

Original Research Article

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Eco-friendly Dyeing of Mulberry Silk Yarn with Bark of *Artocarpus lacucha*

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ABSTRACT

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The present work deals with the dyeing of mulberry silk yarns with *Artocarpus lacucha* to develop variety of harmonizing natural shades using copper sulphate, ferrous sulphate and stannous chloride as mordants. The dyes were extracted in acid, alkaline and neutral medium and dyeing was performed at 70°C. The extent of dyeing was studied on the basis of dye uptake as well as K/S values. Highest dye intensity was obtained when alkaline medium was used for dye extraction. The mordant enhances the fastness properties. The highest colour value of 3.0 was observed for the combination Monkey jack dye mordanted with Copper sulphate. Silk yarn dyed with bark of monkey jack along with different mordants produced shades of brown and beige colour.

Introduction

Dyeing is one of the most delightful textile arts which are an important branch in fashion design. Natural dye is a fascinating phenomenon that enticed researchers to their chemistry and production of fantastic novel hues to explore the point that no other dyes provide a better opportunity of teaching how to protect and respect the environment (Khadijah and Heba, 2013).

Each plant has specific dye components that determine its shade of color and fastness to light (Ellis, 2013).

For thousands of years human beings have used natural colours for a variety of purposes. With the advent of synthetic colorants, the use of natural colorants saw a drastic decline, the main reasons being that, with synthetic colorants, it was possible to obtain a wider range of colours and better colour fastness at a reasonable price.

However, in recent years there is an increasing demand for natural colorants because of the rising interest in preserving ecosystems, attributable to the fact that they are more quickly biodegraded than the synthetic dyes (Gowda *et al.*, 2014).

Materials and Methods

Selection of natural dye

Natural dyes make an important contribution to fabric decoration by producing various beautiful colours and colour harmonies obtained by a combination of varying dyeing methods.

Colouring matter was extracted from the root, stem, leaves, berries and flowers of the various types of plants.

The lakoocha (*A. lakoocha* Roxb) is also known as monkey jack or lakuchi in India, tampang and other similar native names in Malaya, as lokhat in Thailand. The tree is 20 to 30 ft (6-9 m) tall with deciduous, large, leathery leaves, downy on the underside. The fruits are nearly round or irregular, 2 to 5 in (5-12.5 cm) wide, velvety, dull-yellow tinged with pink, with sweet sour pulp which is occasionally eaten raw but mostly made into curries or chutney (Plate 1).

Chemicals

The chemicals used are sodium carbonate, hydrochloric acid, hydrogen peroxide, copper sulphate, stannous chloride and ferrous sulphate (All chemicals were procured from Fisher Scientific, India), sulphuric acid and ferric chloride hexahydrate LR were received from S. D. Fine Chemicals Limited (SDFCL) Mumbai, India. Zinc dust (A.W. 65.37) was received from RFCL Limited, Gujarat, India.

Mordants

Mordants used for the studies were copper sulphate, ferrous sulphate and stannous chloride. The amount of mordant to be used was calculated based on the weight of yarn expressed in terms of percentage as given in the following equation:

$$\text{Amount of mordant} = \frac{\text{Weight of the yarn in gram} \times \% \text{ of mordant}}{100}$$

Extraction of natural colorants

The monkey jack bark was cleaned and dried. The dried dye sources were powdered and 1.0 g was extracted separately in water, alkaline (1.0% of Na₂CO₃) acid (1.0% of HCl) medium. The extraction was performed at 100°C for 60 minutes, keeping 1:50 material to liquor ratio. The dye extracts were then subjected to colorimetric analysis and the optical density value was determined using an UV vis- Spectrophotometre.

Pre-treatment of the silk yarn

Degumming and bleaching methods were carried out for the study by using the procedure adopted by Gogoi (2004). Mulberry silk yarns were weighed accurately at M: L ratio of 1:50 and emerged into the water. Required quantity of washing soda (5 g/ litre) was added to water and started heating at 60⁰C temperature. After 30 minutes the yarns were taken out and washed properly in running water and then air dried. After degumming, the degummed yarns were bleached to improve the whiteness property of the yarn. Required quantity of water was taken and heated at temperature of 50⁰C. 1% hydrogen peroxide was added to the liquor. Sodium silicate was added to the bleaching bath as a stabilizing agent in the middle of the process. Yarns were taken out after 30 minutes and washed properly in the running water and air dried.

Determination of solid content of extracted dye solution

The solid content of the dye extracts was determined by taking a measured extracts in a pre-weighted Petri dish and the contents were

dried in an oven at 100±5°C till completely dried and the residue was obtained. The materials was kept in desiccators to cool down and then weighed. The solid content of the extracted dye solution was obtained as follows:

$$\% \text{ of solid content} = \frac{W_2 - W_1}{\text{Weight of the solution}} \times 100$$

Where,

W₁= weight of the Petri dish

W₂= weight of the Petri dish + solid

1% of solid dye content was obtained from 3 % of extracted dye solution.

Optimization of mordanting methods

In Pre-mordanting method, an aqueous solution was prepared by dissolving required amount of mordant in water. The yarns were boiled in 70°C in this solution for 30 minutes and then immersed in the prepared dye solution for dyeing. In simultaneous method, the mordants and dye were applied simultaneously in the same bath. The mulberry silk yarns were placed in the extracted dye bath and dyed for 15 minutes. After that, the required amount of mordants were added to the dye solution by lifting yarns and mixed properly.

The yarns were then dyed in the solution for 30 minutes. In post mordanting method, the samples were first dyed with dye solution and then mordanted. A mordanting bath was prepared as per recipe for mordanting. After dyeing, the samples were removed with the help of glass rod and then entered in the mordanting bath and heated to a temperature of 60-70°C for 30 minutes. Then the samples were allowed to cool, rinsed and dried in shade.

Colour measurement

Determination of colour co-ordinates

The CIE Lab colour co-ordinate value of all the dyed samples were recorded as average of five times readings in Brightness, Opacity and Colour Tester (Model no UEC-1080).

The colour parameters L*(depth of colour), a*(positive value redness and negative greenness) and b* (positive value yellowness and negative value blueness) were recorded.

The said values were ascertained for all mordants used in study. The lower value L* value indicated greater depth of colour and higher value of a* and b* indicated brightness of colour. The reflectance values were determined using following Kubelka -Munk equation:

$$\frac{K}{S} = \frac{(1 - R)^2}{2R}$$

Where K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance of the dyed fabric at the wavelength of maximum absorption.

Determination of fastness properties of dyed sample

The colour fastness property of yarn is depends on various factors like chemical structure of dye, yarn type, dyeing time, and temperature used on dyeing (Mahale *et al.*, 2003).

All the dyed samples of mulberry silk yarn were evaluated for colour fastness to washing, colour fastness to sunlight, colour fastness to crocking (dry and wet), colour fastness to pressing (dry and wet) and colour fastness to perspiration (acidic and alkaline) by using ASTM procedure.

Results and Discussion

Determination of wavelength for selected dye

It was observed that the absorbance pattern of monkey jack dye showed the highest absorbance of 460 nm (bluish-green colour) as optimum (Table 1). Gohl and Vilensky (1983) expressed that wavelength range for bluish-green was 460-500 nm.

Optimization of dye extraction medium

For optimization of dye extraction medium, dye of powders were extracted separately in plain water, in alkaline medium (1% of Na_2CO_3) and in acidic medium (1% of HCl) with material to liquor ratio 1:50, at 100°C for 60 minutes duration (Table 2). It was evident from the Table 2 that the optical density of extracted monkey jack dye solution in alkaline medium was found maximum (0.900) than in aqueous (0.607) and acidic (0.595). Phukan *et al.*, (2014) also found similar result on silk yarns dyed with *Morinda angustifolia*.

Optimization of alkali (Na_2CO_3) concentration for extraction of dye

The concentration of alkali (Na_2CO_3) for extraction of dye was optimized by extracting monkey jack in different concentration of alkali *viz.*, 0.1g to 1.0 grams with material to liquor ratio 1:50 for 60 minute duration at 100°C (Fig. 1). In monkey jack dye maximum optical density value (0.413) obtained at 0.7 per cent concentration of alkali. Hence, 0.7% concentration of alkali for dye was considered as optimized alkali concentration.

Optimization of dye extraction time

The extraction times for dyes were optimized based on highest optical density values. Dyes were extracted in M: L ratio 1:50 at 100°C for different time period *viz.*, 30, 45, 60, 75 and

90 minutes duration. The optical densities recorded for different time period presented in Table 3. Maximum optical density value (0.631) was obtained from 60 minutes and decreasing trend in optical density values obtained after 60 minutes. It may be due to the saturation of dye molecule. Similar result has been found by Pandey *et al.*, (2014) on silk dyeing with binary mixture of *Ficus religiosa* and *Moringa pterygosperma* leaves.

Optimization of dye material concentration

The concentrations of dyes were optimized by dyeing mulberry silk yarns in different concentration of dye *viz.* 1 to 10/100 gm of yarn. The absorption (%) of dye by the yarns were calculated based on optical densities of the dye liquor before and after dyeing were presented in Figure 2. It was clear from the Figure 2. That, in monkey jack dye maximum absorption (39.60%) was found at 9 per cent concentration of dye and minimum (21.89%) was observed in 1 per cent concentration of dye.

Optimization of dyeing time

For dyeing mulberry silk yarn with monkey jack dyes, the dyeing time was optimized based on (%) absorption of dye. For this purpose dyeing was carried out for different time period *viz.* 30, 45, 60, 75 and 90 minutes duration and optical densities of the dye liquor before and after dyeing was recorded. From the recorded optical density values, absorption (%) of the dye by the yarns were calculated and presented in Table 4. The per cent dye absorption increased with the increase in dyeing time and reached its maximum at 45 minute (36.09) and then decreased. It might be due to optimum saturation of dye molecule. Hence, the optimum dyeing time was selected as 45 min for dyeing silk yarn. Similar observations were made by Singh (2000) and Duarah (2007) in the case of Berberry and *Telanthera ficoidea* dye.

Table.1 Absorbance pattern of monkey jack

Sl. No	Wavelength	OD value
1.	400	1.144
2.	410	1.164
3.	420	1.211
4.	430	1.235
5.	440	1.314
6.	450	1.329
7.	460	1.336
8.	470	1.321
9.	480	1.305
10.	490	1.295

Table.2 Optimization of dye extraction medium

Sl. No.	Extraction medium	Time (min.)	Temperature (°C)	O.D value
1.	Aqueous	60	100	0.607
2.	Alkaline	60	100	0.900
3.	Acidic	60	100	0.595

Table.3 Optimization of dye extraction time

Sl. No.	Extraction time (min.)	Temperature (°C)	Dye absorption (%)
1.	30	100	0.566
2.	45	100	0.584
3.	60	100	0.631
4.	75	100	0.603
5.	90	100	0.579

Table.4 Optimization of dyeing time

Sl. No.	Dyeing time (min.)	Temperature (°C)	Dye absorption (%)
1.	30	70	33.65
2.	45	70	36.09
3.	60	70	34.47
4.	75	70	33.82
5.	90	70	30.19

Table.5 Optimization of mordant concentration

Name of the mordant	Mordant concentration (g/100gm of yarn)	Dye absorption (%)
Copper sulphate	1	45.43
	2	43.69
	3	40.78
	4	36.98
	5	34.43
Ferrous sulphate	1	30.87
	2	34.54
	3	35.98
	4	38.04
	5	36.98
Stannous chloride	1	47.21
	2	45.27
	3	40.47
	4	38.92
	5	36.43

Table.6 Optimization of mordanting time

Name of the mordant	Mordanting time (min.)	Dye absorption (%)
Copper sulphate	15	41.54
	30	42.11
	45	38.30
	60	34.18
	75	33.22
Ferrous sulphate	15	32.07
	30	37.84
	45	35.87
	60	34.21
	75	31.63
Stannous chloride	15	37.25
	30	40.81
	45	38.73
	60	37.52
	75	32.89

Table.7 Optimization of mordanting method

Name of the mordant	Mordant concentration (g/100gm of yarn)	Dye absorption (%)
Copper sulphate	1	45.43
	2	43.69
	3	40.78
	4	36.98
	5	34.43
Ferrous sulphate	1	30.87
	2	34.54
	3	35.98
	4	38.04
	5	36.98
Stannous chloride	1	47.21
	2	45.27
	3	40.47
	4	38.92
	5	36.43

Table.8 Computer colour matching data

Sl. No	Details	L*	a*	b*	C	H	K/S
1	Monkey jack dye	53.52	10.58	14.04	17.58	52.97	0.89
2	Monkey jack with Copper sulphate	52.80	8.32	12.22	14.78	55.71	3.00
3	Monkey jack with ferrous sulphate	50.70	5.98	10.58	12.15	60.49	1.47
4	Monkey jack with Stannous chloride	54.27	10.52	14.26	17.72	53.55	1.41

Table.9 Ratings for colourfastness properties of Monkey jack dyed samples

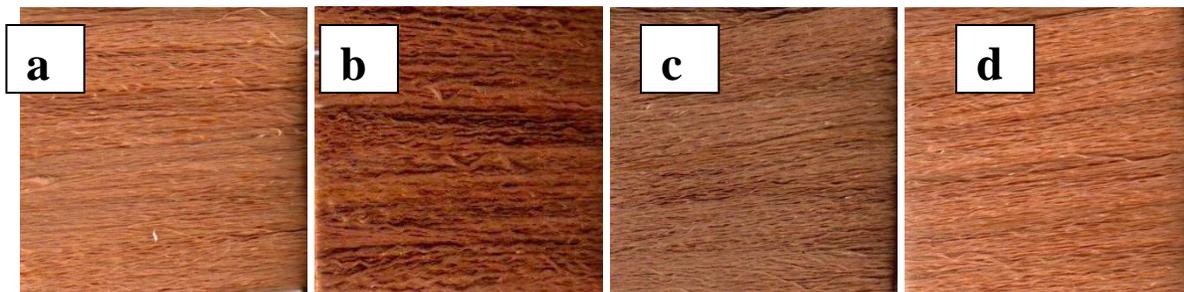
Sl. No.	Mordants used	Sunlight	Washing		Croaking				Perspiration				Pressing			
					Dry		Wet		Acidic		Alkaline		Dry		Wet	
			CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS
1.	Without mordant	3	5	5	5	5	5	5	4	5	5	4	4	4	5	4
2.	Copper sulphate	4	4	5	5	5	4	4	5	5	4	5	5	5	4	4
3.	Ferrous sulphate	3	4	5	5	5	4	4	4	5	4	4	5	4	5	5
4.	Stannous chloride	3	5	5	5	5	4	5	5	5	5	5	5	5	5	5

CC: Colour change, CS: Colour staining; CC Ratings: 1= very poor, 2=poor, 3=fair, 4=very fair, 5=good
 CS Ratings: 1=heavily stained, 2=considerably stained, 3= noticeable stained, 4=slightly stained, 5=negligible or no staining.

Plate.1 a) Dried Monkey jack bark & b) Monkey jack plant



Plate.2 Colour produced by dyes on mulberry silk



- a) Monkey jack
- b) Monkey jack with Copper sulphate
- c) Monkey jack with Ferrous sulphate
- d) Monkey jack with Stannous chloride

Fig.1 Optimization of alkali (Na_2CO_3) concentration for extraction

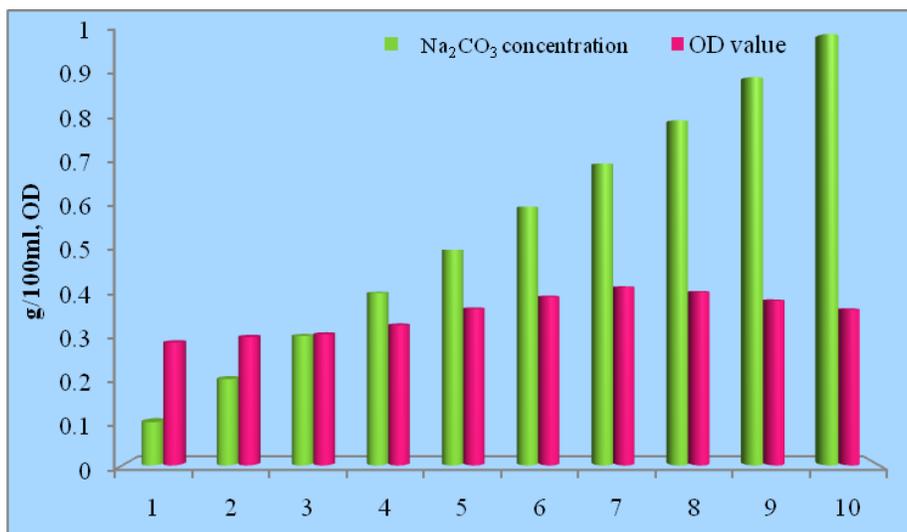
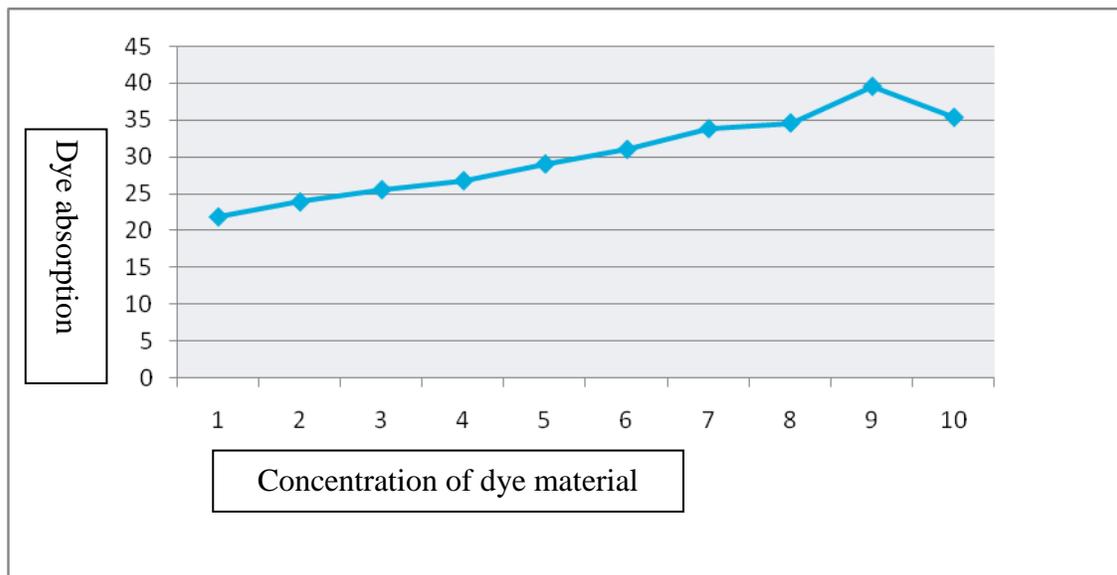


Fig.2 Optimization of dye material concentration



Optimization of mordant concentration

The concentration of CuSO_4 mordant showed maximum absorption (45.43%) at 1 per cent concentration. But as the concentration was increased the absorption (%) was decreased gradually and it was observed that concentration of FeSO_4 showed the highest absorption (38.04%) at 4 per cent concentration in monkey jack. The concentration of SnCl_2 showed maximum absorption (47.21%) at 1 per cent concentration for monkey jack.

Optimization of mordanting time

After optimization of mordant concentration, mordanting time for each mordant was optimized by dyeing mulberry silk yarns with optimized concentration of mordant for different time period viz. 15, 30, 45, 60 and 75 minutes in simultaneous mordanting method (Table 6).

From the results it could be concluded that 30 minutes duration of mordanting time was considered as suitable mordanting time for each mordant. Similar result has been shown

by Pandey *et al.*, (2014) on silk dyeing with binary mixture of *Ficus religiosa* and *Moringa pterygosperma* leaves.

Optimization of mordanting method for all the mordants

From the Table 7, it could be concluded that simultaneous mordanting method was considered as suitable mordanting method for each mordant. Similar observation has been obtained by Kumersan (2014).

Colour measurement

Table 8 shows L^* , a^* , b^* values for dyed samples. Tests were done with the help of Brightness Opacity and colour Tester, UEC1018. The monkey jack dye shows maximum a^* values (10.58). When the dye is mixed with metallic mordants, the redness got decreased. Chroma value shows the colour intensity. The silk yarn dyed with monkey jack in combination of stannous chloride mordant shows highest C value (17.72). The ferrous sulphate mordanted treated dye produced highest hue value (60.49). The total colour value of the dyed material is expressed

in terms of K/S. The least K/S value (0.89) was observed when the silk yarn is dyed with monkey jack dye. There is significant change in the K/S value of the shades dyed either with monkey jack alone or with mordent combination. The total colour value of the monkey jack dye got enhanced in combination with metallic mordants. The addition of mordant increased the K/S value. However, marginal change in the tone of shade was observed with various mordents.

Colour produced by dyes on mulberry silk

Different shades of colour produced after dyeing of mulberry silk yarn with the bark of monkey jack dye were shades of brown and shades of beige (Plate 2).

Evaluation of colour fastness properties

After dyeing in optimum dyeing conditions using different mordants the mulberry yarns were evaluated for their fastness properties like colour fastness to sunlight, colour fastness to washing, colour fastness to crocking (dry and wet), colour fastness to pressing (dry and wet) and colour fastness to perspiration (acidic and alkaline) by using International Grey Scale and compared with the controlled sample (Table 9).

From the table it is observed that, when monkey jack dyes were mordanted with copper sulphate showed very fair (4) colour fastness to sunlight. While ferrous sulphate, stannous chloride and without mordant showed moderate (3) colour fastness to sunlight. All mordanted dyed samples showed in the range of very fair to good fastness properties. In case of rubbing, all dyed samples showed good (5) colour fastness in dry condition. In perspiration, copper sulphate and stannous chloride mordanted sample exhibited good(5) colour fastness in acidic condition, however, the ferrous sulphate

mordanted samples showed comparatively lesser fastness. Ferrous sulphate and stannous chloride mordanted dyed sample showed good (5) and negligible (5) fastness properties in pressing. The overall colour fastness properties of sunlight, washing, rubbing (dry and wet), perspiration (acidic and alkaline) and pressing (dry and wet) were found to be good and satisfactory.

Sheth (2005) reported that light fastness of dye is not only influenced by chemical constituents of dye but also several other factors such as physical state of dye in the substrate, types of bond between dye and fibre, nature of substrate, source of radiation and its intensity, temperature, humidity condition, presence of foreign substance, atmospheric contaminants and after treatments given to dyed yarns/ fabrics for improving performance characteristics.

Colouring of mulberry yarns with different dyestuffs enhances the fibre as well as aesthetic value. In present study it has been found that the, alkaline medium was the most suitable method for dye extraction. The colour intensity of the treated yarns was varied due to using of different mordants. Silk yarn dyed with monkey jack in combination of stannous chloride mordant shows highest C value (17.72). The ferrous sulphate mordant along with monkey jack produced highest hue value (60.49). The total colour value of the dyed material is expressed in terms of K/S.

There is significant change in the K/S value of the shades dyed either with monkey jack alone or with mordant combination. The total colour value of the monkey jack dye got enhanced in combination with metallic mordants. The addition of mordant increased the K/S value. However, marginal change in the tone of shade was observed with various mordents. The overall colour fastness properties of sunlight, washing, rubbing (dry

and wet), perspiration (acidic and alkaline) and pressing (dry and wet) were found to be good and satisfactory.

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