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Dyeing of Silk Fabric with Eco-Friendly Natural Dye obtained from Flower of *Thespesia populnea* using Single mordants

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Abstract : Bleached silkfabric was dyed with natural dye obtained from the flower of *Thespesia populnea.* The colour fastness properties and colour strength of dyed silk fabric was determined andcompared. From the comparative study of fastness properties and colour strength of the dyed silk samples,*Thespesia populnea*in simultaneous mordanting method with 3% mordant combination gives better results.
Keywords : *Thespesia populnea*, Colour strength, Fastness, Mordant, Natural dye, silk.

Introduction

Now-a-days, in the textile industry, the interest in the use of natural dyes has been growing rapidly due to the result of stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes[1]. Environmental pollution is due to the discharge of dyeing industry effluents is the matter of major concern now-a-days. For many years, people were using all dyes were natural substances, derived mainly from plants and animals. The natural dyes present in plants and animals are pigmentary molecules[2] which impart colour to the materials. With the world becoming more conscious towards ecology and environment, there is greater need today to revive the tradition of natural dye and dyeing techniques as an alternative of hazardous synthetic dyes is an extremely crude.

The common drawbacks of natural dyes are their non-reproducible and non-uniform shades, poor to moderate colour fastness and lack of scientific information on the chemistry of dyeing and standardised dyeing methods[3,4]. Many reports are available on application of natural dyes on silk[5,6] and cotton[7,8].The present investigation deals with the extraction of natural dye from the stem extract of the flower of *Thespesia populnea*.

The aim of present work has been carried out to prepare eco-friendly natural dyes from the flower of *Thespesia populnea* and apply them on silk fabric. In the present work an attempt has been made to study the effect of mordanting and dyeing properties[9] of silk fabric such as, washing, rubbing, light fastness and perspiration[10] and also to visualize the effect of myrobolan and metallic mordants have been undertaken.

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Materials and Methods

Materials

The present investigation deals with the extraction of natural dye from the flower of *Thespesia populnea*. Milo (*Thespesia populnea*) is one of the most important trees to Pacific Island peoples. The rich, dark wood is carved into beautiful bowls, tools, small canoes, and figures. Ropes are twisted from the bark. The trees provide protection against wind, salt spray, and the hot sun. The seeds, leaves, and bark provide medicine and food. In ancient times the trees were planted around temple sites. Today the tree is rarer than in the past because of overharvesting in some areas and increased urbanization in others. The tree is easy to grow and should be considered for reforestation and urban forestry projects in the Pacific where suitable sites are available.

Fig: Newly opened flower

Milo is a small evergreen tree averaging 6–10 m (20–33 ft) in height, with a short, often crooked stem and a broad, densecrown. It has glossy green, heart-shaped leaves and yellow hibiscus-type flowers. The tree grows well along warm coastal areas from the east coast of Africa and South and Southeast Asia to Melanesia, Micronesia, and Polynesia. Itis currently naturalized in tropical climates throughout the aworld from the Caribbean to Africa.

Fig: Leaves and older flower

The tree is valuable as a coastal windbreak because it ishighly resistant to wind and salt spray and grows well insandy, saline soils. It propagates easily and grows rapidly.It naturalizes easily and has become a

weed in some areas, so it should not be planted in areas where it is not already present. The tree grows best under full sunlight and tolerates drought conditions. The heartwood is resistant to dry wood termites. Milo has many uses including coastal protection, animal fodder, windbreaks, and living fences. The most common use in the Pacific today is probably asan ornamental tree, despite its valuable timber.

The silk fabric obtained from Gandhigram Rural University, Dindugal**,** was used for the study. Analytical reagents (AR) grade ferrous sulphate, aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, commercial grade acetic acid, common salt, sodium carbonate were used. A natural mordant myrobolan (*Terminalia chebula*) powder[11] was used for the study. The ethanol extract of the flower of *Thespesia populnea* was used to get brown colour component for dyeing of fabrics. Depending upon the mordant used, the colour obtained on textiles from the flower of *Thespesia populnea* extract may give different shades.

The myrobolan (harda) powder was soaked in water(1:10 volume) for overnight (12h) at room temperature to obtain the swelled myrobolan gel. It was then mixed with a known volume of water andheated at 800^0C for 30 min. The resulting solution is cooled and filtered. The filtrate was used as final mordant solution for mordanting.

Methods

Extraction of colour component

For optimizing[12] the extraction method the ethanol extraction of dye liquor was carried out undervarying conditions, such as time of extraction, temperature of extraction bath and material-to-liquorratio. In each case, the optical density or absorbance value at a particular maximum absorbance wavelength (λ420nm) for the ethanol extract of plant parts were estimated by using Hitachi-U-2000 UVVIS absorbance spectrometer.

Dyeing of silk fabrics with the extract of flower of *Thespesia populnea*

The wetted out silk samples were entered into dye baths containing required amount of dye extract and water. After 10 minutes, required amount of sodium carbonate and sodium chloride were added. The dyeing was carried out for one hour at 60°C. The dyed samples were dried in air without washing to make them ready for pre, simultaneous and postmordanting using myrobolan and metallic salts.

Pre-Mordanting of silk fabrics withmyrobolan and metallic salts

Bleached silk fabrics with or without pre-mordanting were further mordanted prior to dyeing using 1-3% ofany one of the chemical mordants, such as aluminiumsulphate, nickel sulphate, potassium dichromate, stannous chloride, copper sulphate and themyrobolan, at 60°C for 30 min with material-to-liquorratio of 1:20. The samples treated with metalsalts were dyed with the dye extract.

Simultaneous -Mordanting of silk fabrics with myrobolan and metallic salts.

The silk sample fabrics were treated with both dye extract and metal salts simultaneously, using 1-3%of any one of the chemical mordants, such as aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, copper sulphate and the myrobolan, at 60°C for 30 min with material-to-liquorratio of 1:20.

Post-Mordanting of silk fabrics withmyrobolan and metallic salts.

The silk sample fabrics were dyed with dye extract. The wetted out silk samples were entered into different dye baths containing required amount of dye extract and water. After 10 minutes required amount of sodium sulphate was added. After 20 minutes required amount of sodium chloride was added. The dyeing was carried out for one hour at 50°C. The dyed samples were taken out, squeezed and used for treatment with metal salts process without washing. The dyed cotton samples were treated with differentmetal salts using 1-3% of any one of the chemical mordants, such as aluminium sulphate, nickelsulphate, potassium dichromate, stannous chloride,copper sulphate and the myrobolan, at 60°C for 30min with material-to-liquor ratio of 1:20.

In all the above three methods, after the dyeing is over, the dyed samples were repeatedly washed with water and then dried in air. Finally, the dyed samples were subjected to soaping with 2gpl soap solution at50°C for 10 min, followed by repeated water wash and drying under sun.

Determination of surface colourstrength[13] (K/S value)

The K/S value of the undyed and dyed silkf abric was determined by measuring surface reflectance of the samples using a computer-aided Macbeth 2020 plus reflectance spectrophotometer, using the following Kubelka Munk equation with thehelp of relevant software:

$$K/S = \frac{(1- R_{\lambda max})^2}{2R_{\lambda max}} = \alpha C_d$$

where K is the coefficient of absorption; S the coefficient of scattering; Cd, the concentration of the due and Rλ max the surface reflectance value of the sample at a particular wavelength, where maximum absorption occurs for a particular dye/colour component.

Evaluation of Colour Fastness[14,15]

Colour fastness to washing of the dyed fabric samples was determined as per IS: 764 – 1984method using a Sasmira launder-O-meter followingIs-3 wash fastness method. The wash fastness rating was assessed using grey scale as per ISO-05-A02(loss of shade depth) and ISO-105-AO3 (extent of staining) and the same was cross-checked by measuring the loss of depth of colour and staining using Macbeth 2020 plus computer-aided colour measurement system attached with relevant software.

Colour fastness to rubbing[16] (dry and wet) was assessed as per IS: 766-1984 method using a manually operated crock meter and grey scale as per ISO-105-AO3 (extent of staining). Colour fastness14 to exposure to light was determined as per IS: 2454-1984 method. The sample was exposed to UV light in a Shirley MBTF Microsal fade-O-meter (having 500 watt Philips mercury bulb tungsten filament lamp simulating day light) along with the eight blue wool standards (BS 1006: BOI:1978). The fading of each sample was observed against the fading of blue wool standards (1-8).Colour fastness to perspiration assessed according to IS 971-1983 composite specimen was prepared by placing the test specimen between two adjacent pieces of fabrics of cotton and stitched all among four sides. The sample was soaked in the test solution (acidic /alkaline) separately with MLR 1:50 for 30 minutes at room temperature. The sample was then placed between two glass plates of perspirometer under load of 4.5kgs (10 lbs). The apparatus was kept in the oven for four hours at 37±2˚C. At the end of this period the specimen was removed and dried in air at a temperature not exceeding 60˚C. The test samples were graded for change in colour and staining using grey scales.

Results and Discussion

The colour strength values of silk fabrics dyed with stem of the flower of *Thespesia populnea* obtained in this study by using single mordanting method are presented and compared inTables 1, 2 and 3.

From the results, it was observed that in all the three dyeing methods, simultaneous method gave excellent results.

In all the three methods of dyeing, the mordants ferrous sulphate and aluminium suphate show excellent results. For dyeing of silk,1%, 2% and 3% mordant concentrations were used for the present study. Among these three concentrations 3% mordant concentration gave better results.

Table 1: Surface colour strength of flower of *Thespesia populnea* dyed silk fabric after pre, simultaneous and post mordanting methods by using 1% mordant concentration (K/S value without mordant : silk- 1.32)

Mordant concentration:1%	K/S(λ=420 nm)		
	Pre mordanting	Simultaneous mordanting	Post mordanting
Nickel sulphate	1.28	2.28	2.01
Aluminium sulphate	1.60	2.45	2.36
Potassium dichromate	1.16	1.17	1.12
Ferrous sulphate	1.68	2.57	2.66
Stannous chloride	1.51	2.51	2.31
Myrobolan	0.94	1.15	1.22

Table 2: Surface colour strength of flower of Thespesia populnea dye dsilk fabric after pre, simultaneous and post mordanting methods by using 2% mordant concentration (K/S value without mordant : silk - 1.32)

Mordant concentration:1%	K/S(λ=420 nm)		
	Pre mordanting	Simultaneous mordanting	Post mordanting
Nickel sulphate	1.41	2.31	2.46
Aluminium sulphate	1.68	2.52	2.57
Potassium dichromate	1.18	1.23	1.38
Ferrous sulphate	1.77	2.61	2.72
Stannous chloride	1.61	2.54	2.57
Myrobolan	1.42	1.22	2.28

Table 3: Surface colour strength of flower of Thespesia populnea dyed silk fabric after pre, simultaneous and post mordanting methods by using 3% mordant concentration (K/S value without mordant : silk - 1.32)

Mordant concentration:1%	K/S(λ=420 nm)		
	Pre mordanting	Simultaneous mordanting	Post mordanting
Nickel sulphate	1.47	2.33	2.09
Aluminium sulphate	1.74	2.50	2.49
Potassium dichromate	1.21	1.22	1.31
Ferrous sulphate	1.81	2.61	2.53
Stannous chloride	1.61	2.53	2.41
Myrobolan	1.00	1.21	1.27

Table 4: Comparison of fastness properties of dyed silk using single mordants

Plant parts used for dyeing	Mordant used	Method	Properties						Reference
			WF	LF	RF		PF		
					Dry	Wet	Acidic	Alkaline	
Flower of *Thespesia populnea*	Ferrous sulphate (3%)	SM	4-5	4-5	5	5	4	4	Present Study
		PM	4	4	4-5	5	**4**	5	
	Aluminium sulphate (3%)	SM	4	4	4	5	4-5	4-5	
		PM	4	4	5	5	4	4	
Stem of *Achras sapota*	Ferrous sulphate (3%)	SM	5	4	5	5	5	5	Kumaresan et al (2016)
		PM	5	4	5	5	5	5	
	Aluminium sulphate	SM	4-5	4	5	5	5	5	

	(3%)	PM	5	4	5	5	4	4	
Flower of *Cordia sebestena*	Ferrous sulphate (3%)	SM	5	5	5	5	5	5	Kumaresan et al (2017)
		PM	5	5	5	5	5	5	
	Aluminium sulphate (3%)	SM	4	5	5	4	4	4	
		PM	5	4	5	5	5	5	
	Aluminium sulphate (12.5%)	SM	5	4	4-5	4	5	5	
Colquhounia coccinea	Ferrous sulphate (2.5%)	PM	4-5	4-5	5	5	5	5	Vankar et al (2010)
	Aluminium sulphate (12.5%)	PM	4	4	4	4	4	4	
Pongamia pinnato	Ferrous sulphate (2.5%)	SM	-	5	4-5	4-5	-	-	Kumar et al (2004)
	Aluminium sulphate (12.5%)	SM	-	5	4-5	4-5	-	-	
Neem tree bark	Aluminium sulphate (12.5%)	PM	3	2-3	4-5	4-5	-	-	Boonroeng et al (2009)

WF-Wash fastness LF-Light fastness PF-Perspiration fastness RF-Rub fastness CS-Colour strength PM-Pre mordanting SM-Simultaneous mordanting

Similar results were obtained in the previous study reported by Kumaresan et al (2012). The present studyshows excellent wash fastness (GS : 5) and light fastness (GS :5) when compared with Vankar et al (2010). A better lightfastness (GS :4-5) was reported in the present study in simultaneous mordanting method.

4. Conclusion

From the comparative study of fastness properties and colour strength of the dyed silk samples,flower of *Thespesia populnea.* in simultaneous mordanting method with 3% mordant combination gives better results.

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