Dyeing of Silk Fabric with a Natural Dye Extracted from the Leaves of *Mimusops Elengi* Linn

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Abstract

A natural dye was extracted from the leaves of mimusops elengi Linn which was applied to the silk fabric in order to examine its suitability as a dye. Leaves were used as the dye source since they are readily available and reproducible than other parts of the plant without making any damage of the plant. The dye staff in leaves of mimusops elengi is easily extractable by aqueous extraction process and the amount of crude dye achieved was exceptionally good. Dyeing of silk fabric was performed by the dye extract solely as well as mixing with mordant by using pre, simultaneous and post mordanting techniques. The color strength and fastness properties of dyed silk fabric were evaluatedby standard methods. A variety of shades and excellent color fastness of dyed silk fabric were observed for this natural dye with different mordant which could be an attractive environmentally friendly cheap alternative for silk dyeing.

Keywords: *Natural dye, Mimusops elengi leaves, mordant, silk, fastness properties*

Introduction

Natural dyes have emerged a great deal of interest in recent years due to increasing global awareness and health issue (Wang et al. 2018; Arora et al. 2017). Natural dyes are known a part of human life from ancient time as coloring materials for food, leather, natural fiber etc. (Khan et al. 2018; Hasan et al. 2015). But the discovery of synthetic dyes and their availability have forced to replace the natural dyes with synthetic dyes for textile and leather dyeing. Currently synthetic dyes are widely used and traded for textile industry. However, recent studies revealed that many synthetic dyes have adverse effect on human health and environment and it is better to avoid them (Mongkholrattanasit et al. 2011a; Bhuiyan et al. 2018). Some synthetic dyes are carcinogenic, toxic and allergic to human health specially aromatic azo-dyes which degrade to aromatic amines that highly carcinogenic. Synthetic dyes are also a big threat for the environment and harmful to aquatic lives if they discharge untreated into the water bodies. Natural dyes can easily reduce these threats because they are not toxic, carcinogenic and allergic. In addition, they are readily biodegradable, environmentally friendly and having potential medicinal properties (Reddy et al. 2012). Different parts of plants such as leaves, barks, flowers, roots, fruits are the main sources of natural dyes (Prabhu et al. 2012). A plenty of dye-bearing plants are available in Bangladesh which can be used for dyeing purpose.

Although the recent interest has been grown in the use of natural dyes on textiles but their uses are still very limited. It is reported that some natural dyes extracted from leaves not only bear colors with excellent fastness properties but also provide antibacterial, UV

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protection and antioxidant properties (Mongkholrattanasit et al. 2011b). The production of textile items having antibacterial activity is of great interest in recent years. Both cellulose and protein based natural fibers are known to be vulnerable to some bacterial attack. Since these natural fibers provide all the basic requirements for bacterial growth therefore apparel made from these fibers may cause health problems of users alongside with making the fabrics malodorous (Ali et al. 2015).

Mimusops elengi is an evergreen important medicinal plant of Sapotaceae family, locally known as "Bakul" which is widely distributed all over the country. Traditionally it is used as an Ayurvedic medicine. Different research groups reported that most of the parts of *M. elengi* have medicinal properties specially its leaves which possess antibacterial, antifungal and antioxidant and food preservation properties (Baliga et al. 2011; Anuradha et al. 2016). The aqueous and alcoholic extracts of *M. elengi* leaves are proven to be very effective on some bacterial strain (Sircar et al. 2016). A paper also describes the use of bark of *M. elengi* as a raw material for color and the dye on wool fabric that showed a good color strength and fastness properties (Bhuyan et al. 2004).

The purpose of this study is to examine the properties of a natural dye extracted from *M. elengi* leaves and seeing its suitability as a dye. In this study leaves have been used as the dye source since leaves are readily available and reproducible than other parts of the plant without making any damage of the plant. The dye extract successfully applied on silk fabrics in different conditions and found excellent results in terms of color yield and fastness properties.

Material and Methods

Materials

Laboratory grade copper sulphate ($CuSO_4.5H_2O$), potash alum ($KAl(SO_4)_2.12H_2O$) and stannous chloride ($SnCl_2.2H_2O$) were purchased from commercial suppliers and used as mordant without any further purification. A commercially available detergent was used for washing off the silk fabric after dyeing.

Extraction

The leaves of *M. elengi* were collected from Rajshahi, Bangladesh and washed thoroughly with water to remove dirt and impurities. They were then dried at room temperature for 15 days and crushed into powder form by using a grinding machine. The crude color components were extracted from this dry crushed leaves using aqueous extraction process. One hundred grams of the crushed leaves were soaked in boiling water (about 500 milliliter) for 15 minutes in a conical flask. The extract was collected by filtration through a piece of fabric and then fresh water was added with residue and boiled for 15 minutes. The same procedure was repeated another two times in order to collect the remaining extract. The filtrates were then combined and filtered again through a very fine meshed fabric to ensure clear dye solution. The crude solid dye was obtained by evaporation under reduced pressure. After drying, the dye mixture was powdered in a mortar. This powder was then ready for dyeing process. About twenty grams of crude solid dye obtained from one hundred grams dry leaves powder of *M. elengi* (yield 20 %).

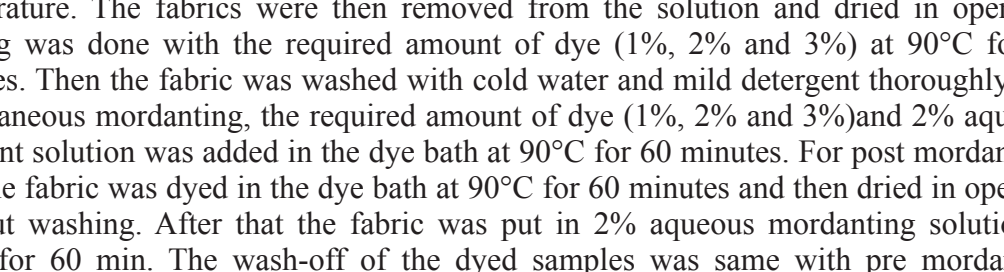

Figure 1 Leaves of *Mimusops elengi* Linn. and extracted crude dye

Dyeing and Mordanting

The dyeing of silk fabrics was carried out in exhaust dyeing method (Mathis) for 60 minutes at 90°C with different concentrations. Mordanting was achieved by using pre, simultaneous and post mordanting process. For pre-mordanting process the samples of silk fabrics were first immersed in 2% aqueous mordant solution for 10 hours at room temperature. The fabrics were then removed from the solution and dried in open air. Dyeing was done with the required amount of dye (1%, 2% and 3%) at 90°C for 60 minutes. Then the fabric was washed with cold water and mild detergent thoroughly. For simultaneous mordanting, the required amount of dye (1%, 2% and 3%)and 2% aqueous mordant solution was added in the dye bath at 90°C for 60 minutes. For post mordanting, first the fabric was dyed in the dye bath at 90°C for 60 minutes and then dried in open air without washing. After that the fabric was put in 2% aqueous mordanting solution at 90°C for 60 min. The wash-off of the dyed samples was same with pre mordanting process.

Characterization

Some important functional groups of the crude dye were determined by Fourier Transform Infrared (FTIR) spectrophotometer. The relative color strength of dyed fabrics was measured on spectrophotometer (Data color 650) by the light reflectance technique using the Kubelka-Munk equation, $K/S = (1-R)^2/2R$, Where R is the reflectance of dyed fabric, K is the absorption coefficient, S is the scattering coefficient. All the samples were measured by a maximum absorption wavelength (λ_{max}) value at 410 nm.

The color fastness to washing was conducted according to ISO 105-2CS: 2002 by wash fastness tester (Gyrowash, James H. Heal & Co. Ltd. UK). The change and staining of color due to washing was assessed by comparing the untreated fabric with the treated fabric samples with respect to the rating of color change and color staining grey scales. The color fastness to rubbing was determined according to ISO 105x2L:2002 by rubbing fastness tester (SDL International, UK). The color staining to rubbing on bleached cotton fabric was assessed by both dry and wet condition.

Result and Discussion

The crude natural dye was extracted from *M. elengi* leaves using water extraction process. The extracted liquid was dried under reduced pressure. A good amount of crude dye in

powder form was observed from this process. It can be very simple and the raw material of the dye is available from our surroundings at cheaper cost. The extracted dye was applied on silk fabric. The degummed and bleached silk fabrics were used for dyeing with varying concentration of dye extract. In case of mordant the dyeing process was performed by using pre, simultaneous and post mordanting methods. In all cases different color strength and color fastness were achieved that shown in the Table 1, 2 & 3.

Table 1: Color strength and obtaining color of silk fabric using 2% mordants with varying concentration of *M. elengi* leaf extract dye

Mordant	Mordanting technique	Dye (%)	K/S Value	Color obtained
Nil	Nil	1	2.470	
		2	3.196	
		3	3.216	
$CuSO_4.5H_2O$	Pre	1	5.001	
		2	6.270	
		3	7.174	
	Simultaneous	1	4.240	
		2	5.029	
		3	5.160	
	Post	1	6.376	
		2	9.775	
		3	10.730	
$SnCl_2.2H_2O$	Pre	3	6.968	
	Simultaneous	3	1.896	
	Post	3	3.446	
$KAl(SO_4)_2.12H_2O$	Pre	3	5.980	
	Simultaneous	3	3.735	
	Post	3	4.316	

The color strength of *M. elengi* dyed silk fabrics was investigated by K/S value which depend on the concentration of dye components on the fabric by using light reflectance and absorbance technique. The color of the un-mordant dyed silk is light reddish brown. However, the shades and color strength depend on different mordant and the concentration of the dye extract. At increased dye concentration the fabrics resulted in deeper shade. The experimental results shown in Table 1 are clearly indicating that the color strength increases with the increased percentage of dye concentration. This trend

was observed clearly up to 3% dye. Above the 3%, the increase of color strength was insignificant.

Without using any mordant the color strength of silk fabric was found to be 3.216 (3% dye concentration). However, in the mordanting method with same dye concentrationthe fabrics showed higher color strength. Three different mordanting techniques were used for copper sulphate, stannous chloride and potassium aluminium sulphate as mordants. It was observed that at the same dye concentration (3%) with different mordant the relative color strength values are higher for dyed sample with copper sulphate. Post mordanting method with copper sulphate significantly increased the color strength value (K/S = 10.73) for silk fabric. Most of the mordanting methods have shown better performance to improve the color yield except stannous chloride in simultaneous mordanting which showed lower color strength value (K/S = 1.896) in comparison with unmordanted dyed samples.

The presence of tannins and flavonoids has been confirmed earlier in *M. elengi* leaf extract along with other organic compounds (Anuradha et al. 2016; Sirkar et al. 2016). Both are phenolic compounds and well documented for coloring the fabrics (Mongkholrattanasit et al. 2011b). The hydroxy, alkoxy or carbonyl groups in the dye molecules can act as ligands and are capable of forming complex with metal ions. The functional groups of silk also have the ability to form coordination bonds with metal ions. For this reason, metallic salts increase the color strength values and enhance the dye uptake capacity. In addition, mordanting techniques have a great influence on the interaction of metal ion with fiber and dye molecules and ultimately on the dye uptake capacity. The sequence of color strength of dyed silk fabric for pre mordanting technique is copper sulphate > stannous chloride > potash alum > no mordant. In case of simultaneous mordanting technique, the sequence is copper sulphate > potash alum > no mordant > stannous chloride and for post mordanting technique, it is copper sulphate > potash alum > stannous chloride > no mordant. Copper sulphate showed better results for color strength than other salts. Similarly, pre and postmordanting techniques gave better color strength than simultaneous. The variation of color was obtained from pale to dark brown with little reddish or yellowish.

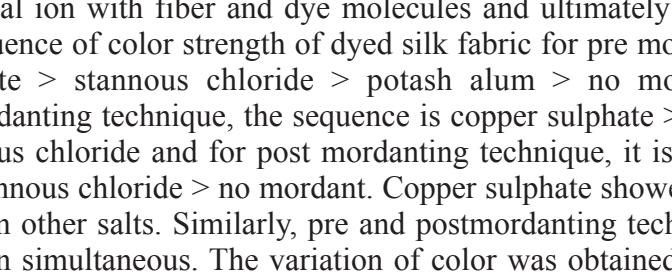

Figure 2: FTIR spectra of the crude dye

The structural characteristics of the dye were studied by FTIR spectra (Fig. 2). The bands were observed at 3298 cm^{-1}, 2900 cm^{-1}, 1774 cm^{-1}, 1608 cm^{-1}, 1512 cm^{-1}, 1417 cm^{-1}, 1325 cm^{-1}, 1277 cm^{-1}, 1131 cm^{-1}, 1045 cm^{-1}, 1012 cm^{-1} and 889 cm^{-1}. The spectrum of

the dye indicates the presence of O-H at around 3300 cm^{-1} and C-H stretching range of 2900 cm^{-1}. The benzene ring vibration is observed at 1512 cm^{-1} and 1608 cm^{-1} and the band 1045 cm^{-1} is responsible for C-O-C stretching. The characteristic peaks are very similar with previously reported tannin based compounds (Mahmoud et al. 2015; Lisperguer et al. 2016; Arasaretnam et al. 2017; Luzardo et al. 2015).

Table 2: Color fastness to washing of silk fabric

Mordants	Mordanting technique	Dye (%)	Color change	Color staining to					
				Acetate	Cotton	Nylon	Polyster	Acrylic	Wool
Nil	Nil	1	5	4-5	5	4-5	5	5	5
		2	4-5	4	5	4	5	5	5
		3	5	4-5	5	4-5	5	5	5
$CuSO_4$ $.5H_2O$	Pre	1	4-5	4	5	4	5	5	5
		2	4-5	4-5	5	4-5	5	5	5
		3	5	4	5	4	5	5	5
	Simultaneous	1	4	3-4	5	3-4	5	5	5
		2	4-5	5	5	4-5	5	5	5
		3	4-5	4-5	5	4-5	5	5	5
	Post	1	4-5	4-5	5	4-5	5	5	5
		2	3-4	4-5	5	4-5	5	5	5
		3	4	5	5	4-5	5	5	5
$SnCl_2$ $.2H_2O$	Pre	3	4-5	4	5	3-4	5	5	5
	Simultaneous	3	4	5	5	5	5	5	5
	Post	3	5	5	5	5	5	5	5
$KAl(SO_4)_2$ $.12H_2O$	Pre	3	5	5	5	4-5	5	5	5
	Simultaneous	3	3-4	4-5	5	4-5	5	5	5
	Post	3	4-5	4-5	5	4-5	5	5	5

Table 3: Color fastness to rubbing of silk fabric

Mordant	Mordanting technique	Dye (%)	Dry rubbing	Wet rubbing
Nil	Nil	1	5	5
		2	5	5
		3	5	5
$CuSO_4.5H_2O$	Pre	3	5	5
	Simultaneous	3	5	4-5
	Post	3	5	4-5
$SnCl_2.2H_2O$	Pre	3	5	5
	Simultaneous	3	5	5
	Post	3	5	5
$KAl(SO_4)_2.12H_2O$	Pre	3	5	5
	Simultaneous	3	5	5
	Post	3	5	5

The fastness properties of dyed samples were measured according to ISO standard method. During washing fastness test, SDC DW multifiber fabric was attached to the dyed sample to measure the staining. The change in color was measured with grey scale by comparing the undyed sample. The wash fastness rating of silk fabrics dyed with or without mordants at different concentrations is presented in Table. 2. The most of the results showed that the color fastness ratings of the fabrics to washing are very good to excellent (4-5 to 5). Only in few cases it was observed fair to good level (3-4 to 4). With mordanting techniques, in some cases fastness properties to washing were excellent (5). These excellent results achieved due to the metal-ligand complex formation and insolubilizing the dye in water solution (Mongkholrattanasit et al. 2015; Gias Uddin 2015). On the other hand, color staining ratings to washing were found to be good to excellent (4 to 5) in most of the cases. The slight staining was observed only for the adjacent acetate and nylon fibers in the multifiber fabric. No significant change in color was observed for both dry and wet rubbing fastness values of the dyed fabric. A very impressive rubbing fastness values were observed for the silk fabric dyed with *M. elengi* leaf extract.

Conclusion

The present research explored the application of natural dye on silk fabric extracted from the leaves of *M. elengi*. The dyed fabrics have been characterized by standard methods. The dye extract itself, without any mordant, gave a very excellent result in terms of color strength and shade. The fastness properties of the sample were very good to excellent. The use of mordants further improved the color strength and showed a variety of shades. The results indicated that the aqueous *M. elengi* leaf extract dye has a very good potential for dyeing the silk fabrics.

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