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Effect of Mordant Types and Methods on the Color Fastness Properties of Silk Fabrics dyed with Brown Seaweeds

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Abstract**:** ***Natural dyes come from plenty of sources including plants, animals, insects or microorganisms. At present, natural dyes are highly demanded in textile application. In this study, brown seaweed (Sargassum spinosum) extracts were utilized for silk coloration with the use of metallic salts mordants (Iron II Sulphate and Aluminium Sulphate) and biomordants (cinnamon and chitosan). Dyeing was performed with pre-mordanting and simultaneous mordanting methods. The dyed fabrics were analyzed in terms of color fastness properties to washing, perspiration, rubbing and light. The results revealed that seaweed dyes with cinnamon mordant gave darker shades and higher absorbency towards silk which is comparable to iron mordant. The approach of using biomordant is parallel to the sustainability demand of natural dyes in textile application.***

Keywords**:** ***Sargassum sp., brown seaweed, natural dyes, biomordant, metallic salts mordant, color fastness.***

I. INTRODUCTION

Mordant which is acting as a fixer is needed in dyeing process. The usage of toxic and carcinogenic mordants which contribute to contamination and harmful effects to environment is not preferred nowadays. Thus, an alternative to metallic salts mordant is needed. Advancements in mordanting processes and selecting new, safe and green mordants in exchange to conventional heavy metal ions has been a vital part in the improvement of natural dyeing developments. The use of biomordants to replace metallic salt mordants is recommended by researchers because of its effectiveness and safer substitute seeing the ecological aspect of contamination and their environmental nature, thus can be settled to the environment deprived of any physical or chemical after-treatment (e.g. precipitation or filtration). Improving traditional mordanting processes and selecting new mordants to replace traditional heavy metal ions has been an important part in the development of natural dyeing of textiles [1]. Biomordant such as tannin is highly recognized as a good natural mordant that gives iron mordant-like effect after dyeing process [2] – [5].

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II. MATERIALS AND METHODS

A. Materials

Seaweed samples of *Sargassum sp.* were collected from Semporna, Sabah. Lightweight 100% silk fabric (Table 1) was used as the substrate during the dyeing experiment. Iron II Sulphate and Aluminium Sulphate (Alum) were used as metallic salt mordant and purchased from R&M Chemicals. Biomordants used in this study were tannin from cinnamon and chitosan from crab shell.

B. Sample Preparation and Extraction

Sargassum sp. seaweeds were cleaned, oven dried for 48 hours at 50°C, ground and sieved into 1mm size. Then the samples were extracted using supercritical CO_2 extraction at optimized condition of 4500 psi and 66°C for 60 minutes [6] – [7]. The extracted product was further prepared into dye solution and kept in a freezer at 4°C prior dyeing process.

Table I- Fabric Specifications

Type of Fabric	Fabric Structure	Weight (g/m^2)	Thickness (mm)	Density (threads/inch)
100% Silk	5-end satin weave	66	0.34	Warp: 334 Weft: 122

C. Dyeing of Silk Fabric

Exhaustion dyeing method was used in this experiment. The dyeing process was carried out in a single dye bath with the mixture of the seaweed extracts, mordant, emulsifier and distilled water in a dye vessel. All samples were dyed at 80°C ± 2°C for 60 minutes (liquor ratio 1: 20) using Labtec Autowash. The constant parameters were the dyeing temperature, dyeing time, concentration of mordant (2%) and concentration of dyes (5%). The control sample was fabric dyed using *Sargassum sp.* dye without treatment of any mordants. Post-mordanting was not conducted in this work since preliminary studies on post-mordanting showed a very low color strength and low color fastness properties [8] – [11].

D. Pre-mordanting Dyeing Method

In this method, silk fabric was firstly treated with mordants for 60 min at 80°C ± 2°C. The mordanted samples were then rinsed in cold water before continued to dye in a new dye solution for another 60 min at 80°C ± 2°C. Then, the samples were rinsed using cold water and allowed to dry at room temperature.

E. Simultaneous Mordanting

Simultaneous mordanting involves dyeing and mordanting process at the same time. Silk fabrics were soaked in the dye solution containing mordant and the dyeing process lasted for 60 min at 80°C ± 2°C. After that, the samples were rinsed using cold water and allowed to dry at room temperature.

F. Color Measurement and Color Coordinates of *Sargassum sp.*

The dyed silk fabrics were measured for color strength (K/S) and color reflectance by HunterLab LabScan XE (LSXE) spectrophotometer and examined using HunterLab EasyMatchQC software. The K/S value is the value used to measure the strength of absorbency using data color spectrophotometer. The depth of color of the dyed fabric was determined by analysing the K/S value of a given dyed sample by Kubelka–Munk equation (Equation 1). The Kubelka-Munk equation defines the relationship between spectral reflectance (R in %) of the sample and its absorption (K) and scattering (S) characteristics as in equation 1:

$$\frac{K}{S} = \frac{[1 - 0.01R]^2}{2[\,0.01R]} \quad (1)$$

The colorant extracted from *Sargassum sp.* was analysed and expressed in terms of their L* a* b* values. The L* values designate perceived lightness or darkness. Value of 0 indicates black and 100 defines white. The values of a* indicate redness (+a) or greeness (-a), while value of b* shows yellowness (+b) or blueness (-b).

G. Color Fastness Properties of Dyed Silk

Color fastness is the resistance of a material (silk fabric for this particular study) to change any of its color characteristics or extent of transfer of its colorants to adjacent white materials in touch. The color fastness is usually rated either by loss of depth of color in original sample or it is also expressed by staining scale, for example the accompanying white material (plain silk/cotton) gets tinted or stained by the color of the original fabric. However, among all types of color fastness, light fastness, wash fastness and rub fastness are considered general for any textiles. Meanwhile, perspiration fastness is considered specific for certain apparels [9]. In this study, color fastness of dyed silk fabric was analysed in terms of washing fastness, rubbing/crocking fastness, light fastness and all methods were carried out in accordance with Malaysian Standard (MS ISO) adopted from ISO Standard. All samples were graded from scale 1(the worst) to 5 (the best) as indicated by AATCC. The summary of the tests is given in Table 2. The grey scale consists of nine pairs of non-glossy neutral grey colored chips that illustrate the perceived color differences. These scale were used to assess color change and to measure the staining amount referral to undyed fabrics during fastness tests.

H. Color Fastness to Washing

A sandwiched sample of size 10 cm x 4 cm was prepared for every dyed fabric. Then, a soap solution with liquor ratio 1:50 was prepared according to the weight of the sandwiched sample. All sandwiched samples were soaked in the soap solution in a vessel and rotated for 30 minutes at 60°C using Labtech Auto wash. After the process completed, the samples were left to dry in a hanging station followed by evaluation on the color changes and staining using the standard grey scale under the lighting cabinet.

I. Color Fastness to Perspiration

The test is to determine the fastness of colored textiles to the effect of perspiration (AATCC, 2005a). Color fastness to perspiration was carried out in accordance to MS ISO 105-E04-1996. A sandwiched sample sized 6 cm x 4 cm was prepared for every dyed fabric and soaked into the alkaline perspiration solution as described in Table 3. All sandwiched samples were stirred into alkaline soap solution for 30 minutes at 1: 50 liquor ratio. After that, the sandwiched samples were placed in between two plates of 11.5 cm x 6 cm with thickness 0.15 cm under the pressure of 4.5 kg mass weight in the perspirometer. Then, the perspirometer was kept in the oven at 37°C ± 2°C for 4 hours and left hang to dry later on. Finally, the samples were evaluated using grey scales under lighting cabinet.

Table II- Types of color fastness test according to standard method

Color Fastness Test	Standard method	Equipment
Washing	MS ISO 105-C01-1966	Auto-wash
	MS ISO 105-A05-2003	Change in Color
	MS ISO 105-A04-2003	Staining
Perspiration	MS ISO 105-E04-1996	Perspirometer
	MS ISO 105-A05-2003	Change in Color
	MS ISO 105-A04-2003	Staining
Rubbing/Crocking	MS ISO 105-X12-2001	Crockmeter
	MS ISO 105-A04-2003	Staining
Light	MS ISO 105-B02-2001	Light Fastness Tester

Table III- Alkaline perspiration solution

Ingredients	Amounts
L-histidine monohydrochloride mono-hydrate	0.5 gram
Sodium chloride	5 grams
Crystallized disodium hydrogen orthophosphate	5 grams
Distilled water	1 Liter
Sodium hydroxide to get pH 8.0 solution	0.1 N

J. Color Fastness to Rubbing

This test method was developed to measure the amount of color transferred from the surface of colored textile material onto other surfaces by rubbing (AATTCC < 2005b) in accordance to MS ISO 105-X12:2001.

A sized of 20 cm x 11 cm specimen was set and placed on the crock meter (refer appendix) a rubbing finger which was covered with 5 cm x 5 cm cotton cloth was rubbed to the specimen for ten times (of complete turns) at the rate of one turn per second for both dry and wet tests. At the end, color which stained on the white cotton cloths were evaluated under lighting cabinet using the standard grey scales.

K. Color Fastness to Light

The test provides general principles and basic procedures to determine color fastness to light for textile materials (AATCC, 2005c) correspond to MS ISO 105-B02:2001 using light fastness tester. In this test, all specimens were exposed to light until the color change in conjunction with a blue wool with specific ratings from 1 to 8 (Blue wool standard), which indicates that the higher the rating, the better is the fabric. AATCC Blue Wool Light fastness Standard is a group of dyed wool fabrics allocated by AATCC to be used in defining the light exposure's amount of specimens during the light fastness testing (AATCC, 2005d).

III. RESULTS AND DISCUSSION

A. Color Absorption for Dyed Silk

The color strength or depth of shade of *Sargassum sp.* dyed fabric was investigated by K/S value as represented by Equation 1. This equation represents the nature of the coloring material layer and as an easy way to determine a color as a concentration due to the light absorbing and scattering [12]. The function of K/S is directly proportional to the concentration of colorant in the substrate. The spectral plots and 2D plots are given in Figure 1 and Figure 2, respectively. Table 4 shows the dyeing sequence and codes used in the study.

Higher K/S values (Figure 1) indicate a greater amount of dyestuff in the fabric resulting in deeper shade. It is clear that all dyes show similar graph trends because they were from the same source. The K/S values were highest for silk dyed with Iron mordant in simultaneous mordanting method followed by silk dyed in simultaneous mordanting with cinnamon mordant. However, the value was low for silk dyed in pre-mordanted with alum and the lowest is for silk dyed with chitosan in pre-mordanting method.

These results are parallel with the observation of swatches of dyed silk (Table 6) which shows the darker shades are from iron mordanted (004, 005) fabrics followed by cinnamon mordanted fabrics (006, 007). Likewise, the brighter shade are from alum mordanted fabrics (002, 003) followed by chitosan mordanted fabrics (008, 009). The closely depth of shade between iron mordant and cinnamon mordant and between alum mordant and chitosan mordant possibly make biomordants replaceable with metallic salts mordants.

B. Color Fastness Properties of Dyed Silk

Table 7 shows the summary of fastness properties evaluated from the dyed silk fabrics. All dyed samples were average rated from 4 to 4/5 for the change in color while staining rated from 4/5 to 5. This rating is considered excellent where 5 is the best. The result for fastness properties of all dyed fabrics to perspiration also gave good rating of 4/5 for change in color, and the rating for staining ranged from 4 to 4/5. The same goes to fastness properties result for rubbing/crocking which was from 4 to 5 for dry rub and 4 to 4/5 for wet rub. Conversely, the fastness properties to light for all dyed samples were poor with rating of 3–4. Another study needed to improve the problem of low light fastness of natural dyes dyed on natural fabric.

Table IV- Numbering Codes for Dyed Silks

Codes	Dyeing and Mordanting Method
Control	Control Sample (Dyed without mordant)
002	Pre-mordanted with Alum
003	Simultaneous mordanted with Alum
004	Pre-mordanted with Iron II sulphate
005	Simultaneous mordanted with Iron II sulphate
006	Pre-mordanted with Cinnamon
007	Simultaneous mordanted with Cinnamon
008	Pre-mordanted with Chitosan
009	Simultaneous mordanted with Chitosan

Table V- Color Measurement Values

Fabric Code	L*	a*	b*
Control	91.91	-0.63	4.09
002	86.16	-1.81	14.70
003	86.86	0.23	12.75
004	76.01	6.06	26.27
005	74.17	2.33	15.78
006	87.57	0.20	10.60
007	75.02	4.46	21.58
008	90.51	-0.82	7.99
009	84.94	0.01	14.61

Table VI- Swatches of dyed silk

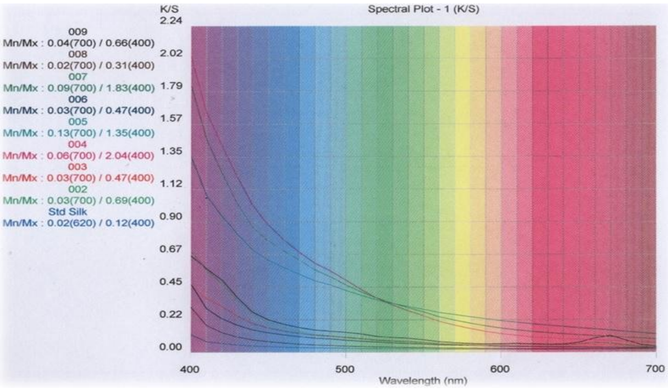

Fig. 1. K/S Values of dyed silks

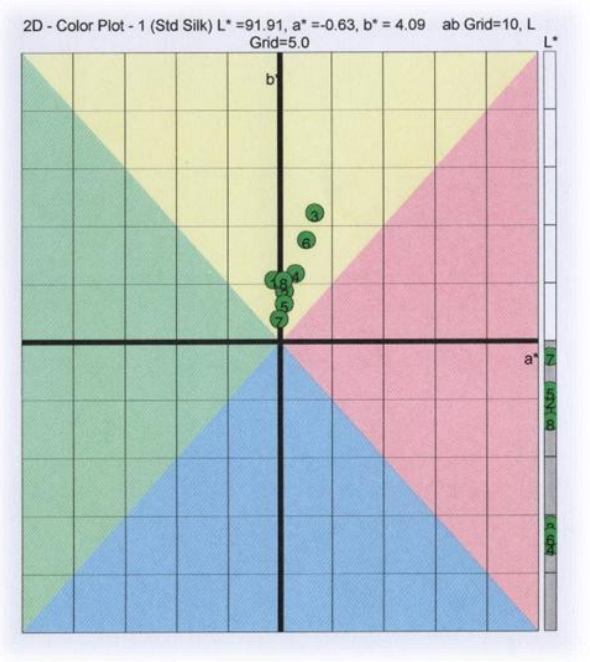

Fig. 2. 2D Plot of Dyed Silks

Table VII- Fastness Properties of Silk Fabric

Mordanting Method	Mordants	Washing			Perspiration			Rubbing/Crocking		Light
		Color	Staining		Color	Staining				
		Change	Silk	Cotton	Change	Silk	Cotton	Dry	Wet	
Pre-Mordanting	No mordant	4/5	5	5	4/5	4	4	4/5	4	3
	Alum	4/5	5	5	4/5	4/5	4/5	4/5	4	3
	Iron	4/5	4/5	4/5	4	4/5	4/5	4	4	4
	Chitosan	4/5	5	5	4/5	4	5	5	4/5	5
	Cinnamon	4/5	4/5	4/5	4	4/5	4/5	4	4	4
Simultaneous Mordanting	No mordant	4/5	5	5	4/5	4	4/5	4/5	4/5	3
	Alum	4/5	5	5	4/5	4/5	4/5	4/5	4/5	4
	Iron	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	3
	Chitosan	4/5	5	5	4/5	4/5	4/5	4/5	4/5	4
	Cinnamon	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	3

IV. CONCLUSION

In conclusion, *Sargassum sp.* seaweed can be utilized as a natural dye source that produces unique and interesting shades on textiles. The shades vary depending on the mordant used which gave acceptable fastness properties. The approach of using biomordant during dyeing process have assisted to improve dyeing sustainability. Cinnamon acts as a good biomordant which imitate iron mordant that gave closely similar shade and fastness properties comparable to iron mordant. Similar to biomordant of chitosan which might imitate alum mordant in its shade and fastness properties.

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