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Effects of selected mordants on colour characteristics of green coconut husk dye on silk

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ABSTRACT

Coconut husk is a common waste material thrown away by the street vendors and coconut based food processing industries. The disposal of this waste often involves huge cost for the industries. Hence, further value addition of this waste can be rewarding. In this context, an attempt was made to extract dye from green coconut husk and to study its colouring characteristics on silk. The natural dyes need the support of mordants to acquire the properties as colour fastness to washing, rubbing as well as exposure to light. The mordants also affect the shades of colour. Thus, the colour intensity and fastness properties of silk fabric was determined after treating the fabric with five mordants, viz. copper sulphate, ferrous sulphate, aluminum sulphate, alum and citric acid. Each mordant was used at three concentrations, viz. 1, 3 and 5%. It was observed that the different mordants and their concentrations affected the final shades of colours as well as the fastness properties. In view of the treatments considered in this study, the $CuSO_4$ and $FeSO_4$ mordants at 3 and 5% levels and alum at 5% level can be recommended for application on silk.

Key words: *Silk, Green coconut husk dye, Mordants*

Introduction

The green coconut husk is an important by-product of the coconut water processing industry. A huge amount of coconut husk is also thrown away by the street vendors, which often remains scattered on roadsides and open fields. Another add-on issue is that the coconut husk is not quickly biodegradable. In this context, further value addition of coconut husk can be very rewarding for the producers and processors and in addition to reducing the food processing wastes; it can also help in protecting environment. An important application of the green coconut husk could be in the preparation of dyeing materials, particularly for textiles. In the recent years, the emphasis on using natural dyes in the textile materials has been increasing because of environmental issues, to minimize the damage to the environment caused by the production and application processes used for the synthetic colours and the effluent produced. Kashyap *et al.* (2016) reported that the natural dyes are considered eco-friendly as these are renewable and biodegradable, are skin friendly and may also provide health benefits to the wearer. Most of the commercial dyers and textile export houses have been exploring the possibilities of using naturally obtained colours for dyeing and printing of different textiles for targeting niche market. Many studies have been conducted to obtain dyes from different natural sources and on their ap-

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plication on the textiles and other materials (Nawaz, 2019; Alam *et al*., 2020; Singhee, 2020).

For successful and effective application of natural dyes for any fibre, the appropriate and standardized techniques of dyeing, specific for that particular fibre need to be adopted. Besides, adoption of proper techniques/ procedures can help to obtain newer shades with acceptable colour fastness properties. Proper standardisation can also leadto reproducible colour yield. Further, it has been established that the natural dyes are mostly non-substantive and there is a requirement of using mordants (which are usually metallic salts) to act as bridging material to create substantivity of natural dyes. The metallic mordant, after combining with dye in the fibre, forms an insoluble precipitate and thus, both the dye and mordant get fixed and become wash fast to a reasonable level.

Thus, the study was planned to assess the feasibility of preparing natural dye from green coconut husk and to use different mordants to study the colour shades and colour fastness properties. Five mordants, viz. copper sulphate, aluminum sulphate, ferrous sulphate, citric acid and alum, were considered for study, each at three concentrations to test the applicability of these mordants for using the dye obtained from coconut husk dyes.

Materials and Methods

Preparation of dye

The green coconut husk was cleaned, and cut into smaller sizes by a laboratory cutter and then was shade dried until the equilibrium moisture content. The moisture content of the coconut husk was found to be 9-11% (wet basis), as determined by hot air oven. Subsequently, these materials were chopped into fine 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 dye was prepared as per the method suggested by Kumaresan *et al.* (2011) and Sahoo *et al.* (2012), in which the powdered dye was taken in water in a ratio of 1:10 (w/w basis) and was boiled for one hour under pressure in a pressure cooker. It led to the preparation of a 10% stock solution. A filter paper (Whatman No. 4) was used to filter the liquid. The dye was stored in a refrigerator till further use.

Collection and degumming of fabric

White mulberry silk fabric was degummed in a solution prepared by dissolving 5 $g.l^{-1}$ neutral soap and 1% (w/w) sodium carbonate in water with material liquid ratio of 1:40, as per Sahoo *et al.* (2014). The temperature of the bath was gradually increased to 90°C and after that it was maintained for one hour. After removing the silk fabric samples from degumming bath and squeezing to remove the excess liquid, these were further rinsed under running water to remove any residual detergent and other chemicals. Then the samples were dried under shade.

Mordanting and dyeing

The dye was used on the fabric with five different mordants, viz., copper sulphate ($CuSO_4$), ferrous sulphate ($FeSO_4$), aluminum sulphate ($Al_2(SO_4)_3$), alum (K_2SO_4, $Al_2(SO_4)_3$,$24H_2O$), and citric acid ($C_6H_8O_7$) using different concentrations. Each mordant was used at three concentrations of 1%, 3% and 5% of the weight of fabric. Pre-mordantingmethod was adopted with a material liquid ratio in the mordant solution as 1:40(Gulzarani and Gupta, 1992; Sahoo *et al.*, 2014). The degummed silk was kept in the mordant solution initially at normal temperature, after which the temperature was raised up to 90°C; it was held for 30 minutes at that temperature. The mordant solution was allowed to cool and the sample was dried indoor by normal air circulation.

The fabric samples were dyed using open dye beaker baths with material liquid ratio of 1:40 at 90 °C temperature for one hour. The dyed samples were allowed to cool down to about 50 °C and then were washed by running water to remove the superficially deposited or unfixed dye particles or unreacted mordanting residues. The samples were dried indoor. A sample was also dyed without using any mordant to check the differences.

Measurement of colour

The colour values of the samples were measured in the Hunter Lab colorimeter in the L, a and b scale (Merdan *et al.*, 2012; Teklemedhin and Gopalakrishnan, 2018; Aung *et al.*, 2020). The changes from the control, i.e. undyed fabric was denoted by ΔL, Δb, and Δb. The total colour difference ΔE was calculated as $\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2}$. The change in Chroma value (ΔC) was another parameter found out as $\Delta C = \sqrt{\Delta a^2 + \Delta b^2}$, which did not consider the change in lightness/darkness of the samples.

Measurement of colour fastness

The dyed samples were tested for the fastness of colour against washing, rubbing and exposure to sunlight (Kittinaovarat, 2004; Sati and Jahan, 2006). The colour fastness to washing Test-1 involved the procedure recommended as per IS/ISO 105-C10(BIS, 2006), in which neutral soap (5 $g.l^{-1}$) was used as the reagent. The test specimen of 10 cm × 4 cm was placed in between the two adjacent, undyed test cloth pieces and stitched along all four sides to form a composite specimen. The composite specimen was placed in the container separately and necessary amount of soap solution was added to give a material liquid ratio of 1:50. It was heated to 40±2°C and the samples were agitated in launderometer at 40±2 rpm for 30 min. Then the samples were removed and rinsed in cold water. After taking the dyed sample out from the composite specimen and then drying at room temperature, the change in color and the degree of staining of the two pieces of adjacent fabrics were evaluated with the help of SDC Grey scale as per ISO 105 A02 (BIS, 2021) and the rating was assigned. In the colour fastness to washing Test -2,the temperature was maintained at 50±2°C and the time of agitation was 45 min.

The IS test method: 686:1985 (BIS, 2016) was used for determination of colour fastness today light. The test specimen of 1 cm × 6 cm size was taken, one third portions of the test specimen and blue wool standards were covered with the help of opaque card sheet, and were exposed to sunlight from 9 a.m. to 4 p.m. for 48 h. Then the fastness was assessed by comparing the fading of the specimen with that of blue wool patterns. The fastness was represented with a score of 1(very poor) to 8 (outstanding).

The test for colour fastness to rubbing/ crocking was done as per ISO 105-X12:2001 (BIS 2019). For the dry crocking test, two test specimen were placed on the base of the crock meter so that these rested flat (on the abrasive cloth) with the long dimension in the direction of rubbing. The dry undyed test cloth (5 cm × 5 cm) was mounted over the end of the finger which projected downward from the weighed sliding arm. A spherical spiral wire clip was used to hold 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. Thereafter the undyed test cloth was evaluated. For the wet crocking test, the undyed test cloth was completely wetted with distilled water and squeezed, and then mounted on the finger. The remaining procedure was same as that of dry crocking test.

SDC grey scale was used for assessing the colour change and the staining (BIS, 1996).

Results and Discussion

Colour of dyed silk fabrics

The silk fabric samples were treated with five different mordants, as mentioned above, each at concentrations of 1%, 3% and 5% and then coloured with the dye. It was observed that the colours had different shades and intensities, which depended on the type of mordant and its concentration.The Hunterlab L, a and b values along with the change in colour (ΔE) and the Chroma value (ΔC) of the samples after dyeing with different mordants are given in Table 1. The colour parameters of undyed silk fabric was also found out and given in the table. As observed from the changes in L values, dyeing imparted darkness to the samples.

In general, the changes in L values (ΔL) were maximum for the $CuSO_4$ mordanted samples followed by the $FeSO_4$ mordanted ones. The variations in shades were not uniform with the different levels of concentrations of mordants. The statistical analysis revealed that the maximum ΔL value was for the $CuSO_4$ mordant at 3% level (31.72±0.91), which was not significantly different from that obtained at 5% level. The minimum ΔL value was for the alum mordant at 1% level (18.82 ± 1.00), and it was not significantly different from the $Al_2(SO_4)_3$ at 3% level.

The maximum changes in Δa and Δb values were also for the $CuSO_4$ mordanted samples. The statistical analysis also revealed that the $CuSO_4$ at 3% gave the maximum change in a value (15.08±0.33). The alum at 1% level gave the minimum change in a value (10.5±0.37), though there was no significant difference between this and $FeSO_4$ mordanted at 1% and 3% levels. $CuSO_4$ at 3% gave the maximum change in b value (23.46±0.396), though the statistical analysis revealed that this value was not significantly different from those values obtained for $CuSO_4$ at 1% and 5% levels. The $FeSO_4$ mordanted samples showed minimum change in b value; the $FeSO_4$ at 1% level showed Δb value as 19.4±0.51, though it was not significantly different from the $FsSO_4$ at 3% and 5% levels as well as alum at 1% level.

The changes in chroma value (ΔC), which considers both the parameters a and b, are shown in Fig. 1. The maximum changes were observed for the $CuSO_4$ samples. The $CuSO_4$ at 3% level showed the maximum change in Chroma value (27.889±0.50), which was not statistically different from the $CuSO_4$ at 5% level. The minimum value of Chroma was for the $FeSO_4$ 1% treated samples (22.175±0.554). The Chroma values for the $FeSO_4$ 3% and 5% levels as well as that for alum at 1% were not significantly different those treated with $FeSO_4$ at 1% level.

The changes in colour (ΔE) were maximum for the $CuSO_4$ treated samples (Fig. 2). It was observed that the maximum change in colour was for the $CuSO_4$ 3% mordanted sample (42.239±0.92), which was significantly different from the next highest, i.e. 5% level of the same mordant. The minimum change in colour was for alum 1% mordanted samples (29.392±0.97) and it was significantly different from the next, i.e. $Al_2(SO_4)_3$ at 3% level.

In view of the above analysis, it is concluded that the shades of the colour would be dependent on the mordants and their concentrations. As the consumers will be having different preferences for fabric colours, different types of mordants and different concentrations can be used to prepare the variety of shades. Kittinaovarat (2004) reported that the use of different mordants and mordanting methods affected the dye shade and depth of shade differently on the dyed fabrics both with and without chitosan. Arik *et al.* (2020) also observed that various colour

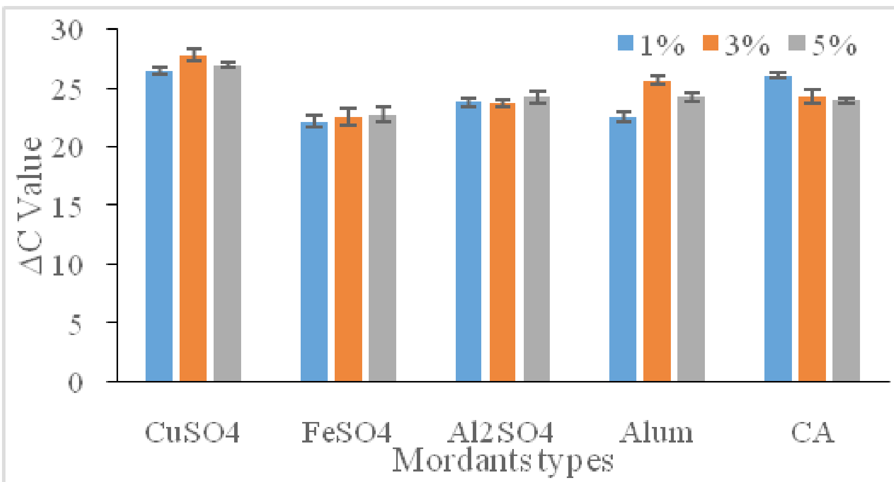

Fig. 1. Chroma values of the dyed silk samples

Table 1. Colour values of the silk fabric dyed with green coconut husk dyes

Mordant types	Levels, %	L	a	b	ΔC	ΔE
$CuSO_4$	1	61.88 ± 0.87	14.9 ± 0.16	24 ± 0.25	26.54 ± 0.29	38.78 ± 0.47
	3	58.42 ± 0.91	16.28 ± 0.32	24.72 ± 0.39	27.88 ± 0.5	42.23 ± 0.92
	5	59.94 ± 1.22	15.36 ± 0.23	24.18 ± 0.15	26.94 ± 0.19	40.47 ± 0.95
$FeSO_4$	1	65.06 ± 0.58	11.94 ± 0.32	20.66 ± 0.51	22.17 ± 0.55	33.48 ± 0.68
	3	63.46 ± 0.32	12.38 ± 0.46	20.8 ± 0.59	22.51 ± 0.74	34.91 ± 0.66
	5	61.02 ± 0.72	12.66 ± 0.41	20.96 ± 0.49	22.79 ± 0.63	36.98 ± 0.59
$Al_2(SO_4)_3$	1	68.88 ± 0.72	13 ± 0.24	21.96 ± 0.27	23.82 ± 0.33	31.93 ± 0.59
	3	69.76 ± 1.03	12.8 ± 0.27	21.94 ± 0.16	23.71 ± 0.27	31.27 ± 0.84
	5	68.8 ± 0.43	13.34 ± 0.4	22.26 ± 0.38	24.25 ± 0.52	32.3 ± 0.61
Alum (%)	1	71.32 ± 1	11.7 ± 0.36	21.24 ± 0.35	22.57 ± 0.47	29.39 ± 0.97
	3	67.66 ± 0.62	14.06 ± 0.24	23.46 ± 0.27	25.65 ± 0.34	34.11 ± 0.54
	5	69.14 ± 0.86	13.1 ± 0.31	22.4 ± 0.27	24.26 ± 0.36	32.09 ± 0.72
CA (%)	1	64.14 ± 0.85	14.68 ± 0.11	23.62 ± 0.18	26.1 ± 0.17	36.85 ± 0.55
	3	69.18 ± 0.74	13.12 ± 0.47	22.44 ± 0.39	24.3 ± 0.55	32.09 ± 0.88
	5	69.28 ± 0.29	13.2 ± 0.2	22 ± 0.14	23.96 ± 0.2	31.77 ± 0.2
Undyed silk sample		90.14 ± 0.215	1.2 ± 0.089	1.26 ± 0.53	-	-

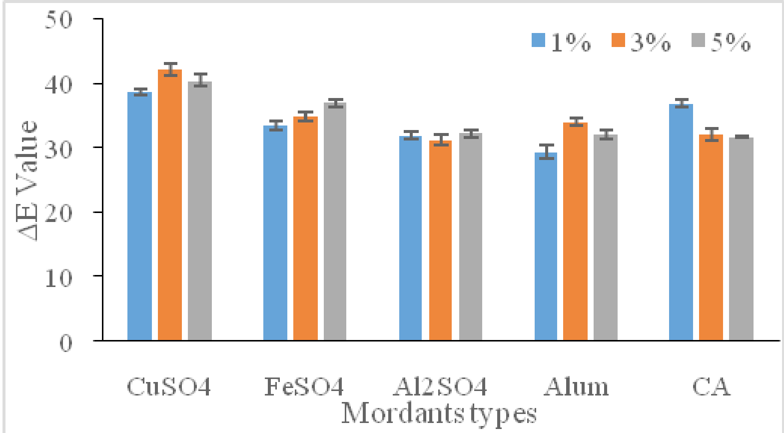

Fig. 2. Change in colour (ΔE) of the dyed silk samples

shades were obtained according to the mordanting materials and methods for dyeing with red cabbage extract dye.

Colour fastness

Table 2 gives the effect of washing on colour fastness of dyed silk samples. It was observed that the samples mordanted with $CuSO_4$, $Al_2(SO_4)_3$, $FeSO_4$ and citric acid as well as alum at 5% showed good to excellent fastness, whereas alum at 1% and 3% showed good fastness. The unmordanted sample showed fair to good fastness. The stain on silk was found to be negligible for all mordants and unmordanted sample.

The washing fastness (test 2) also revealed that the change in colour was good for $CuSO_4$, $Al_2(SO_4)_3$, and $FeSO_4$ and citric acid mordants, each at 1% level. However, 3% and 5% levels all these mordants exhibited good to excellent scores. For alum, the colour change was rated good for 1% and 3% levels and good to excellent for 5% level. The stain on silk was slight for unmordanted silk, and slight to negligible for $CuSO_4$ at 3% and 5% levels. For all other treatments the stain on silk was considered negli-

Table 2. Colour fastness of silk samples dyed with coconut husk to washing

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

Scores for washing fastness tests: 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

Table 3. Colour fastness of silk samples dyed with coconut husk to exposure to light and rubbing

Name of Mordant	Mordant concentration	Light fastness	Rubbing fastness Test	
			Dry rubbing	Wet rubbing
No mordant		2	4	4
$CuSO_4$	1%	4	4/5	4/5
	3%	4	4/5	4/5
	5%	4	4/5	4/5
$FeSO_4$	1%	4	4/5	4/5
	3%	4	4/5	4/5
	5%	4	4/5	4/5
$Al_2(SO_4)_3$	1%	3	4/5	4/5
	3%	3	4/5	4/5
	5%	3	4/5	4/5
Alum	1%	4	4/5	4/5
	3%	4	4/5	4/5
	5%	4	4/5	4/5
Citric acid	1%	3	4/5	4/5
	3%	3	4/5	4/5
	5%	3	4/5	4/5

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

Scores for rubbing fastness tests: 5-Excellent; 4-Good, 3- Fair 2-Poor, 1-Very poor

gible. In an earlier study by Sahoo *et al.* (2014), it was observed that the different mordants exhibited different washing fastness. Venilla and Santhy (2008) studied fastness properties of natural dye from tamarind leaves and found that colour change of cotton fabrics dyed with tamarind leaves after washing showed that the alum had higher fastness rating followed by copper sulphate or ferrous sulphate ranked next.

Table 3 gives the effect of rubbing and exposure to sunlight on colour fastness. The light fastness was fairly good for $CuSO_4$, $FeSO_4$ and alum mordanted samples and moderate for $Al_2(SO_4)_3$ and citric acid for all levels of mordants. The light fastness of the unmordanted sample was poor. The dry and wet rubbing fastness were found to be good to excellent for all dyed samples, whereas un-mordanted samples showed good crocking fastness.

Thus, from the above observations, it was observed that the light fastness was achieved up to fairly good level for the dye, which was for the $CuSO_4$, $FeSO_4$ and alum mordanted ones. The most acceptable conditions for colour fastness to washing tests I and II were for $CuSO_4$, $Al_2(SO_4)_3$, $FeSO_4$ and citric acid at 3% and 5% levels and alum at 5% level. The rubbing fastness tests gave good to excellent ratings for all mordants. In view of the treatments considered in this study, the $CuSO_4$ and $FeSO_4$ mordants at 3 and 5% levels and alum at 5% level can be recommended as these had the most acceptable traits so far as the light fastness and other parameters are considered.

Based on the above study, it can be concluded that the green coconut husk can be used as a raw material for preparation of dye for silk and the final colours will depend on the type of mordant and its concentration. $CuSO_4$ mordanted samples yielded the maximum change in colour out of the five selected mordants. The light fastness could be achieved up to fairly good level with the $CuSO_4$, $FeSO_4$ and alum mordants. $CuSO_4$, $Al_2(SO_4)_3$, $FeSO_4$ and citric acid at 3% and 5% levels and alum at 5% level could be used for most acceptable colour fastness to washing tests I and II. The rubbing fastness was good to excellent for all mordants. In view of the treatments considered in this study, the $CuSO_4$ and $FeSO_4$ mordants at 3 and 5% levels and alum at 5% level can be recommended for application on silk.

Conflict of Interests: Authors have declared that there is no conflict of interests.

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