. Jute yarns in weft were seen to have higher dye fixation than cotton warps.

Research on dyeing of jute with natural dyes

Patel F., (2010) conducted a research on dyeing of minor fibres with natural dyes. Two cellulosic minor fibres namely sisal and jute and two protein minor fibres namely eri silk and kutch goat hair was taken for the study. Madder, ratanjot, marigold, heena, catechu and falme of forest were the six dyes selected and three dye mixtures of two dyes were derived for the study. The variables of the study were variation of pH during dye application, use of natural mordants tea and pomegranate rind and composite dyeing. A total of 180 shades were produced. Products designed and exhibited were highly appreciated and showed a good applicability of the dyes used.

Samanta A.K., Agarwal P. & Datta S. (2008) studied on Physico-chemical studies on dyeing of jute and cotton fabrics using jackfruit wood extract: Part II- Dyeing Kinetics and Thermodynamic studies. Bleached cotton and jute fabrics were double pre-mordanted applying 20% myrobolan (harda) as 1st mordant and 20% alum or 20% ferrous sulphate as 2nd mordant and subsequently dyed with aqueous extract of jackfruit wood under optimized conditions of dyeing. The physic-chemical parameters of dyeing, such as dyeing affinity, rate of dyeing, absorption isotherms and associated thermodynamic parameters like heat of dyeing (Δ H), entropy of dyeing (Δ S) and Gibb's free energy (Δ G), have been assessed to explain the interaction among different fibre-mordant-dye systems. From the observations, it was found that all the dyeing processes are endothermic, Δ H values being positive. However, the negative Δ G values of all the systems in some otherwise favorable conditions of dyeing need to be further studied.

Pan N.C., Chattopadhyay S.N. &Day A. (2003) conducted a study on dyeing of jute with vegetable dyes. Raw jute fabric was bleached and mordanted with different concentration with potash alum. Then the mordanted bleached jute fabrics were dyed with jackfruit leaf and marigold flower petals seperately. The results revealed that the vegetable color dyed jute fabrics premordanted with 10% potash alum showed better dye uptake and wash fastness properties. Brightness index value of these samples premordanted with potash alum was lower as compared to without mordanted jute fabrics with the same dyes.

Chattopadhyay D.P.and etal. (1997) studied the effect of mordants and mordanting techniques on the dyeing of jute and cotton with the natural dye adula. Different concentrations of mordant and salt are used and in applied through different techniques in pre-mordanting, simultaneous mordanting and post mordanting. With jute, simultaneous mordanting was found better at low concentration of mordants as compared to other two techniques. In case of cotton, the depth of shades found was too light and among the three techniques of mordanting, pre-and post gave comparatively better results. With both jute and cotton, the color depth found was higher with FeSO₄ than to the alum.

Research on product diversification of jute

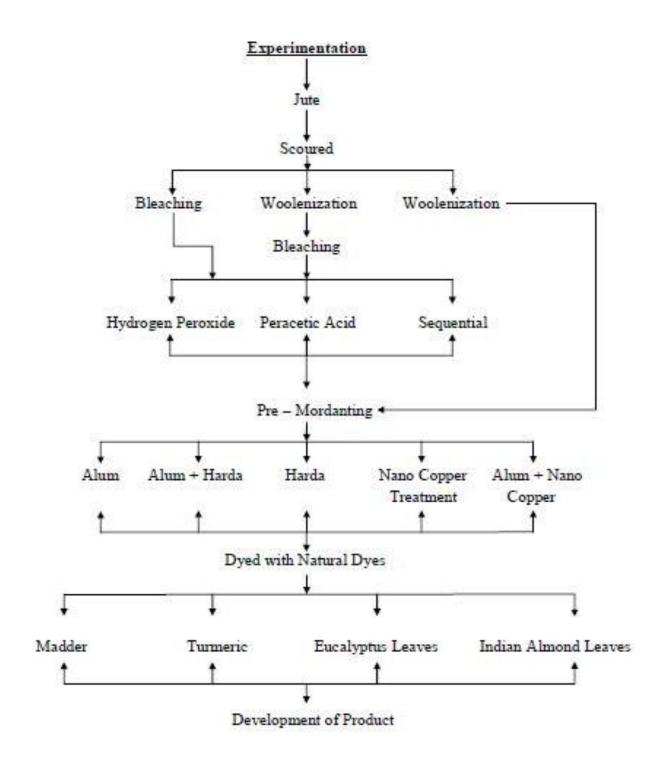
Malhan S. (1999) has done a study on value addition and product diversification of minor fibre fabric (Jute & Banana) using Hand Block Printing. Motifs of Sanganeri printing of Rajasthan with contemporary colors using pigments were used for printing. Various household articles were constructed like cushion covers, lamp shades, table mats, tray and tray covers, wall hanging, partition panel, bed spreads. Then these were evaluated for market acceptability and the results revealed that the majority of the respondents accepted the product for household use.

Raval, P. (1999) printed the cotton banana union fabric and cotton jute fabric using various colored pigments. When tested or wash, light and crock fastness it was observed that out of the three pigments used i.e. pigment red, pigment yellow and pigment blue, pigment red and yellow gave moderate to good wash fastness on both the fabrics. It was also seen that the cotton banana union fabric was quite resistance to light fading incase of all pigments but the crocking fastness of these dyes came out to be poor on both the fabrics.

Sharma, B. (1993) worked on construction of different types of skirts from jute and jute multi fibre fabrics and the evaluation results revealed that the majority of respondents (98 percent) appreciated the garments and were ready to add them to their wardrobe. When asked about the suitability, higher number preferred jute cotton fabrics best for skirt constructions followed by jute viscose fabrics.

RESEARCH DESIGN

The research was experimental and exploratory study to optimize the pre treatments carried out onto jute fabrics for better dyeing of jute fabric with natural dyes.



METHODOLOGY

The study undertaken had an experimental and exploratory structure and was divided into two phases explained as follows:

PHASE I: Pilot study

A pilot study was an essential step for this research since it was exploratory in nature. Pilot work had been divided into two parts: 1) Development of colour palatte and 2) Pre-treatments.

- 1) **Development of colour palatte**: Four natural dyes namely Madder, Turmeric, Eucalyptus leaves and Indian Almond leaves and three metal mordants alum, copper sulphate and ferrous sulphate were purposively selected for the study. Colour palatte was developed using these dyes and mordants individually on the bleached jute fabric. Bleaching was done using three bleaching agents namely Sodium Hypochlorite, Hydrogen Peroxide and Peracetic acid with the standard recipe. After bleaching, dyeing in natural dyes was carried out for pre and post mordanted sample. Thus, number of shades developed for these three bleaching agents were 36 shades individually, making the total sample as 72 samples for pre and post mordanting. K/S and L a^{*} b^{*} values were observed and analyzed. Based on the results, it was decided to carry forward only alum as a mordant for further study.
- 2) Pre-treatments: Bleaching and Woolenization were the two pre-treatments for the study. Bleaching was carried out using three bleaching agents namely Sodium Hypochlorite, Hydrogen Peroxide and Peracetic acid. These samples were then dyed with Madder. The physical properties of these samples were tested and the results were compared.

Woolenization of the sample was carried out using standard recipe. Then the sample was dyed with Madder and tested various physical properties. Also, there were experiments carried out to investigate the effect of altering the sequence of bleaching and woolenization. Hence, one set of jute samples was bleached first and then woolenized and another set of jute samples was woolenized first and then bleached with all the three bleaching agents.

It was observed that the samples treated with sodium hypochlorite resulted reasonably higher loss in strength and was also seen from the SEM images show the damage caused to the sample. Hence, it was not used for further study.

Further experiments were carried out to optimize the recipe for hydrogen peroxide and peracetic acid. Variations in terms of time and percentage were used for hydrogen peroxide whereas for peracetic acid, variation was time and pH. For hydrogen peroxide, time variation was 60, 90 and 120 minutes while the percentage variation was 6%, 8% and 10%. In case of peracetic acid, time was 60, 90 and 120 minutes while pH was self (pH 2), pH 6 and pH 7. Then with all these variations in both the bleaching systems, premordanting with alum was done. These bleached, pre-mordanted samples were dyed using four natural dyes namely Madder, Turmeric, Eucalyptus leaves and Indian Almond

leaves. Total samples resulted from these experiment were 72. K/S values of all 72 samples were taken and analyzed.

The best sequence of the pre-treatment was taken further for the next phase. The sequence taken further was Woolenization followed by bleaching using the best optimized bleaching condition obtained from the pilot work.

PHASE II: Experimentation

Experimentation was carried out using the results obtained from the pilot work. 100% jute fabric was taken and scoured properly then used. The scoured jute fabric was then woolenized using the standard recipe and then bleached with hydrogen peroxide, peracetic acid and their combination i.e. sequencial bleaching. The optimized condition for hydrogen peroxide was 10% concentration of hydrogen peroxide for the time duration of 90 minutes and for peracetic acid, the optimized condition was pH 7 for 90 minutes. In case of sequencial bleaching, initially the jute sample was bleached with peracetic acid and then bleached with hydrogen peroxide.

I] Conditions for pre-treatments:

- **Woolenization:** The scoured jute sample was treated with 15% concentration of sodium hydroxide for 30 minutes at room temperature maintaining the material liquor ratio 1:40. The sample was then neutralized using dilute acetic acid.
- **Hydrogen Peroxide Bleaching:** The scoured fabric was bleached with 10% (v/v) of hydrogen peroxide (30%) using 10% of sodium metasilicate as stabilizer maintaining the material liquor ratio 1:20. The fabric was treated in this solution for 60 minutes at 95^oC. The samples were then neutralized using dilute acetic acid.
- **Peracetic acid Bleaching:** Peracetic acid was prepared by reacting hydrogen peroxide with acetic acid. The most commonly used method for the preparation of peracetic acid was direct, acid catalyzed reaction of 30-98% hydrogen peroxide with acetic acid. For this study, peracetic acid was prepared by reacting hydrogen peroxide (30%) with glacial acetic acid in 1:2 molar ratio at room temperature for 24 hours using sulphuric acid as catalyst. The scoured jute fabric samples were treated with freshly prepared peracetic acid for 90 minutes at pH 7 and the temperature was maintained at room temperature.
- **Sequential Bleaching:** The scoured jute sample was subjected to two consecutive bleaching treatments with a particular bleaching agent under the same condition. The jute sample was first treated with Peracetic acid and then treated with Hydrogen peroxide.

II] Application of Mordants:

From the pilot work it was found that pre- mordanting resulted the best result. Hence, for further experimentation, pre-mordanting was finalized. The mordants used were 1) Alum, 2) Harda, 3) Harda followed by Alum, 4) Nano Copper and 5) Nano Copper followed by Alum. Each sample was pre-mordanted with corresponding mordant separately.

- Alum: For corresponding bleaching treatment, the samples were pre-mordanted with alum. The concentrations of alum were 5%, 10%, 15%, 20% and 25%. Then these pre-mordanted samples were dyed. To optimise the concentration of alum, the K/S values of dyed samples were observed and analysed.
- **Harda**: Bleached sample were premordanted using varying concentration of Harda i.e. 5%, 10%, 15%, 20% and 25% for each bleaching treatment. To optimise the concentration of harda, the K/S values of dyed samples were observed and analysed.
- Harda and Alum: The samples were first pre-mordanted with 10 % Harda and then the samples were dried. Without washing, the harda pre-mordanted samples were treated with 10% Alum keeping M:L ratio as 1:20. These samples were further dyed.
- Nano- Copper: For synthesis of nano- copper, following method was used: The 100ml solution of copper sulphate was kept on shaker plate and the solution was slowly reduced by drop wise addition of very dilute chilled solution of sodium borohydride till the solution turned to light yellow. The bleached jute fabric was treated with nano copper using two techniques i.e. 1) applied directly on bleached jute fabric 2) added into dye bath. The nano Copper was applied to bleached jute fabric using exhaust method at 40^oC keeping material to liquor ratio 1: 40. After 1 hour, the temperature was rised to 80^oC and the material was treated for 30 minutes more. Then the samples were thoroughly washed and dried in air. In other method, the jute fabric was dyed for 15 minutes at 40^oC keeping material to liquor ratio 1: 40 and after 15 minutes of dyeing, the Cu nano solution was directly added into the dyebath. Further the sample was dyed for 30 minutes.
- **Nano-copper and Alum:** The samples were first pre-mordanted with nano- copper and then dried. Without washing, the nano- copper applied and pre-mordanted samples were treated with 10% Alum keeping M:L ratio as 1:20. These samples were then dyed.

III] Application of Natural dyes:

As a natural dye source; madder, turmeric, eucalyptus leaves and Indian Almond leaves were used. 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.

IV] Testing of physical properties of fabric: The treated fabrics were evaluated for the following physical properties:

- 1. Fabric Count: Thread count (number of yarns/cm²) in woven fabrics was determined as per ASTM 2775-98 standards
- 2. Fabric Thickness: Compress-o-meter was used to determine the thickness of the fabric. It was measured as per ASTM D 1777-96 standard test method.
- 3. GSM: The mass unit (gm/m²) was measured as per ASTM D 3776-96 standard test method.
- 4. Tensile Strength and Elongation of Fabric: It was 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)
- 5. Stiffness: Stiffness or bending length was measured using "Bending Cantilever Test Method" ASTM No: 1388-55T on Shirley stiffness tester.
- 6. Crease Recovery: The crease recovery was determined by "Shirley Wrinkle Recovery Tester" manufactured by T.J.Edward Inc. as per AATCC standard test method 66.
- 7. Optical Indices and K/S values: The optical indices and K/S values were determined on Spectrphotometer Premier Colorscan SS5100A.

RESULTS AND DISCUSSION

The research was aimed of exploring suitable pre-treatments for obtaining improved dyeing properties of jute using natural dyes. Two sources of natural dyes i.e. root and leaves were purposively selected for the study. The root sources were Turmeric and Madder while the leave sources were Eucalyptus Leaves and Indian Almond leaves.

PHASE I: Pilot study

Pilot study was carried out to finalize the mordants, mordanting techniques and the pretreatments for the study. Four natural dyes namely Madder, Turmeric, Eucalyptus leaves and Indian Almond leaves and three metal mordants – alum, copper sulphate and ferrous sulphate were purposively selected for this study. Colour palatte was developed using these dyes and mordants on the bleached jute fabric. Two aspects were studied: one the shade and amount of colour obtained on jute fabric and second the dyeability of the jute fabric with selected mordants and dyes. The number of shades developed for these three bleaching agents were 36 shades each, making the total sample as 72 samples for two treatments. The colour ranged from bright yellow to dark brown, marron to dark coffee, olive green to dirty dark green and dark grey. The K/S value of madder ranged from 5.84 to 13.79, turmeric ranged from 3.50 to 27.48, Eucalyptus leaves ranged from -1.97 to 18.89 and for Indian Almond leaves ranged from -1.89 to 18.90. It was decided by the researcher and guide based on results to carry forward only alum as a mordant and pre-mordanting technique for further study.

Bleaching and Woolenization were the two pre-treatments finalized. Bleaching was carried out using three bleaching agents namely Sodium Hypochlorite, Hydrogen Peroxide and Peracetic acid. It was observed that with sodium hypochlorite there was mush loss of strength and also SEM images show the damage created to the jute sample with this. Hence, it was not carried forwarded for rest of the study. Hydrogen Peroxide and Peracetic acid showed better results.

The sequence to be followed for the pre-treatments was decided on the basis of the results obtained for whiteness, yellowness and brightness. The treatment with alkali on jute fabrics gives a yellow shade to the fabric. Hence, it was decided to do woolenization followed by bleaching.

It was observed from the appearance of the pretreated sample and K/S values for Hydrogen peroxide: 10% at 90 minutes was the best result and for Peracetic acid at pH 7 for 90 minutes was the best result. Those parameters were preferred which caused less damage to the fabric. Hence, these two conditions were taken further for the study. The optimized condition for Hydrogen peroxide and Peracetic acid was 10% concentration at 90 minutes and pH 7 at 90 minutes respectively.

PHASE II: Experimentation

Experimentation was carried out using the results obtained from the pilot work.

I] Application of pre-treatment:

The scoured jute fabric was woolenized using the standard recipe and then bleached with hydrogen peroxide, peracetic acid and their combinations. The optimized condition for hydrogen peroxide was 10% concentration of hydrogen peroxide for the time duration of 90 minutes and for peracetic acid, the optimized condition was pH 7 for 90 minutes. In case of sequential bleaching, initially the jute sample was first bleached with peracetic acid and then with hydrogen peroxide. All the samples were tested for optical indicies namely whiteness, yellowness and brightness indicies. The best result amongst the three bleaching treatments obtained for sequential bleaching which is also supported by the findings of Chattopadhyay et al.(3). They have also reported that sequential bleaching produce better whiteness with less loss in strength, weight and abrasion resistance. Moreover, it also improves softness of bleached jute fabric to an appreciable extent.

II] Application of mordants:

The pre-mordanting technique was selected for this study. The mordants used were 1) Alum, 2) Harda, 3) Harda and Alum, 4) Nano Copper and 5) Nano Copper and Alum. The application concentration of alum and harda were 5%, 10%, 15%, 20% and 25%. These pre-mordanted samples were dyed. The mordant concentration were optimized from the colour depth of the dyed samples. The results revealed that the concentration of alum which gave the best results was 10% and for harda it was 15%. The concentration taken for sequential mordanting of Harda and Alum was 10% each.

III] Application of Natural dyes:

As a natural dye source; madder, turmeric, eucalyptus leaves and Indian Almond leaves were used. The samples were pre-mordanted with the optimized condition of mordants and then dyed with the selected natural dyes. For each dye, five mordant sets were used making total of five samples for each dye. The total numbers of samples obtained for four dyes were 20. K/S value was obtained for each sample.

CONCLUSION

For the present study, two pre-treatments were optimized, one was bleaching and the other was woolenization. The jute samples were bleached using three bleaching agents namely Hydrogen Peroxide bleaching, Peracetic acid bleaching and Sequential bleaching then these samples were pre-mordanted with various mordants like alum, harda, copper-nano, and their combinations. All these pre-mordanted samples were dyed with four natural dyes namely madder, turmeric, eucalyptus leaves and Indian almond leaves. Another set of samples was only woolenized and

then pre-mordanted and finally dyed. The third set was first woolenized, then bleached, followed by pre-mordanting and finally dyed.

The results of the first set i.e. bleaching-mordanting-dyeing sequence revealed that sequential bleaching produced the best result so far as colour depth is concerned.

After dyeing, only woolenized samples were a darker compared to only bleached samples. Even the brightness, whiteness indicies were higher compared to the bleached samples.

Hence, woolenization-bleaching-mordanting-dyeing sequence was chosen for further study. The results revealed a better K/S as well as high whiteness, brightness indicies. Even SEM images showed the same.

REFERENCES

- 1. Bandyopadhyay B.N., and et al., (1999) "Estimation of lignin in jute fibres using FT-IR spectroscopy", Indian Journal of Fibre & Textile Research, Vol 24, Sep. 156-159.
- 2. Bhattacharya N., Doshi B.A., and Shasrabudhe A.S., (1998) "Dyeing jute fibres with Natural Dyes", American Dyestuff Reporter, 26-29.
- 3. Chattopadhyay D.P., Sharma J.K., & Chavan R.B. (2003) "In-situ peracetic acid bleaching of jute", Indian Journal of Fibre & Textile Research, 456-461
- 4. Chattopadhyay D.P., and et al., (1999) "Effect of caustic pretreatment at varying tension level on dyeing behaviour of jute, flax and ramie", Indian Journal of Fibre & Textile Research, 74-77.
- 5. Chattopadhyay D.P., and et al., (1997) "Effect of mordants and mordanting techniques on the dyeing of jute and cotton with adula", Man Made Textiles in India, 361-365.
- 6. Cook J. (1968), Handbook of textile fibres, published by Merrow Publishing.
- George E. (1994) "A Study on Light and Wash Fastness Characteristics of Chemically Pretreated Jute Based Fabrics Dyed with Reactive dyes.", Unpublished Master's Dissertation (Home); M.S.University of Baroda.
- Ghosh S.B., Bajaj P & Kothari V.K. (2003) "The Effect of different dyes and finishes on UV protection property of jute/cotton union fabrics", Indian Journal of Fibre & Textile Research, 431-436.
- 9. Gogoi N. & Kalita B. (1999) "Dyeing of silk with natural dyes (part I)", Colourage, 23-26.
- 10. Gupta R. (1992) "A study on the Physico-Chemical Characteristics of Chemically Modified Jute and Jute Multifiber Fabrics.", Unpublished Master's Dissertation (Home); M.S.University of Baroda.
- 11. Kashyap R., Sharma L., & Ojha S., (2012) "Product diversification on jute fabric through Dabu printing", Colourage, 41-43.
- 12. Kundu, A. and etal. (1993) "Enhanced bleaching and softening of jute pretreated with polysaccharide degrading enzymes", Textile Research Journal 63(8), 451-454.
- Malhan S. (1999) "A Study on Value Addition and Product Diversification of Minor Fibre Fabrics (Jute and Banana) Using Hand Block Printing.", Unpublished Master's Dissertation (Home); M.S.University of Baroda.
- 14. Mondal I (2002) "Effect of hydrogen peroxide bleaching on sulphonated jute-cotton blended fabrics", Indian Journal of Fibre & Textile Research, Vol 27, Sep., 280-283.
- 15. Pandey, S., Chattopadhyay, S. & Pan, N. (1993) "Recent developments in bleaching of jute and allied fibres", Colourage, 29-34.
- 16. Pan N.C., Day A. & Mahalanabis K., (1999) "Infrared Spectra of Bleached Jute", Colourage, 15-20.

- 17. Pan N.C., Chattopadhyay S.N. &Day A. (2003) "Dyeing of jute with vegetable dyes", Journal of Textile Association, 243-245.
- 18. Patel B., and et al., (2014) "Changes in cotton treated with choline chloride", The Indian Textile Journal, 74-78.
- 19. Patel F.,(2010) "Dyeing of minor fibres with natural dyes", Unpublished PhD Dissertation (Home); M.S.University of Baroda.
- Raval P.(1999) "An Experimental Study on Color Fastness Properties of Eco-friendly Pigment Printed, Chemically Pretreated Cotton, Banana and Jute Fabrics.", Unpublished Master's Dissertation (Home); M.S.University of Baroda.
- 21. Samanta A.K., and et al (2007) "Hydrogen peroxide and potassium per-oxodisulphate combined room temperature bleaching of jute, cotton and jute-cotton union fabrics—An energy- efficient ecofriendly process", Indian Journal of Fibre & Textile Research, 221-231.
- 22. Samanta A.K., and et. al (2008) "Physico-chemical studies on dyeing of jute and cotton fabrics using jackfruit wood extract: Part II- Dyeing Kinetics and Thermodynamic studies", Indian Journal of Fibre & Textile Research, 66-72.
- 23. Samanta A.K., and et. al (2008) "Dyeing of jute with binary mixtures of jackfruit wood and other natural dyes- Study on colour performance and dye compatibility.", Indian Journal of Fibre & Textile Research, 171-180.
- 24. Saptharishi L., Jute Handlooms of India, Mapin Pub. Pvt. Ltd., Ahmedabad.
- 25. Sharma B. (1993) "Designing and Construction of Skirts from Jute and Jute Multifiber fabrics", Unpublished Master's Dissertation (Home); M.S.University of Baroda.
- 26. Sharma I.C., Chattopadhyay D.P, Mukhopadhyay A., Boruah R.K., & Vishwanath (1999) "The efect of degumming followed by sequential oxidative and reductive bleaching on mulberry and tasar silk fabrics", Indian Journal of Fibre & Textile Research, 290-293.
- 27. Teli M.D., Adivarekar R.V., Bhagat M., & Manjrekar S.G., (2002) "Response of jute of the dyes of synthetic and natural origin", Journal of the Textile Association, 129-134.
- 28. Verma C., & Venkatachalam V., (2002) "The effect of mordants on mango (bark) dye for dyeing of jute/cotton union fabric", Colourage, 49-54.
- 29. <u>www.jute-industry.com/jute-ecofriendly</u>
- 30. <u>www.ministryoftextile.com</u>

Endorsement from the Supervisor:

Ms. Sukriti Patel has researched extensively on the topic "Effect of Various Pre-Treatment on the Dyeing Behavior of Jute with Natural Dyes" vide Registration No. 126, Dated : 25/11/11. She has presented her progress of work in seminars well attended by teachers and students of the department. She has personally presented the following papers:

Sr. No.	Title of the paper	Type of Seminar	Organized By	Published
1.	Experimental Study Of Eucalyptus And Indian Almond Leaves As A Source Of Dye For Textiles (Oral)	Regional Science Congress	Faculty of Family and Community Sciences, The Maharaja Sayajirao University of Baroda, Vadodara held on September 15 th & 16 th , 2012.	Abstract
2.	Developing A Color Palette On Jute With Indian Almond Leaves And Eucalpytus Leaves (Poster)	National Seminar on "Dynamics of Craft Development- A Technopreneur Approach"	Department of Clothing and Textiles, The Maharaja Sayajirao University of Baroda, Vadodara held on December 21 st – 22 nd , 2012	Abstract
3.	Effect Of Bleaching Agents On Dyeing Behaviour Of Naturally Dyed Cotton Fabric (Poster)	International Seminar on "Enhancing Health, Wellbeing and Sustainability Opportunities, Challenges & Future Directions"	Nirmala Niketan, College of Home Science, Mumbai held on January 10 th – 12 th ,2013	Abstract
4.	Indian Almond Leaves As A Dye Source (Oral)	International Seminar on "Environment and its impact on society",	J.D.Birla Institute, Kolkata held on August 18 th – 20 th , 2013	Full length
5.	Effect Of Nano Copper on Dyeing Of Jute With Natural Dyes (Oral)	International Seminar on "Application of Nano Materials in Textiles"	Anchor Institute (Textile Sector), Department of Textile Engineering, Faculty of Technology & Engineering, The Maharaja Sayajirao University of Baroda, Vadodara held on April 23 rd – 25 th , 2015	Full length
6.	Study On The Effect Of Bleaching Conditions On Colour Development Of Jute With Natural Dye (Oral)	National Seminar on "Recent Scenario in Science and Technology (RSST)"	Faculty of Technology & Engineering, The Maharaja Sayajirao University of Baroda, Vadodara Under DST Purse Programme held on February 27th, 2016.	Abstract

Ms. Sukriti Patel published a paper entitled "Effect of bleaching agents on colour depth of jute fabric dyed with natural dyes", in Journal of Textile Science and Engineering, Volume 6 Issue 4 1000268.

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