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BIOCHEMISTRY

Effectiveness of Different Mordants and Concentrations on the Dyeing Properties of Jackfruit (*Atrocarpus heterophyllus*) Bark on Silk

Tusharbala Sahoo[1]*, Goutama Bhattacharya[2] and Sanjaya K Dash[3]

[1]College of Community Science, Orissa University of Agriculture and Technology, Bhubaneswar, Odisha, India
[2]Institute of Textile Technology, Choudwar, Odisha, India
[3]College of Agricultural Engineering and Technology, Orissa University of Agriculture and Technology, Bhubaneswar, Odisha, India

*Corresponding author: sk_dash1006@hotmail.com

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ABSTRACT

A study was carried out to assess the effect of different mordants and concentrations on the colouring characteristics of jackfruit bark dye on silk. Six types of mordants, viz. citric acid, alum, ferrous sulphate, aluminum sulphate, copper sulphate and potassium dichromate ($K_2Cr_2O_7$) were used, each at 1, 2 and 3% of weight of the fabric (o.w.f.). The material liquor ratio was maintained at 1:40. The Hunterlab colour scale was used to analyse the variations in colour and intensities. The results indicated that the maximum change in colour with respect to the undyed silk fabrics was for 2% alum treated samples with the ΔE* value of 27.08±0.29. However, the values were not significantly different from the samples treated with $FeSO_4$ mordant at 2% and 3% levels, $CuSO_4$ mordant at 2% level and alum at 3% level. The change in chroma (ΔC*) value was maximum for the alum (2%) mordant (24.44±0.2). The analysis on colour fastness with respect to washing, rubbing and sunlight also indicated that all mordants and concentrations used in the study excepting 1% alum offered adequate fastness characteristics. The unmordanted samples exhibited moderate fastness. In view of the colour fastness with respect to washing, rubbing and exposure to light, the jackfruit bark dye can be applied on silk with any of the selected mordants at 3% level.

Highlights

- The jackfruit bark has a good potential to be used as a natural dye
- The colour shades and intensities of the jackfruit bark dyed on silk vary with the type of mordant and its concentration
- Mordanting is essential for the fastness of colour of the jackfruit bark dyed silk

Keywords: Mordant, wash fastness, rubbing fastness, alum, ferrous sulphate, copper sulphate, aluminium sulphate, potassium dichromate, citric acid

Natural dyes produce very uncommon, soothing and soft shades when compared to the synthetic dyes, which are widely available at cheaper prices and which produce a wide variety of colours. It sometimes cause skin allergy and other harms to the human body. It produces toxicity/chemical hazards during its synthesis, and release undesirable and perilous chemicals (Samanta and Agarwal 2009; Sahoo *et al.* 2012). Hence, there is an increased interest and emphasis for the textiles (preferably natural fibre product) dyed with eco-friendly natural dyes, and more and more plant materials are being explored for the production of the dye materials (Samanta and Agarwal 2009). Natural dyes offer a wide range of shades and various parts of the plants, animals/ creatures, sediments, microorganisms, etc. can be used to prepare these dyes (Sahoo *et al.* 2014). The main plant parts used for this purpose are the roots, bark, leaves, flowers and fruits.

Recently, many commercial dyers and textile export houses have started re-looking to the maximum possibilities of using natural dyes for dyeing and printing of different textiles and for targeting niche market. Scientific studies related to the development of natural dyes have been of a great research interest and there have been many approaches to make use of the flowers, leaves and the barks of the tree as natural dyes (Samanta and Konar 2011; Rungruangkitkrai and Mongkholrattanasit 2012; Sahoo *et al.* 2013; Alemayehu and Teklemariam, 2014). India has a rich biodiversity and many plant parts can be used for the preparation of dyes. For the successful commercial use of these natural dyes and for any particular fibre, appropriate and standardized techniques of dyeing have to be adopted. Besides, appropriate scientific dyeing techniques/ procedures can aid to obtain newer shades with acceptable colour fastness behaviour and reproducible colour yield (Sahoo *et al.* 2014).

Jackfruit (*Atrocarpus heterophyllus*) trees are commonly found in the natural ecosystem of Odisha, particularly in the tribal regions. If the barks of the plants could be commercially used for the preparation of dyes, that would add to the income and livelihood of the tribal people. However, it is important that there should be a standardized procedure for the extraction of dye and for the application method on the fabrics. In this context, a study on the effects of the dyeing process variables, dyeing kinetics and the test of compatibility of selective natural dyes has become very important.

Some recent studies have also indicated antimicrobial properties of some natural dyes (Rungruangkitkrai and Mongkholrattanasit 2012; Kanchana *et al.* 2013; Sahoo *et al.* 2014). In general, the textile materials and clothing provide a large surface area and absorb the moisture required for microbial growth and thus are susceptible to microbial attack. Many of the plants used for the extraction of dye are classified as medicinal and some of these have recently been shown to have antimicrobial properties (Kanchana *et al.* 2013; Sahoo *et al.* 2014). Thus, textile dyed with natural dyes having antimicrobial properties can be useful in developing clothing to protect human beings against the common infections. However, dyes commonly used in textile are seldom screened for its use as antimicrobial agents for textile finishing.

Natural dyes are mostly non-substantive and must be applied on textiles with the help of mordants, usually a metallic salt, having an affinity for both the colouring matter and the fibre. Different types and selective mordants or their combination can be applied on the textile fabrics to obtain varying colour or shade, to increase dye uptake and improve the colour fastness behaviour of any natural dye (Rungruangkitkrai and Mongkholrattanasit 2012). Therefore, the present study was planned to check the colour intensities of jackfruit bark dyes on silk with the help of different mordants at three levels of concentrations and to study the colour fastness, wash fastness and rub fastness so as to standardize the type of mordant and concentration. The antimicrobial properties of the dyes were also studied to explore the possibility of using the dyed fabrics as antimicrobial ones.

MATERIALS AND METHODS

Extraction of dye

The bark of the jackfruit tree was collected from the natural ecosystem of Daringbadi in Kandhamal district of Odisha in the month of March. After initial cleaning, the barks were dried under shade till it became completely dry and suitable to be pulverized. The final moisture content of the barks was observed to range between 8.9 and 11.3% (on dry weight basis). The barks were then chopped manually into tiny pieces. A hammer mill was then used to convert the chopped pieces into fine powder. The hammer mill was used to maintain the temperature of the powder below 50°C as higher temperature during pulverization process might influence colour fastness and the antimicrobial properties of the dye (Sahoo *et al.* 2014). The powder was screened on a 200 mesh sieve.

The powdered dye was taken in water (@ 1:10 w/w basis, i.e. 100 g powder in 1 litre water) and was boiled under pressure for 1 hour. After observing the amount of evaporation of water, the final water content was adjusted to the level of 10% stock solution. The liquid was then passed through filter paper (Whatman No. 4) and kept in a refrigerator for further use. Fig. 1 shows the different stages of the preparation of dye from the bark. The dye solution was subjected to light of wavelength 300-700 nm using UV visible spectrophotometer (Perkin-

Fig. 1: Preparation of dye solution from jackfruit bark

Elmer) and the λ_{max} was found to be in the range of 300-400 nm.

Collection and degumming of silk sample

White mulberry silk fabric was obtained from the Odisha Cooperative Tasar and Silk Federation Limited, Bhubaneswar. It is required to degum the silk before dyeing because the natural gum sericin present in the silk reduces dye absorption as well as the lusture of the fibre (Sahoo *et al.* 2013). The silk was degummed in a solution prepared by dissolving 5 gpl neutral soap and 1% (w/w) sodium carbonate in water with material liquid ratio (M:L) of 1:40. The temperature of the bath was gradually raised from room temperature to 90°C and the process continued for one hour. Then the silk fabrics were taken out from degumming bath and squeezed to remove the excess liquid and thereafter rinsed under running water to make it free from the traces of detergent and other chemicals. Subsequent drying was carried out under shade.

Mordanting

Six different mordants, viz., citric acid ($C_6H_8O_7$), alum ($KAl(SO_4)_2.12H_2O$), ferrous sulphate ($FeSO_4$), aluminum sulphate ($Al_2(SO_4)_3$), copper sulphate ($CuSO_4$), and potassium dichromate ($K_2Cr_2O_7$) were used, each at 1, 2 and 3% of weight of fabric (o.w.f.). These mordants (chemicals) basically serve as the cross linking agents to create an affinity between the fibre and the dye.

Pre-mordanting of the fabrics are preferred to post mordanting and simultaneous mordanting because of higher effectiveness (Samanta and Agarwal 2009; Saravanan and Chandramohan 2011), and hence, the same was adopted in the present study. The quantities of mordants were taken at three levels, viz. 1, 2 and 3% of the weight of the fabric (o.w.f.). The mordant was thoroughly mixed with distilled water to obtain the material liquor ratio (MLR) of 1:40. Degummed silk fabric was put into the mordant bath at normal temperature. The temperature was then raised to 90°C for 30 minutes.

The mordant solution was allowed to cool and the sample was dried in the laboratory by normal air circulation (Kumaresan *et al.* 2012).

Dyeing silk fabric

Open dye beaker baths were used for dyeing the silk fabric. The temperature and the time of soaking were maintained at 90°C and 1 hour, respectively. The dyed samples were allowed to cool to 45-50°C and were then washed by running water to remove the unfixed dye particles, un-reacted mordanting agents and any extra deposits from the surface (Sahoo *et al.* 2013). Then soaping by non-ionic detergent (NID) for 10 minutes was carried out to remove the remaining particles and other chemical reagents. The samples were dried in the laboratory using air circulation.

Measurement of colour

The colour intensities and variations were analysed with the HunterLab colorimeter with the CIE L*a*b* colour scale (Sahoo *et al.* 2012; Saravanan *et al.* 2013). The L* value represents lightness, a* value represents redness/greenness (Positive 'a*' values indicate amount of redness, negative 'a*' values indicate amount of greenness) and b* value represents yellowness/blueness (positive 'b*' indicate yellowness and negative 'b*' indicate blueness). The total colour difference ΔE* was calculated as per equation 1 (Sahoo *et al.* 2014), and was compared with the control (undyed silk).

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \qquad \ldots(1)$$

The change in chroma value was also calculated as follows.

$$\Delta C^* = \sqrt{\Delta a^{*2} + \Delta b^{*2}} \qquad \ldots(2)$$

Five replications were taken for all the individual parameters and the statistical analysis was conducted with SAS 9.3 to find out the individual effects of the type of mordant and concentration and the interaction effects.

Colour fastness tests

Bleeding of colour occurs during laundering, if the dyes are held loosely by the fibre, i.e. dyes that have not penetrated the fibre sufficiently, or dyes which are held only by weak forces such as hydrogen bond or Vander Waal's forces (Livneh 2005; Sahoo *et al.* 2014). Similarly, the poor rubbing fastness also indicates if some dye molecules are superficially held on the fabric. The dyed fabric samples were tested for colour fastness for washing, rubbing and the exposure to sunlight.

Standard procedures are available for testing the colour fastness under different conditions (Saravanan and Chandramohan 2011; Kumaresan *et al.* 2011; Kumaresan *et al.* 2012; Sahoo *et al.* 2014). Colour fastness to washing Test -1 (IS 687:1979) determines the effect of washing on the colour fastness of the textiles. The reagent used was neutral soap (05 g/l). The size of the test specimen was 10 cm × 4 cm, which was sandwiched in between the two adjacent, undyed test cloth pieces (cotton and silk) and stitched along all the four sides. Each composite specimen was separately placed in the container with a soap solution (MLR of 1:50), and was preheated (40±2°C). Then the samples were agitated for 30 min in launderometer (digiWash SS™) with 40±2 rev.min^{-1}. The samples were then removed and rinsed in cold water. The stitches were ripped out along the two long sides and one short side. The composite specimen was opened and dried in air at room temperature. SDC Grey scale (Make: Paramount, ISO 105 A02 1993 BS EN 20105-A02 1995) was used to determine the change in colour of the treated test specimen and the degree of staining of the two pieces of the adjacent fabrics. The ratings of 1 to 5 was used in which 1 indicates very poor and 5 indicates excellent (Kumaresan *et al.* 2012; Saravanan *et al.* 2013; Sahoo *et al.* 2013).

Colour fastness to washing Test -2 was done as per IS 3361:1979 (BIS 1979; Sahoo *et al.* 2014). The method was same as above except that the temperature was maintained at 50±2°C and the time was 45 min. Neutral soap (05 g/l) was used as the reagent.

Colour fastness to sunlight was tested as per IS 686:1985 (BIS, 1985). Colour fastness to rubbing/crocking was tested as per IS 766:1998, which is based on ISO 105/X-1984. Two test specimen from each fabric sample was used, one each for dry and wet tests. The procedures as followed earlier by Sahoo *et al.* (2013) were followed in the present study. To assess the colour change and the staining for both these tests, SDC grey scale was used.

Antimicrobial activity

Textile materials and garments are susceptible to microbial attack, as these provide large surface area and absorb moisture required for microbial growth (Sahoo *et al.* 2012; Sahoo *et al.* 2014). The growth of microbes often results in objectionable odour, dermal infection, product deterioration, allergic responses and often related diseases. Geetha *et al.* (2011) also mentioned that antibiotic susceptibility tests were used to determine the inhibitory activity of the antibacterial agent against bacteria. As the jackfruit plant also has many medicinal properties, the dye obtained from the jackfruit bark could also have some antimicrobial effect. Thus, the sensitivity of the jackfruit bark dye for four different strains of bacteria and two fungi were also studied.

The extracted dye solution was put to agar test with four strains of bacteria, viz. Escherichia coli (E. coli), Streptococcus sp., Staphylococcus leuteus and Salmonella sp., and two strains of fungi, viz. Aspergillus nigricans and Candia albicans as per Kirby-Bauer disk diffusion susceptibility test (Bauer *et al.* 1966; Jorgensen and Turnidge, 2007; Sharma *et al.* 2011; Geetha *et al.* 2011). The detailed procedure is available in Sahoo *et al.* (2014). The Kirby-Bauer test results were interpreted using a table that related zones diameter to the degree of microbial resistance.

RESULTS AND DISCUSSION

Variation in colour of dyed fabrics

As discussed earlier, the silk fabrics were treated with six different mordants, viz. citric acid ($C_6H_8O_7$), alum ($KAl(SO_4)_2.12H_2O$), ferrous sulphate ($FeSO_4$), aluminum sulphate ($Al_2(SO_4)_3$), copper sulphate ($CuSO_4$), and potassium dichromate ($K_2Cr_2O_7$) at three different concentrations, viz. 1%, 2% and 3% and were then coloured with the dye derived from the jackfruit bark. The different colour shades obtained for the treatment combinations were compared. It was observed that there was extensive variation in the colours depending on the type of the mordant and its concentration. Kumaresan *et al.* (2011, 2012) and Sahoo *et al.* (2012) also observed that the types of mordants and their concentrations caused variations in the colour of silk fabrics dyed with different natural dyes.

The effect of the mordants and their level of concentrations on the changes in colour (ΔE^*), chroma value (ΔC^*) and lightness/ darkness (ΔL^*) obtained for the dyed cloth are shown in Fig. 2. The mean colour parameters of undyed silk were observed to be L*: 88.30±0.84, a*: 2.52±0.14 and b*: -2.24±0.44.

As observed from Fig. 2, the increase in the level of concentration of mordants up to 2% level increased the ΔE^* values for the dyed samples for alum, aluminum sulphate and copper sulphate, whereas for the other mordants, further increase to 3% level increased the ΔE^* values. In case of citric acid, the maximum ΔE^* was in the 1% level, which reduced with increase in the level of mordants. In all cases, the unmordanted samples had the minimum ΔE^* values. Sahoo *et al.* (2012), in a study conducted for mahua bark dye, found out that the maximum ΔE^* value of 52.95±0.29 was observed for 3% $CuSO_4$ mordant treated samples; the values were not significantly different from 2% and 1% $CuSO_4$ treated samples.

The change in chroma values were almost in a similar pattern as the ΔE^* values. It indicated that there was uniformity in change in the darkness of the samples, which were also further validated by comparing the ΔL^* values.

The statistical analysis of the effects indicated that both treatments and their levels caused significant differences in the colour. The maximum change in colour with respect to undyed silk fabrics was for 2% alum treated samples with the ΔE^* value of 27.08±0.29. However, the values were not significantly different from samples treated with $FeSO_4$ mordant at 2% and 3% levels, $CuSO_4$ mordant at 2% level and alum at 3% level. The change in chroma (ΔC^*) value was maximum for the alum mordant (@ 2%) (24.44±0.2), though the value was not significantly different from the alum mordant at 3%. In general, the ferrous sulphate mordant exhibited the highest degree of colour. The ΔL^* values indicated that $CuSO_4$ mordanted samples were the lightest followed by those treated with alum.

Colour fastness

Table 1 gives the effect of washing, rubbing and the exposure to sunlight on colour fastness of jackfruit

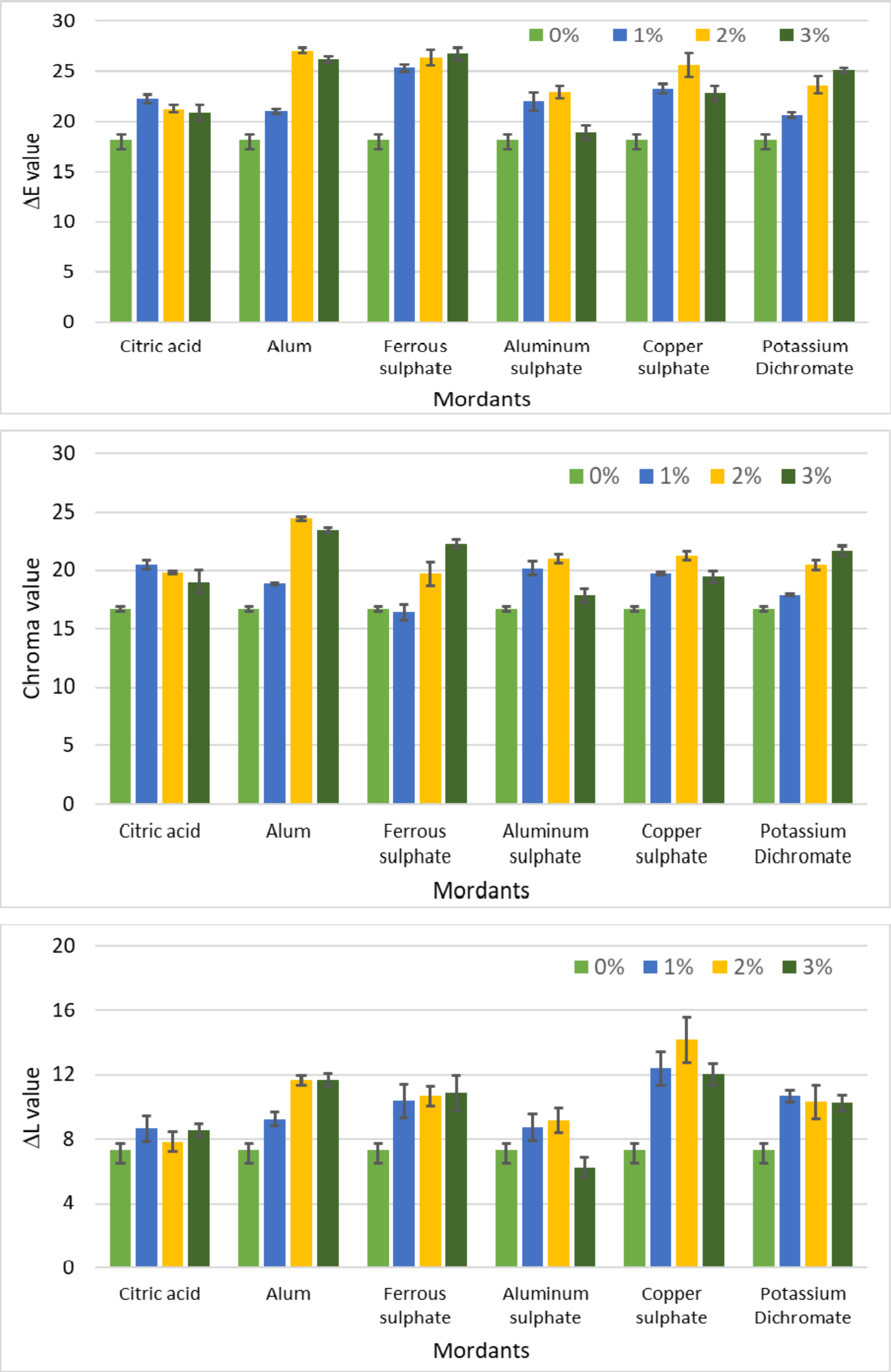

Fig. 2: HunterLab colour parameters of jackfruit bark treated samples

Table 1: Colour fastness of samples dyed with jackfruit bark

Name of Mordant	Mordant concentration	Light fastness	Colour fastness to washing (Test 1)		Colour fastness to washing (Test 1)	Colour fastness to washing (Test 2)			Rubbing fastness Test	
			Change in colour	Stain on cotton	Stain on silk	Change in colour	Stain on cotton	Stain on silk	Dry rubbing	Wet rubbing
No mordant		**2**	**3/4**	**5**	**5**	**3**	**5**	**4**	**4**	**4**
Citric acid	1%	3	4/5	5	5	4	5	5	4/5	4/5
	2%	3	4/5	5	5	4	5	5	4/5	4/5
	3%	3	4/5	5	5	4	5	5	4/5	4/5
Alum	1%	4	4	5	5	4	5	5	4/5	4/5
	2%	4	4	5	5	4	5	5	4/5	4/5
	3%	4	4/5	5	5	4	5	5	4/5	4/5
$FeSO_4$	1%	4	4/5	5	5	4	5	5	4/5	4/5
	2%	4	4/5	5	5	4/5	5	4/5	4/5	4/5
	3%	4	4/5	5	5	4/5	5	4/5	4/5	4/5
$Al_2(SO_4)_3$	1%	3	4/5	5	5	4	5	5	4/5	4/5
	2%	3	4/5	5	5	4/5	5	5	4/5	4/5
	3%	3	4/5	5	5	4/5	5	5	4/5	4/5
$CuSO_4$	1%	4	4/5	5	5	4	5	5	4/5	4/5
	2%	4	4/5	5	5	4/5	5	4/5	4/5	4/5
	3%	4	4/5	5	5	4/5	5	4/5	4/5	4/5
Potassium dichromate	1%	4	4/5	5	5	4	5	5	4/5	4/5
	2%	4	4/5	5	5	4/5	5	4/5	4/5	4/5
	3%	4	4/5	5	5	4/5	5	4/5	4/5	4/5

Scores for washing fastness test: *5-Excellent; 4-Good, 3- Fair, 2-Poor, 1-Very poor*

Scores for staining: *1-Much change, 2-Considerable change, 3-Noticeable change, 4-Slight change and 5-Negligible change*

Score for light fastness test: *1- very poor; 2- poor; 3- moderate; 4- fairly good; 5- good; 6- very good; 7- excellent; 8- outstanding*

Parameters for colour fastness to washing (Test 1): *Temperature 40±2°C, agitation time 30 min with (40±2) rev/min*

Parameters for colour fastness to washing (Test 2): *Temperature 50±2°C, agitation time 45 min with (40±2) rev/min*

bark dyed samples. The loss of colour during laundering is referred to as the lack of wash fastness or bleeding of colour. As discussed, there is colour loss during laundering if dyes are held loosely by the fibre, and poor rub fastness may be very good example of the existence of the physically held dye molecules. As expected, the mordanted specimen exhibited better colour fastness compared to the samples on which no mordant was applied.

Colour fastness to washing (Test 1). All the mordants and concentrations excepting 1% alum offered good to excellent colour fastness, whereas samples without mordant and 1% alum treated samples had good colour fastness (4).

The samples mordanted with citric acid, $FeSO_4$, $CuSO_4$, $Al_2(SO_4)_3$ and potassium dichromate showed good to excellent fastness (scores: 4-5), whereas alum showed good fastness (score 4). All specimen including un-mordanted samples exhibited excellent colour fastness staining on undyed cotton. Staining on undyed silk fabric also had excellent colour fastness except samples without the application of the mordant.

Colour fastness to washing (Test 2). The alum and citric acid showed good fastness, including 1% level of other mordants. The other mordants, viz. $FeSO_4$, $CuSO_4$ $Al_2(SO_4)_3$ and potassium dichromate both at 2% and 3% levels showed good to excellent fastness (score 4-5). Staining on undyed cotton samples and non-mordanted samples had excellent fastness. Staining on undyed silk was good to excellent (4-5) and excellent (5) for 1% and 2% copper sulphate and $FeSO_4$ mordanted samples, respectively. In other mordanted samples, it was excellent with scores of 5.

Colour fastness to crocking. All the dyed specimen with mordants had excellent (5) and good to excellent (4-5) fastness to both dry and wet crocking except unmordanted samples, which had good (4) colour fastness.

Colour fastness to sunlight. The samples dyed with alum, aluminum sulphate, potassium dichromate and citric acid mordants exhibited fairly good (4) fastness, whereas those dyed with copper sulphate and ferrous sulphate exhibited good (5) fastness when exposed to day light. Unmordanted samples had moderate fastness.

Similar results on the colour fastness have been earlier observed by Kumaresan *et al.* (2011, 2012) and Sahoo *et al.* (2012). However, Sahoo *et al.* (2012) observed that alum did not offer acceptable fastness to the washing for mahua dye. They further observed that the silk fabrics dyed with mahua bark dye with alum, $Al_2(SO_4)_3$ and citric acid as mordants exhibited moderate light fastness whereas specimen dyed with $CuSO_4$ as mordant exhibited fairly good colour fastness. Saravanan and Chandramohan (2011) also observed that different chemical mordants such as copper sulphate, ferrous sulphate, alum, potassium dichromate, nickel sulphate and stannous chloride and natural mordants as myrobolan were useful in maintaining the colour fastness of silk dyed with the barks of the peepal (*Ficus religiosa. L.*) tree. The scale of fastness varied with the type of the mordant and the mordanting method.

Thus, in view of the above, viz. fastness for washing, rubbing and the exposure to light, the jackfruit dye can be applied with any of the selected mordants at 3% level.

Antimicrobial property

It was observed that the selected strains of bacteria and fungi were resistant to the jackfruit dye. An earlier study by Bhuiyan *et al.* (1997) has indicated that the aqueous extracts of neem bark showed no detectable antimicrobial activity whereas the acetonic extract (<1% w/v) produced appreciable antimicrobial effects. Similarly, Nimbekar *et al.* (2012) also observed that the pattern of inhibition varied with the solvent used for extraction and the microorganism tested. The methanolic extract showed significant antibacterial activity. Thus, the method of extraction also plays an important role on the antimicrobial effect and hence the antimicrobial properties of the jackfruit dye need further investigation.

CONCLUSION

- All the mordants and concentrations were found to significantly affect the colour intensity, variations and fastness properties of silk coloured with jackfruit bark dye. The maximum change in colour with respect to the undyed silk fabrics was for 2% alum treated samples with the ΔE^* value of 27.08±0.29. However, the values were not significantly different from the samples treated with $FeSO_4$ mordant at 2% and 3% levels, $CuSO_4$ mordant at 2% level and alum at 3% level.
- The change in chroma (ΔC^*) value was maximum for the alum mordant (@ 2%) (24.44±0.2). The ΔL^* values indicated that $CuSO_4$ mordanted samples were the lightest followed by those treated with alum.
- All the mordants and concentrations excepting 1% alum offered good to excellent colour fastness, whereas the samples without mordant and 1% alum treated samples had good colour fastness.
- All the dyed specimen with mordants had excellent (5) and good to excellent (4-5) fastness to both dry and wet crocking except unmordanted samples, which had good (4) colour fastness. The samples dyed with alum, aluminum sulphate, potassium dichromate and citric acid mordants exhibited fairly good (4) fastness, whereas those dyed with copper sulphate and ferrous sulphate exhibited good (5) fastness when exposed to day light. Unmordanted samples had moderate fastness.
- In view of the above, viz. fastness for washing, rubbing and exposure to light, the jackfruit dye can be applied with any of the selected mordants at 3% level.
- The jackfruit bark dye almost offered no resistance to the growth of bacteria species taken for the study. However, further studies are required to confirm this effect.

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