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Colour Intensity, Fastness and Antimicrobial Characteristics of Silk Fabric Dyed with Mahua Bark

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Abstract:
A study was conducted to analyse the shades and colour fastness property of silk fabrics dyed with colour extracted from mahua bark at different concentrations of mordants. Four types of mordants, viz. $CuSO_4$, $Al_2(SO_4)_3$, alum and citric acid, were used for the study. The colours of the fabrics were quantified with the help of Hunterlab colour scale. For mahua bark dye, the maximum ΔE* value (52.95±0.29) was observed for 3% $CuSO_4$ mordant treated samples. However, the values were not significantly different from 2% and 1% $CuSO_4$ treated samples. The change in chroma was maximum for the 3% $Al_2(SO_4)_3$ mordant (34.89±0.33), though that offered by 1% and 2% $Al_2(SO_4)_3$, and 1% and 3% $CuSO_4$ were not significantly different from the above. The colour fastness property was measured in a scale of 1-5 (very poor, poor, fair, good and excellent). All the mordants except alum helped in maintaining good to excellent fastness to washing. The staining on undyed silk exhibited excellent colour fastness for all three mordants except $CuSO_4$, which showed good to excellent colour fastness. The dry crocking and wet crocking fastness characteristics were also compared for all the treatments. The specimen dyed with alum, $Al_2(SO_4)_3$ and citric acid exhibited moderate light fastness whereas specimen dyed with $CuSO_4$ exhibited fairly good colour fastness. The samples without mordants showed poor fastness. The antimicrobial profile indicated that the mahua bark dye was not sensitive to the growth of *E. coli, Streptococcus sp.* and *Salmonella* as indicated by the poor zone of inhibition. However, it was sensitive to *S. leuteus.* Similarly, the fungal isolates, viz. *Aspergillus nigricans* and *Candida albicans* used for the study were resistant to the dye.

Keywords: chroma value, colour fastness, crocking fastness, mordant

1.0 Introduction:
Natural dyes produce very uncommon, soothing and soft shades as compared to synthetic dyes. On the other hand, synthetic dyes, which are widely available at cheaper prices and which produce a wide variety of colours, sometimes cause skin allergy and other harms to human body, produce toxicity/chemical hazards during their synthesis, release undesirable and perilous chemicals. Hence, there is increased interest and emphasis toward use of textiles (preferably natural fibre product) dyed with eco-friendly natural dyes, and more and more plant materials are being explored for production of dye materials (Samanta and Agarwal 2009). Most of the commercial dyers and textile export houses have started re-looking at the possibilities of using natural dyes for dyeing and printing of different textiles for targeting niche market. Mahua (*Madhuca indica)* is one such plant, the bark of which can be used for extraction of dye. As the plant is commonly seen in the natural ecosystem of the Indian sub-continent, the availability of the raw material should not pose a problem. However, relevant scientific studies on standardisation of dyeing methods, dyeing process variables, dyeing kinetics and test of compatibility of selective natural dyes have become very important.

Natural dyes are mostly non-substantive and must be applied on textiles by the help of mordants, usually a metallic salt, having an affinity for both the colouring matter and the fibre. These metallic mordants, after combining with dye in the fibre, form an insoluble precipitate and thus, both the dye and mordant get fixed and become wash fast to a reasonable level. The concentration of mordants directly affects the colour of the dyed fabric and by varying the concentration, the intensity of the colour can also be changed. Another important aspect is that the textile materials and clothing are known to be susceptible to microbial attack, as these provide large surface area and absorb moisture required for microbial growth. Due to the fact that many natural plant materials can inhibit the growth of microorganisms, traditionally different plants have been used as natural dyes in textile and carpet industries. It is also believed that these dyes are less allergic and more stable than the chemical ones. As the mahua plant also has many medicinal properties, the dye obtained from the mahua bark could also have some antimicrobial effect, which can prove beneficial for the textile manufacturer and users.

Therefore, we planned to study the intensity and fastness property of colour of silk fabrics dyed with colour extracted from mahua bark at different concentrations of mordants. The anti-microbial profile was also studied to explore the possibility of its use in antimicrobial clothing. Mahua (*Madhuca indica*) trees are commonly found in the natural ecosystem of India, particularly in the tribal regions and its successful use as colour has a great commercial importance for the tribal people of the country.

2.0 Materials and Methods:

2. 1 Extraction of Dye:

The barks of mahua trees were collected from Daringibadi in Kandhamal district of Odisha. The barks were then cleaned and dried under shade until the equilibrium moisture content. The moisture content of the bark in the equilibrium condition was observed to be between 9.89 and 12.36 g water per g dry matter, as determined by hot air oven. Subsequently, the barks were chopped into tiny pieces manually and then ground into powder form (200 mesh) by a pulveriser, taking care that the powder was not heated beyond 55°C. The powdered dye was taken in water in a ratio of 1:10 (w/w basis, i.e.100 g powder in 1litre water) and was boiled under pressure for 1 hour. Thus, it was a 10% stock solution (final water content was adjusted to that level). The liquid was filtered by 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.

Mahua bark

After preliminary size reduction

Pulverised bark

Extracted Dye

Fig. 1 Different stages of the preparation of dye from mahua bark

2.2 Measurement of Optical density (λ_{max})

The dye solution was subjected to light of wavelength 300-700 nm using UV visible spectrophotometer (Perkin-Elmer) to find out the optical density (OD) value. The wave length at its highest peak optical density obtained was taken as the suitable wave length (λ_{max}) for calculating dye absorbency for the colour.

2.3 Collection and degumming of silk sample

White mulberry silk fabric was obtained from the Odisha Cooperative Tasar and Silk Federation Limited, Bhubaneswar. Degumming of silk before dyeing is required because the natural gum sericin present in the silk reduces dye absorption as well as the lusture of the fibre. 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 was continued for one hour. Then the silk fabric was taken out from degumming bath and squeezed to remove the excess liquid and thereafter rinsed under running water to make it free from traces of detergent and other chemicals. Then it was dried under shade.

2.4 Mordanting

Majority of the natural dyes require a chemical, usually a metal salt or other cross linking agent, to create an affinity between the fibre and the dye. These chemicals are known as "mordants". Four mordants, viz., alum (K_2SO_4,$Al_2(SO_4)_3$,$24H_2O$), copper sulphate ($CuSO_4$), aluminum sulphate ($Al_2(SO_4)_3$) and citric acid ($C_6H_80_7$), were used for the study. The mordanting of the fabrics can be done in three ways as pre-mordanting, post mordanting and simultaneous mordanting; however, the pre-mordanting process is more efficient and commonly used (Samanta and Agarwal, 2009; Saravanan and Chandramohan, 2011). Thus, the same was adopted in the present study. The quantity of mordant was taken at three levels viz. 1, 2 and 3% of the weight of the fabric. Known quantity of mordant was added to distilled water to get the material-liquid ratio (M:L) as 1:40 and was dissolved completely. Degummed silk fabric was put into mordant bath at normal temperature. After that temperature was raised up to 90°C for 30 minutes. The mordant solution was allowed to cool and the sample was dried within the laboratory by normal air circulation (Kumaresan *et al.,* 2012).

2.5 Dyeing silk fabric

The silk fabric was dyed using open dye beaker baths with M:L:: 1:40 at 90°C temperature for one hour. The dyed samples were allowed to cool up to 50°C and then washed by running water to remove the unfixed dye particles, un-reacted mordanting agents and any extra deposits from the surface. Then soaping by non-ionic detergent (NID) for 10 minutes was carried out to remove remaining particles and other chemical reagents. The samples were dried in the laboratory by air circulation.

2.6 Measurement of colour

The colour values of the samples were measured in the HunterLabcoloriometer with the CIE L*a*b* colour scale. The maximum for L* is 100 which represents a perfect reflecting diffuser. The minimum for L* is zero which represents black. Positive 'a' is red, negative 'a' is green. Positive 'b' is yellow and negative 'b' is blue. The color axes are based on the fact that a colour can not be both red and green, or both blue and yellow, because these colours oppose each other. The average colour values for the samples were recorded and the total colour difference ΔE* was calculated, which was a single value that takes into account the differences between the L*, a* and b* of the sample and standard.

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

The change in chroma value was also calculated as follows.

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

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

2.7 Colour fastness tests

Colour fastness of the dyed samples was assessed for washing, rubbing and exposure to sunlight. The loss of colour during laundering is referred to as lack of wash fastness or bleeding of colour. The loss of colour occur during laundering if 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. During dyeing it is very common that after saturation some dye molecules may be held superficially on the fabric. It is essential to wash off these dye molecules with appropriate post-treatments. Poor rub fastness may be very good example of existence of physically held dye molecules.

2.7.1 Colour fastness to washing Test -1 is designed to determine the effect of washing on the colour fastness of the textiles. The specimens were tested as per IS/ISO 105-C 10: 2006 (BIS, 2006). The reagent used was neutral soap (05 g/l). The test specimen of 10 cm X 4 cm was placed in between the two adjacent, undyed test cloth pieces (cotton and silk) and stitched along all four sides to form a composite specimen. Each composite specimen was placed in the container separately and necessary amount of soap solution was added to it to give material:liquid:: 1:50, which was preheated (40±2°C). The composite samples were agitated for 30 minutes in launderometer (digiWashSS™) with (40±2) rev/min. Then the composite specimen was 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. The change in colour of the treated test specimen and the degree of staining of the two pieces of adjacent fabrics was evaluated with the help of SDC Grey scale (Make: Paramount, IS/ISO 105 A-05-02: 1993 BS EN 20105-A02 1995) and the rating was assigned from 1 (very poor) to 5 (excellent) (BIS, 1996).

2.7.2 Colour fastness to washing Test -2 was done as per IS/ISO 105 C-10: 2006 (BIS, 2006). The method was same excepting the temperature and time of treatment. Here the temperature was 50±2°C and the time was 45 min. The reagent used was same neutral soap (05 g/l).

2.7.3 Colour fastness to sunlight was tested as per IS 686: 1985 (BIS, 1985). The test specimen (1cm x 6 cm) was placed along with the standard blue wool patterns, with scores 1 to 8, which denote light fastness ratings, where 1 is very poor and 8 is outstanding. One third portion of the test specimen and blue wool standards were covered with the help of opaque card sheet, and exposed to day light by mounting in the exposure rack. The rack was placed facing south in the northern hemisphere at an angle of 45°. The rack was exposed to sunlight from 9 am to 4 pm for 48 hours. The fastness was assessed by comparing the fading of the specimen with that of blue wool patterns. The scores for light fastness test were taken as 1(very poor) to 8 (outstanding).

2.7.4 Colour fastness to rubbing/crocking was tested as per 766: 1988 (BIS, 1988), which was based on ISO 105/X-1984. Two test specimen from each fabric sample was used, one each for dry and wet tests.

2.7.5 Dry crocking test: Two test specimen were placed on the base of the crock meter (Make: Paramount) so that it rested flat (on the abrasive cloth) with its long dimension in the direction of rubbing. The 5 cmX5 cm of dry undyed test cloth (cotton) was mounted over the end of the finger which projects downward from the weighed sliding arm. A spherical spiral wire clip held the test cloth in place. The finger was covered onto the test specimen and it was crocked back and forth 20 times by making 10 complete turns. The undyed test cloth was removed and evaluated.

2.7.6 Wet crocking test: The undyed (white colour) test cloth was thoroughly wetted in distilled water and then squeezed. Thereafter it was mounted on the finger. The remaining procedure was same as that of dry crocking test. To assess the colour change and the staining for both the above, SDC grey scale was used.

2.8 Antimicrobial activity test

The AGAR test of the extracted dye solution was carried out with four stains of bacteria, viz. Escherichia coli (E.coli), Streptococcus sp., Staphylococcus leuteus and Salmonella sp., and two stains of fungi, viz. Aspergillusnigricans and Candia albicans as per the standard procedure.

3.0 Results and Discussion:

3.1 Measurement of optical density (λ_{max}) of dyes

The optical density (λ_{max}) spectra of the mahua dye as obtained from the spectrophotometer are shown in Fig. 2. As mentioned in section 2.2, 300-700 nm wavelength was used to find out the optical density value (OD).The wavelength at its highest peak optical density obtained was taken as the suitable wave length (λ_{max}) for calculating dye absorbency for each colour. It was observed that the peak wavelength (λ_{max}) for the dyes were in the range of 300-400 nm.

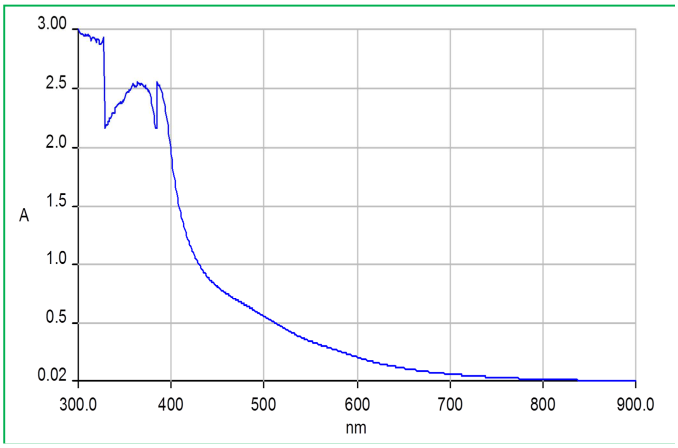

Fig. 2 Absorption spectra for mahua bark dye

3. 2 Colour of dyed silk fabrics

As mentioned earlier, the silk fabrics were treated with four different mordants viz. alum (K_2SO_4,$Al_2(SO_4)_3$,$24H_2O$), copper sulphate ($CuSO_4$), aluminum sulphate ($Al_2(SO_4)_3$) and citric acid ($C_6H_80_7$) at three different concentrations, viz. 1%, 2% and 3% and were then coloured with the dye obtained from the mahua bark. The different shades obtained on the silk fabric samples are shown in Fig. 3. It was observed that there was a wide variation in the colour depending on the type of mordant and its concentration.

3. 3 HunterLab colour parameters

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. 4. The mean colour parameters of undyed silk were observed to be L*: 88.277±0.843, a*: 2.46±0.143 and b*: -2.195±0.445. The positive values of ΔL* ($L^*_{initial\ value}$- $L^*_{final\ value}$) indicate that the fabric became darker after the treatment. The statistical analysis of the observations indicated that $CuSO_4$ exhibited higher ΔE* values than the other mordants and effect of each mordant was significantly different from each other. The ΔE* values were maximum for 3% mordant concentrations through there was no significant different with 2% concentration level (least significant difference 0.5258). However, the chroma value was maximum for the $Al_2(SO_4)_3$, followed by that of $CuSO_4$ mordants, they were significantly different, though. The change in chroma values were more for 3% mordant levels, which was not significantly different from 1% mordant level.

Considering the interaction effects of type of mordant and concentration thereof, the maximum ΔE* value (52.95±0.29) was observed for 3% $CuSO_4$ mordant treated samples. However, the values were not significantly different from 2% and 1% $CuSO_4$ treated samples. The change in chroma were maximum for the 3% $Al_2(SO_4)_3$ mordant (34.89±0.33), though that offered by 1 and 2% $Al_2(SO_4)_3$, and 1 and 3% $CuSO_4$ were not significantly different from the maximum value. The ΔL* values were higher for $CuSO_4$mordanted samples with 2 and 3% treatments attaining the maximum values (ΔL* value 41.34±1.25 for 2% and 41.01±0.49 for 3% $CuSO_4$).

3.4 Colour fastness test of dyed fabrics

Table 1 gives the effect of washing, rubbing and exposure to sunlight on colour fastness of mahua bark dyed samples. As expected, the mordanted specimen exhibited better colour fastness compared to samples on which no mordant was applied.

3.4.1 Colour fastness to washing (Test 1). All the mordants except alum helped in maintaining good to excellent (4-5) fastness to washing. Alum offered fair to good (3-4) fastness. The staining on undyed silk exhibited excellent colour fastness for all the three mordants except copper sulphate, which showed good to excellent (4-5) colour fastness.

3.4.2 Colour fastness to washing (Test 2). All four mordanted specimen had fair to good colour fastness whereas un-mordanted samples had poor to fair colour fastness. The staining on undyed cotton and silk exhibited excellent fastness and good to excellent fastness, respectively. Thus, mordanting, in general, helped in better retention of colour.

3.4.3 Colour fastness to crocking. Dry crocking colour fastness was found good to excellent for all dyed samples except those treated with 3% citric acid, which showed excellent fastness. For wet crocking, all the three mordants offered good fastness. Only 3% citric acid mordanted samples showed good to excellent colour fastness.

3.4.4 Colour fastness to sunlight. The specimen dyed with alum, aluminum sulphate and citric acid exhibited moderate (3) light fastness whereas specimen dyed with copper sulphate exhibited fairly good (4) colour fastness. The samples without mordants showed poor (2) fastness. In view of the above, viz. fastness for washing, rubbing and exposure to light, the mahua dye can be applied with copper sulphate and citric acid mordants at 3% level.

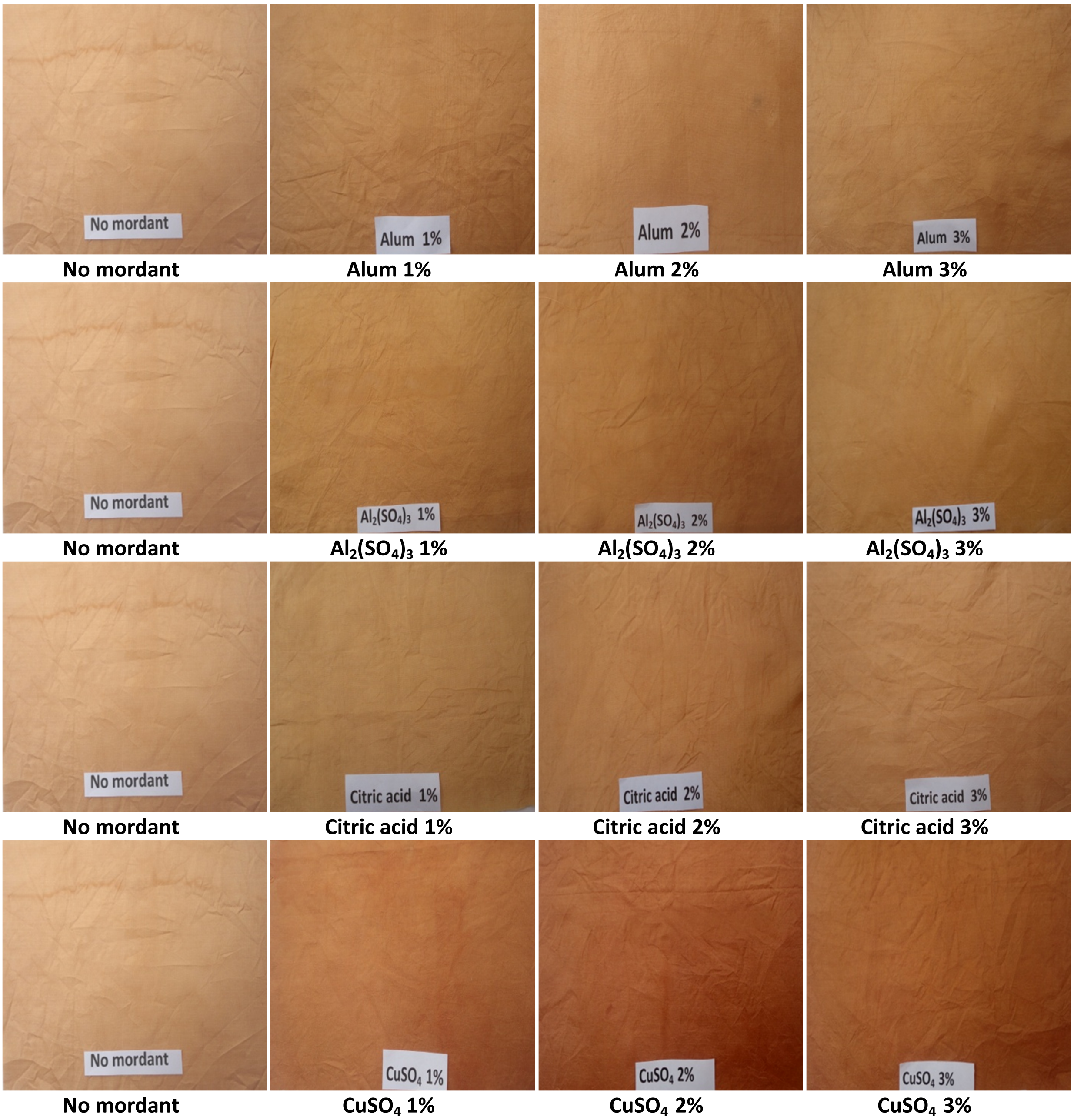

Fig. 3 Different shades obtained on silk fabric treated with different mordants and dyed with mahua bark

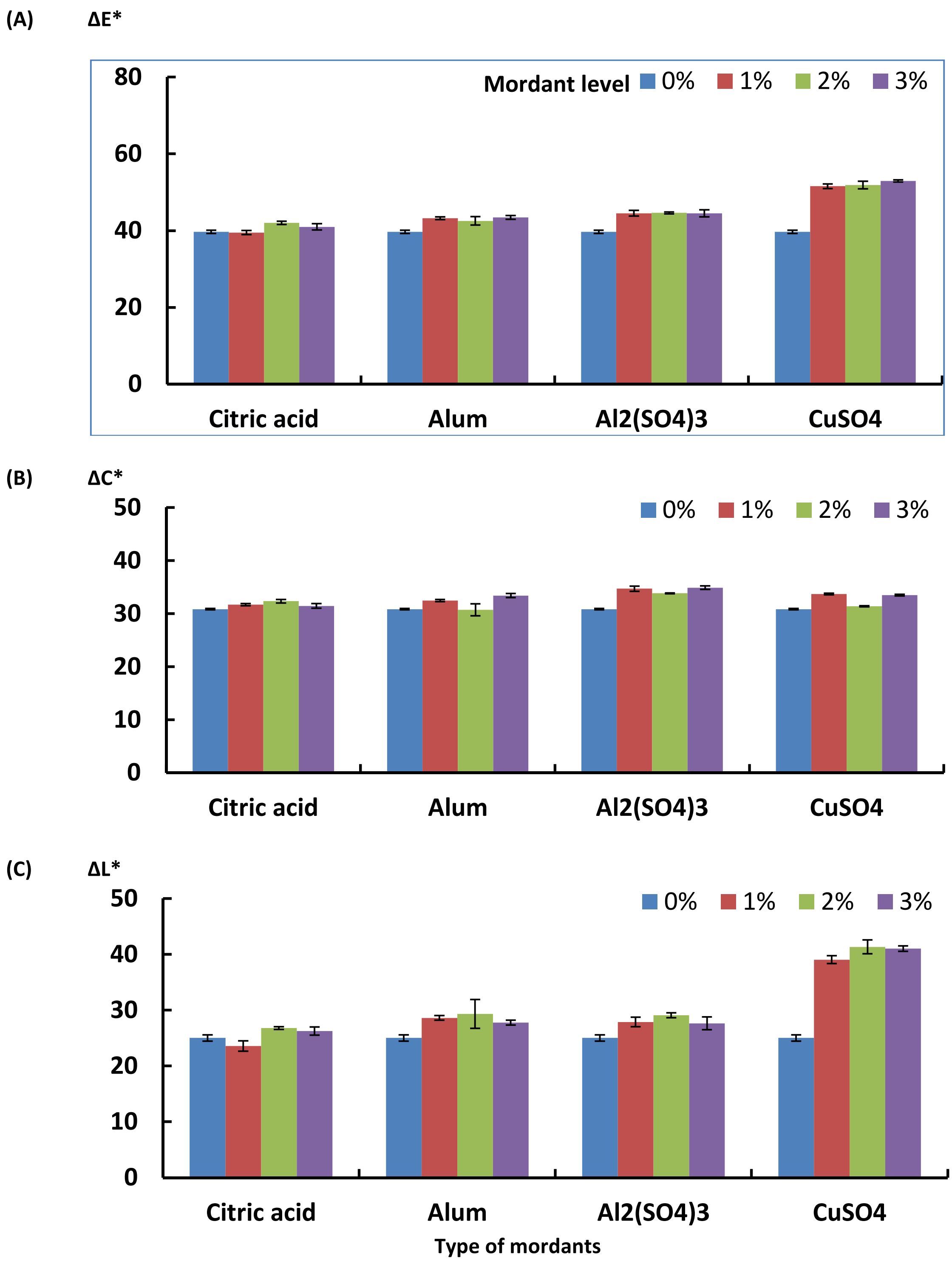

Fig.4 Hunterlab colour parameters of mahua bark treated samples

Table 1: Colour fastness of silk samples dyed with Mahua bark

Name of Mordant	Mordant concentration	Light fastness	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	4	2/3	4/5	4	4	3
Alum	1%	3	3/4	5	5	3	5	4/5	4/5	4
	2%	3	3/4	5	5	3	5	4/5	4/5	4
	3%	3	4	5	5	3/4	5	4/5	4/5	4
CuSO4	1%	4	4	5	4/5	3/4	5	4	4/5	4
	2%	4	4/5	5	4/5	4	5	4	4/5	4
	3%	4	4/5	5	4/5	4/5	5	4/5	4/5	4
Al2(SO4)3	1%	3	4/5	5	5	3/4	5	4/5	4/5	4
	2%	3	4/5	5	5	4	5	4/5	4/5	4
	3%	3	4/5	5	5	4	5	4/5	4/5	4
Citric acid	1%	3	4	5	5	3/4	5	4/5	4/5	4
	2%	3	4	5	5	3/4	5	4/5	4/5	4
	3%	3	4/5	5	5	4	5	5	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

3.5 Antimicrobial property test

The results obtained from the antibiogram profile of the bacterial isolate (Table 2) indicate that, out of the four different selected bacteria, the mahua bark dye was not sensitive to the growth of *E. coli*, *Streptococcus sp.* and *Salmonella sp.* as indicated by the poor zone of inhibition. However, it was sensitive to *S. leuteus, i.e.* the mahua bark dye offered selective sensitivity. Similarly it was also observed that the two fungi, the *Aspergillus nigricans* and *Candida albicans* were resistant to the mahua dye.

Earlier studies have indicated that the method of extraction also plays important role on the antimicrobial effect (Duddukuri *et al.,* 2011; Nimbekar *et al.,* 2012) and hence the antimicrobial properties of mahua dye need further investigation.

Table 2 Antimicrobial profile of bacterial isolates for mahua dye

Type of isolate	Zone size (mm)	Result
Bacteria isolates		
E.coli	0	R
S. leuteus	11	S
Streptococcus sp.	0	R
Salmonella sp.	0	R
Fungi isolates		
Aspergillusnigricans	0	R
Candida albicans	0	R

S: Sensitive, R: Resistant

4.0 Conclusion:

1. For mahua bark dye, the maximum ΔE* value (52.95±0.29) was observed for 3% $CuSO_4$ mordant treated samples. However, the values were not significantly different from 2% and 1% $CuSO_4$ treated samples. The change in chroma were maximum for the 3% $Al_2(SO_4)_3$ mordant (34.89±0.33), though that offered by 1 and 2% $Al_2(SO_4)_3$, and 1 and 3% $CuSO_4$ were not significantly different from the above value.
2. All the mordants except alum helped in maintaining good to excellent fastness to washing. The staining on undyed silk exhibited excellent colour fastness for all three mordants except $CuSO_4$, which showed good to excellent colour fastness.
3. Dry crocking colour fastness was found to be good to excellent for all dyed samples except those treated with 3% citric acid, which showed excellent fastness. For wet crocking, all the three mordants offered good fastness. Only 3% citric acid mordanted samples showed good to excellent colour fastness.
4. The specimen dyed with alum, $Al_2(SO_4)_3$ and citric acid exhibited moderate light fastness whereas specimen dyed with $CuSO_4$ exhibited fairly good colour fastness. The samples without mordants showed poor fastness.
5. In view of the colour fastness, the mahua dye should be applied with $CuSO_4$ and citric acid mordants at 3% level.
6. The mahua bark dye was not sensitive to the growth of *E. coli, Streptococcus sp.* and *Salmonella* as indicated by the poor zone of inhibition. However, it was sensitive to *S. leuteus.* Similarly, both the fungal isolates viz. *Aspergillus nigricans* and *Candida albicans* were resistant to the dye.

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