International Journal of Chemical, Materials and Biomolecular Sciences
ISSN: 2415-6620
Vol:8, No:5, 2014

Textile Dyeing with Natural Dye from Sappan Tree (*Caesalpinia sappan* Linn.) Extract

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***Abstract*—**Natural dye extracted from *Caesalpinia sappan* Linn. was applied to a cotton fabric and silk yarn by dyeing process. The dyestuff component of *Caesalpinia sappan* Linn. was extracted using water and ethanol. Analytical studies such as UV–VIS spectrophotometry and gravimetric analysis were performed on the extracts. Brazilein, the major dyestuff component of *Caesalpinia sappan* Linn. was confirmed in both aqueous and ethanolic extracts by UV–VIS spectrum. The color of each dyed material was investigated in terms of the CIELAB (L*, a* and b*) and K/S values. Cotton fabric dyed without mordant had a shade of reddish-brown, while those post-mordanted with aluminum potassium sulfate, ferrous sulfate and copper sulfate produced a variety of wine red to dark purple color shades. Cotton fabric and silk yarn dyeing was studied using aluminum potassium sulfate as a mordant. The observed color strength was enhanced with increase in mordant concentration.

***Keywords*—**Natural dyes, Plant materials, Dyeing, Mordant.

I. Introduction

THERE is a growing demand for eco-friendly and non-toxic colorants [1]. Since natural dyes are biodegradable and less toxic and allergenic than synthetic dyes [2], dyes derived from natural sources are regaining popularity for applications not only in coloration of textiles [3] , [4] but also as food ingredients [5] and cosmetics [6].

Thailand has an abundance of dye yielding plant species in different parts of the country. Traditionally, the rural folks of different region dyed their materials from leaves, roots and bark of the plants, mostly by boiling to get the desired color.

Caesalpinia sappan L. (*Leguminosae*) is widely distributed in Thailand, Indonesia, Vietnam, Burma, India, and south and southwest China [7]. Its heartwood has long been used as a Traditional Chinese Medicine for the treatment of infectious diseases such as an anti-inflammatory [8], antioxidant [9] and for antimicrobial activities [10].

It is reported that sappan tree barks and heartwood are rich in tannin and can produced for use in historical paintings and textiles [11]. The major coloring component of sappan tree is brazilin but brazilein, which is the oxidized product of brazilin, is also present (Fig. 1) [12]. In this study, the dyeing properties of cotton fabric and silk yarn using an aqueous extract of sappan tree heartwood as a natural dye has been investigated. Different factors affecting dyeing ability were examined.

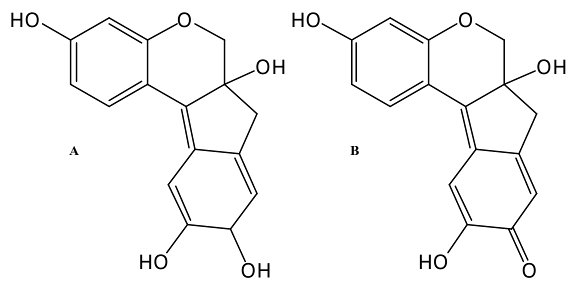

Fig. 1 The structure (A) brazilin and (B) brazilein

II. Experiment

A. Materials

Caesalpinia sappan Linn. was obtained in the form of dried heartwood slices from a local market in Bangkok, Thailand. De-gummy mulberry silk used for dyeing was supplied from Sisaket province, Thailand and used without any treatment. The cotton fabric, purchased locally, was soaked in a detergent solution containing 2.5g/L at 80–100°C for an hour to remove starch and other stiffening agents. The material to liquor (M:L) ratio was maintained at 1:80, after which the fabrics were washed with distilled water.

Fig. 2 Dried heartwood slices of *Caesalpinia sappan* Linn.

B. Chemicals

Mordants such as copper sulphate ($CuSO_4{\cdot}5H_2O$) ferrous sulphate ($FeSO_4{\cdot}7H_2O$) and alum [$Al(NH_4)(SO_4)_2{\cdot}12H_2O$] were procured Sigma-Aldrich Chemical Co. Distilled water was used in extraction, preparation of all chemical solutions, and dyeing processes.

C. Extraction of Colorants

The extraction of colorants from *Caesalpinia sappan* Linn.

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using water and ethanol were compared. The collected heartwood of *Caesalpinia sappan* Linn. was dried and later crushed to small pieces before being used for dye extraction. The dye extraction was performed by mixing the plant material and distilled water (or ethanol) in the weight ratio of 1:3 and boiling for 1h. The beaker was covered using aluminum foil to prevent loss of solvent by evaporation. The resulting solution was filtered to remove the residue. The extract samples were analyzed using a UV-visible spectrophotometer

D.Gravimetric Analysis

At the end of the extraction process, the samples taken from both ultrasound and control extracts were filtered and taken in clean, dried and weighed glass vessels. The extracts were dried in a hot-air oven until all the water evaporated and only the extract was left. The vessels were then cooled in a desiccators and the constant weight of the colorant extract obtained per gram of the plant material used were calculated. The yield was calculated using the following equation [13]:

$$\%\, Yield\ of\ natural\ colorant = \frac{natural\ dye\ extract\ obtained\ (g)}{\%\ amount\ of\ plant\ material\ used\ (g)}$$

E. Dyeing of Cotton Fabrics

After extraction of colorants, the cotton fabrics and silk yarn were dyed immediately in an aqueous extract at a liquor ratio of 30:1 for 1 hour without further heating.

F. Method of Mordanting

The post-mordanting method involved using 1% solution of copper sulphate ($CuSO_4{\cdot}5H_2O$), 1% solution of ferrous sulphate ($FeSO_4{\cdot}7H_2O$), 1% and 5% solutions of alum [$Al(NH_4)(SO_4)_2{\cdot}12H_2O$] . Mordanting was carried out for 30 min at room temperature. After mordanting, fabrics were wrung thoroughly and dyed again for 30 min then washed and dried.

III. Analytical Methods

A. UV-Visible Spectrophotometric Analysis

0.7 % yield of colorant was obtained from this extraction method. The extract samples were analyzed using a UV-visible spectrophotometer JASCO. The UV-VIS spectrums of the extract samples were obtained in the region of 200-700 nm. The UV–visible spectrum of *Caesalpinia sappan* Linn. extract is shown in Fig. 2. Strong absorption at 445nm, a typical wavelength for brazilein identification has been obtained. To observe brazilin and brazilein, UV detection at 254 and 280nm, as usually chosen for polyphenols detection, can be applied besides visible wavelength detection [14]. In Fig. 2, two peaks at 254 and 280nm were obtained in both aqueous and ehtanolic extract.

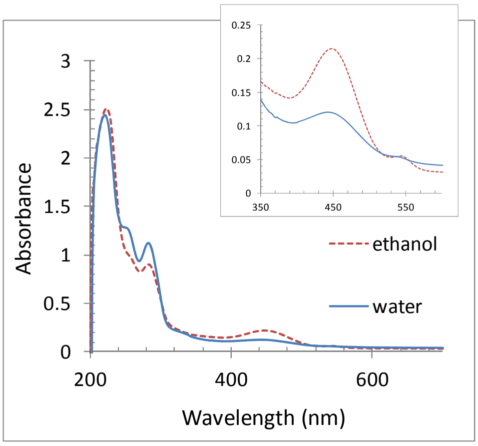


Fig. 3 UV–VIS spectrum for *Caesalpinia sappan* Linn. dye aqueous and ethanolic extract.

B. Color Strength and Color Depth Measurements

The color strength and color depth of dyed samples were determined by light reflectance technique using a Hunter Lab spectrophotometer Color Quest XE. The color strength (K/S) value of samples was evaluated using the "Kubelka-Munk equation" [15]:

$$K/S = (1 - R)^2/2R$$

where R is the reflectance of the dyed sample; K is the absorption coefficient and S is the scattering coefficient.

The color of the dyed samples are given in CIELab coordinates (L*, a*, b*): L* corresponding to the brightness (100 = white, 0 = black), a* to the red – green coordinate (+ve = red, -ve = green) and b* to the yellow – blue coordinate (+ve = yellow, -ve = green)

TABLE I
Color Values of Dyed Cotton by Post-Mordanting

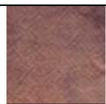
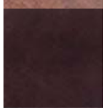
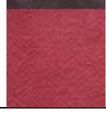
Mordants	Color co-ordinates L*	a*	b*	Color obtained	
Without mordant	53.92	14.63	8.59		reddish brown
1% $CuSO_4$	31.40	12.43	-3.47		dark purple
1% $FeSO_4$	34.88	8.53	-3.86		dark lavender
1% $AlK(SO_4)_2$	42.00	32.62	8.44		wine red

TABLE II
COLOR VALUES OF DYED COTTON AND SILK BY POST-MORDANTING USING 1% AND 5% ALUM SOLUTIONS

Textile	Mordants	*K/S**	Color co-ordinates		
			L*	a*	b*
Cotton	1% $AlK(SO_4)_2$	4.71	47.62	27.16	13.58
	5% $AlK(SO_4)_2$	5.23	47.54	29.07	10.92
Silk	1% $AlK(SO_4)_2$	-	36.34	37.65	17.50
	5% $AlK(SO_4)_2$	-	28.57	34.04	16.20

* K/S value was measured only for cotton fabrics.

Fig. 4 Dyed silk yarn with *Caesalpinia sappan* Linn. and pre-mordanted using (a) 1% and (b) 5% alum solution. The darker shade was obtained from higher mordant concentration.

Mordanting generally improved dye performance and help the dyer to achieve a broad spectrum of colors on a wide range of natural as well as synthetic fibers with expanded shade ranges and better fastness properties [16], [17]. Copper sulfate and ferrous sulfate mordants are well known for their ability to form coordination complexes and to chelate with the dye and interact with the fibers. In this study, cotton fabric dyed without mordant had as hade of reddish-brown, while those post-mordanted with aluminum potassium sulfate, ferrous sulfate and copper sulfate produced a variety of wine red to dark purple color shades and color strength was enhanced with increase in mordant concentration (Table II and Fig. 3.).

When compared with cotton, silk yarn can be more successfully dyed with *Caesalpinia sappan* Linn. When dyed on silk, the darker and duller color shade was obtained. This may due to phenolic compounds in dyestaff component that can form hydrogen bonds with the carboxyl group of protein fibers. Furthermore, the anionic charge on the phenolic groups forms an ionic bond with cationics (amino groups) on the protein substrate [18]

ACKNOWLEDGEMENTS

The author expresses sincere thanks to the Faculty of Science and Technology, Suan Sunandha Rajabhat University for necessary facilities to carry out this work. This research was supported by grant Suan Sunandha Rajabhat University.

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