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Effect of natural mordants on Eri matka silk yarn dyed with the bark of *Mangifera indica* dye

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Abstract

Eco-friendly natural dyes are highly recommended in recent years which are necessary to form a good sustainable environment. Eri matka silk yarn which is spun by hand with the help of an earthen pot found in Manipur is selected for the study. Optimization of various dyeing parameters of dye extracted from the bark of *Mangifera indica* was evaluated. Tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) is used as natural mordants to determine the effects of mordant. CIE lab and colour strength (K/S) of dyed yarns were also evaluated and observed that myrobalan (*Terminalia chebula*) mordanted yarn obtained a greater depth of colour than tea leaves (*Camellia sinensis*) mordanted. FTIR and SEM were evaluated and found that FTIR shows the difference of spectrum in the control and dyed eri silk yarn and SEM analysis observed that the dyed Eri matka silk seems to be more cylindrical and bigger in diameter than the control fiber. Finally, it enhances the quality of the yarn and produced different shades of yellow-orange and brown colour which exhibited fair to good ratings with slightly to negligible stain. It can be also said that the bark of *Mangifera indica* has good potential for dyeing silk yarn.

Keywords: Colourfastness, Eri matka silk, Mango bark, Mordant, Natural dye, Optimization

Introduction

Dyeing of textiles with eco-friendly naturally available sources was practiced by mankind since time immemorial. Natural dyes are known for obtaining numerous colour, non-toxic, non-allergic properties which are neither carcinogenic nor harmful to the environment (Sangeetha *et al.*, 2015) ^[13] and exhibit better biodegradability, and are more compatible with the environment. A wide range of plants and its part is used as natural mordants and they contain polyphenols compound which produces colouring component which includes tannin, catechin, gallic acid, ellagic acid, and so on. The polyphenolic water-soluble compound tannins have phenolic hydroxyl groups that facilitate for effective cross-links between proteins and other macromolecules. Tannins are the most important substance which is necessary and suitable for dyeing with natural dyes (Win and Swe, 2008) ^[16]. Myrobalan (*Terminalia chebula*) is the fruit of the trees usually contains major tannin constituent's viz. hydrolyzable tannins, chebulagic acid, chebulinic acid, gallic acid, and ellagic acid. Myrobalan contains 58-60% of ellagic acid by weight tannins in the peel of the fruit. It is a substitute for tannic acid and obtained yellowish brown colouring matter on textiles (Jain *et al.*, 2016; Shabbir *et al.*, 2016) ^[7, 14]. Tea (*Camellia sinensis*) is consumed widely as a beverage all over the world. Tea leaves present the polyphenol compound 'Catechin' which is used as a colourants on textiles (Farid, 2015) ^[6].

The mango (*Mangifera indica*) is considered an indigenous tree in Southern Asia, especially Myanmar and Assam state of India. The tree is evergreen; often reaching 15-18 metres (50-60 ft.) in height and requires optimum temperature ranges from 24 to 27°C (75-81°F), humidity (above 50%), and soil pH ranges 5.5-7.5 to grow (Bally, 2006) ^[3]. The mango bark content of 1 mg 100 g⁻¹ of tannin, 0.75mg 100 g⁻¹ of phenols, and 6.86 mg 100 g⁻¹ of flavonoids substances respectively which produce colour compounds used for the dyeing purpose of textile products (Okwu *et al.*, 2008) ^[9].

From Eri silkworm (*Samia Cynthia ricini*) open-ended cocoons spun silk known as Eri silk is

produced. It is also known as *Endi* or *Errandi* in Sanskrit nomenclature and is the only domesticated non-mulberry multivoltine silk (Banerjee *et al.*, 2018; Bondhare *et al.*, 2019) ^[4, 5]. It has a highly unique appearance that resembles cotton, is soft and durable as well as suitable for winter wear clothes. In Manipur, eri silk is spun by hand with the help of an earthen pot from the filaments of the cocoon and plied together by using a mud pot known as “Eri matka silk”. It has not uniform in diameter of the yarn and has less strength property than spun silk yarn with a spinning machine due to low twist. Eco-friendly dyeing with natural dyes and natural mordants is concentrated nowadays to minimize environmental issues. This study was planned and done to find out greener alternative dyed yarns to satisfy the consumers growing demands for eco-friendly products. Since there has not been any research work found particularly on the dyeing of eri matka silk yarn of Manipur and also considering the importance of natural dye, the present research work was conducted with an objective to optimize the dyeing parameters of selected dye on eri matka silk and to study the effect of natural mordants on colourfastness properties of natural dyes.

Methodology

Materials



Fig 1: Mango bark and dried powdered mango bark



Fig 2: Tea leaves and powdered tea leaves



Fig 3: Myrobalan and powered myrobalan



Fig 4: Raw eri matka silk yarn

Mango (*Mangifera indica*) barks were selected for the extraction of dye (Fig. 1) and tea leaves (*Camellia sinensis*) (Fig. 2) and myrobalan (*Terminalia chebula*) (Fig. 3) were selected as natural mordants and were collected from the Assam Agricultural University campus. Eri matka silk yarns (Fig. 4) with 46's count were selected and collected from Sangaitheh, Imphal West district of Manipur.

Extraction of dye

Barks of mango trees were washed thoroughly with water to remove dirt and dry properly under sunlight. An electrically power high-efficiency grinder machine is used to ground the processed barks into powder form and kept in an air-tight container in a dry and cool place. An aqueous medium is used for extraction of dye from barks of mango trees by taking 2 grams of dye material in 100 ml of distilled water at 100°C for 60 min. The extracted dye liquor was filtered with fine filter paper 3 times to get clear dye liquor. Dye absorption by the fiber was determine from the percentage of dye absorption by subjecting the dye solution before and after dyeing through visual light of a specific wavelength using a spectrophotometer.

Degumming

Eri matka silk yarns were degummed by immersing into the soda ash solution (5g/lit.) at 60 °C for 30 mins. The material-to-liquor ratio during the treatment was at 1:50. The eri matka silk yarns are taken out and washed thoroughly with 2g/L detergent at 65°C for 10 mins. (Uddin, 2015) ^[15].

Bleaching

The degummed eri matka silk yarns were bleached with 1% hydrogen peroxide solution at 50 °C for 30 minutes. The material to liquor ratio during the treatment was at 1:50 followed by washing with 2g/L detergent at 65 °C for 10 mins. (Uddin, 2015) ^[15].

Mordanting

The pre-mordanting method was carried out on eri matka silk yarn using 6% of tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) at 60 °C for 45 min. keeping the material-to-liquor ratio of 1:50.

Dyeing of eri matka silk yarn

Bleached eri matka silk yarns were weighed for dyeing at 70°C for 60 min by taking a material-to-liquor ratio of 1:30. The optical density obtained from the dye liquor before and after dyeing was recorded (Gogoi, 2004) ^[10]. The dye absorption (%) of the yarn determined by using the following formulae:

$$\% \text{ of dye absorption} = \frac{\text{OD of the liquor before dyeing} - \text{OD of the liquor after dyeing}}{\text{OD of the liquor before dyeing}} \times 100$$

Surface colour strength (K/S value) of non-mordanted and mordanted dyed silk yarn

The K/S value of the non-mordanted and mordanted dyed silk yarn was determined by measuring the surface reflectance curve between 570-610 nm using a computer-aided Macbeth 2020 plus reflectance spectrophotometer using Kubelka Munk equation.

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

Where K = the coefficient of absorption; S = the coefficient of scattering; C_d = the concentration of the hue and R = the surface reflectance value of the dyed sample at a particular wavelength, where maximum absorption occurs for a particular dye/colour component (Kumarasen, 2018) [8].

Colour co-ordinates of dyed silk yarn

The colour coordinate value of all the dyed samples was determined by CIE Lab system using spectrophotometer by taking an average of five times readings in Brightness, Opacity, and Colour Tester. 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 to show the colour difference between mordanted and non-mordanted samples. The lower value of L^* determines the higher depth of colour.

a^* = redness (positive a^*) and greenness (negative a^*)

b^* = yellowness (positive b^*) and blueness (negative b^*)

Fourier Transform Infrared Spectroscopy (FTIR)

A double-beam Fourier Transform Infrared Spectroscopy (FTIR) spectrophotometer was used to determine Fourier Transform Infrared Spectroscopy (FTIR) for control and dyed

silk yarn. The frequency range is measure as wave numbers typically over the range of 4000-600 cm^{-1} . The resultant absorption spectrum indicates the presence of various chemical bonds and functional groups in the sample.

Scanning Electron Microscopy (SEM)

To assess the surface morphology of the samples, Scanning electron microscopy (SEM) was done for control and dyed silk yarn using field emission Scanning Electron Microscopy (SEM) (make-Carl Zeiss, model-Sigma). High-quality and spatial resolution was used to observe the detailed visual image of the sample. The fibres cut into fine pieces with the help of sharp scissors and then mounted on specimen holders with the help of electro conductive tapes. The samples were coated with gold in an ion sputter coater a low vacuum with a layer 150-200 nm thick and examined with 1000X magnification.

Evaluation of colourfastness

Colour fastness to sunlight, washing, crocking (dry and wet), perspiration (acidic and alkaline), pressing (dry and wet) for all the dyed eri matka silk yarn samples were done by using AATCC (1965) method. Colour Change (CC) and Colour Staining (CS) parameters of dyed samples were evaluated using ASTM (1968) Gray Scale.

CC Ratings: 1= very poor, 2= poor, 3= fair, 4= very fair, 5= good, 6= very good

CS Ratings: 1= heavily stained, 2= considerably stained, 3= noticeable stained, 4= slightly stained, 5= negligible or no staining

Results and discussion

Optimization of dyeing conditions

Table 1: Optimization of dyeing condition for the selected dye

Dye extraction time				Dye material concentration			Dyeing time		
Sl. No.	Dye extraction time (min)	Temperature (°C)	Optical density value	Dye material concentration (g/100ml)	Extraction time (min)	Dye absorption (%)	Dyeing time (min)	Temperature (C°)	Dye absorption (%)
1	30	100	0.355	1g	75	24.23	30	70	31.53
2	45	100	0.397	2g	75	27.03	45	70	37.38
3	60	100	0.437	3g	75	33.87	60	70	39.03*
4	75	100	0.456*	4g	75	37.02*	75	70	24.17
5	90	100	0.439	5g	75	21.52	90	70	23.27
6	105	100	0.415	6g	75	20.20	—	—	—
7	120	100	0.395	—	—	—	—	—	—

The results of optimum dyeing conditions are shown in Table 1. The optimum dye extraction condition was considered based on highest optical density measured by using spectrophotometer. The highest optical density value i.e. 0.456 in 75 minutes duration. Hence, 75 minutes was considered as optimum time for extraction of dye from mango bark. The optimum dye

material concentration and dyeing time were considered based on maximum dye absorption percentage (%). The maximum dye absorption percentage (%) i.e. 37.02% was found in 4% concentration of dye for eri matka silk yarn. The maximum dye absorption percentage (%) i.e. 39.03% was found in 60 mins. of dyeing time.

Optimization of mordanting condition

Table 2: Optimization of mordanting condition for the selected dye

Sl. No.	Name of the mordant	Mordant concentration		Mordanting time	
		Concentrations of mordant (%)	Dye absorption (%)	Time of mordanting (min.)	Dye absorption (%)
1	Tea leaves	2	13.84	15	16.71
		4	16.05	30	17.62
		6	16.78*	45	20.66*
		8	14.87	60	13.67
		10	13.69	75	11.09
2	Myrobalan	2	12.37	15	16.53
		4	13.10	30	20.54
		6	18.26*	45	22.15*
		8	16.20	60	19.90
		10	15.75	75	12.84

The results of optimum mordanting conditions are shown in Table 2. The optimum mordanting conditions such as mordant concentration and mordanting time were considered based on maximum dye absorption percentage (%). The maximum dye absorption percentage (%) i.e. 16.78% was found in 6% mordant concentration and 20.66% was found in 45 mins. of mordanting time in case of dyeing of eri matka silk yarn using tea leaves (*Camellia sinensis*) as mordant. The maximum dye absorption

percentage (%) i.e. 18.26% was found in 6% mordant concentration and 22.15% was found in 45 mins. of mordanting time in case of dyeing of eri matka silk yarn using myrobalan (*Terminalia chebula*) as mordant.

Dyeing of eri matka silk with different mordants Tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*):



Fig 5: Control and dyed yarn

Different shades of colour obtained after dyeing and mordanting of eri matka silk yarn with mango bark dye in optimized condition were yellow ochre, tawny gold, and dark raw sienna colour are shown in Fig. 5.

Surface colour strength (K/S value) non-mordanted and mordanted dyed silk yarn

Table 3: Evaluation of Colour Strength (K/S) values of mango bark dyed yarn

Dyed yarns	Colour Strength (K/S value)
Non-mordanted	21.64
Tea leaves mordanted	23.53
Myrobalan mordanted	25.75

The results of colour strength (K/S) value for both non-mordanted and mordanted dyed silk yarn are shown in Table 3. K/S value of the non-mordanted dyed silk yarn was found less compared to mordanted yarn. Myrobalan mordanted dyed yarn obtained the highest K/S value 25.75 followed by tea leaves mordanted dyed yarn i.e. 23.53.

The presence of a hydroxyl group in the structure of tea leaves mordant and dye obtained from mango bark and a carboxyl group present in the structure of myrobalan mordant is capable to form a metal complex with a positive charge amino group of silk yarn.

CIE lab values

Table 4: CIE lab colour co-ordinate values of mango bark dyed yarn

Dyed yarns	CIE Lab values		
	L*	a*	b*
Non-mordanted	67.03	9.88	30.44
Tea leaves mordanted	59.88	8.97	32.86
Myrobalan mordanted	49.53	8.29	28.32

The results of CIE lab values for both non-mordanted and mordanted dyed silk yarn are shown in Table 4. The non-mordanted dyed sample showed the highest L* value of 67.03 compared to an L* value of 59.88 in tea (*Camellia sinensis*) leaves mordanted and L* value of 49.53 in myrobalan (*Terminalia chebula*) mordanted dyed silk yarn. Therefore, the L* values of the dyed yarns decreased with the use of mordant which indicates that the shade gets darker as compared to non-mordanted dyed yarn. In non-mordanted and mordanted yarns, a* and b* values were studied. The positive values of a* and b* indicated the redness and yellowness present in the dyed yarn.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR for control and dyed silk yarn samples were determined and observed that the spectrum in the control yarn was found different from the spectrum of the dyed yarn (Fig.6).

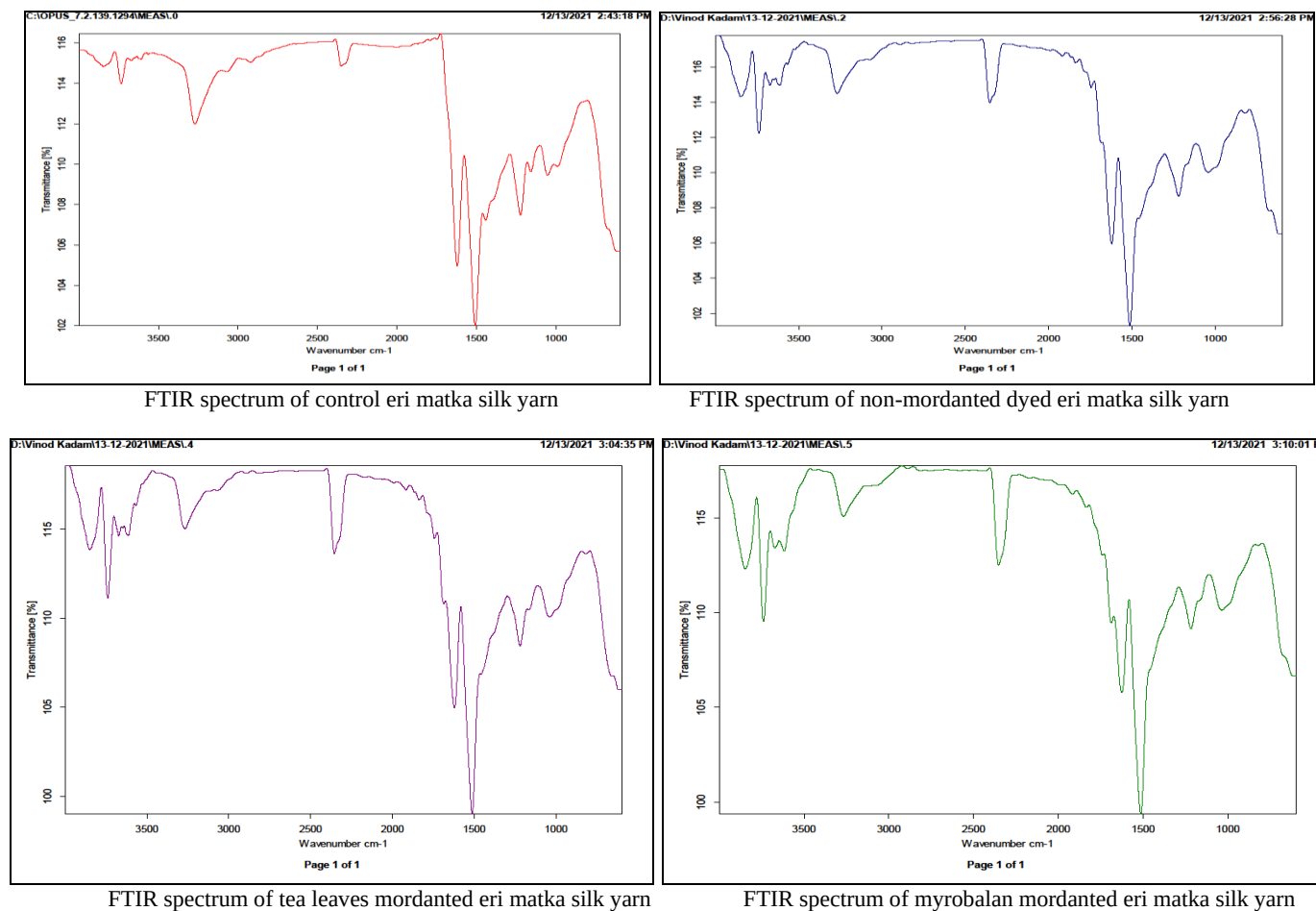


Fig 6: FTIR spectrum for control, non-mordanted and mordanted dyed eri matka silk yarn

The peaks of all the samples indicated the presence of functional groups. Peaks in between 1625-1650 cm^{-1} imply the presence of an alpha-beta unsaturated carboxyl group which is a characteristic of yellow flavonoid pigment (Nissankararao, 2011) [12]. All the figures showed specific peaks between above mentioned range. Stretches band found around 1610-1570 cm^{-1} depicts the presence of an aromatic ring and stretches between 3030-3010 cm^{-1} depicts the presence of C-H bond which is usually considered a band for flavonoid pigments (Aksoz and Ertan, 2012) [2].

The peaks between 1610-1570 cm^{-1} and 3030-3010 cm^{-1} in all the figures determined the presence of flavonoid in the dye pigment.

The spectrum revealed that the control and dyed yarn mordanted with tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) showed different peaks and different functional groups. This might be due to pre-treatment processes like degumming and bleaching and various other dyeing processes.

Scanning Electron Microscopy (SEM)

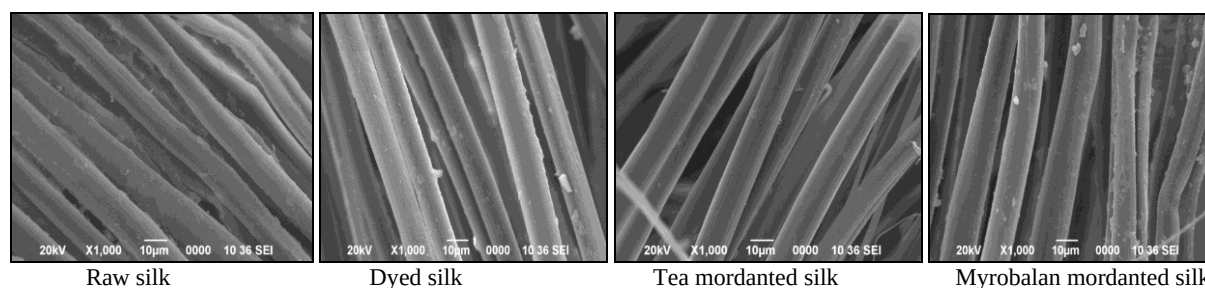


Fig 7: SEM images of raw and dyed silk yarn at 1000X

SEM for control and dyed silk yarn samples were determined and observed (Fig.7). The control silk showed irregularities and gummy substances on the surface of the rod-like structure of the fiber. This might be due to the presence of sericin in the control

silk fiber. The dyed silk fibre showed a smooth cylindrical surface with a bigger size and this might be due to the removal of sericin by degumming and bleaching pre-treatment and also the presence of dye in the fiber of the dyed yarn.

Colourfastness properties of dyed eri matka silk yarns**Table 5:** Rating for colourfastness properties of dyed samples

Sl. No.	Mordant used	Sunlight	Washing		Crocking				Perspiration				Pressing			
			CC	CS	Dry		Wet		Acidic		Alkaline		Dry		Wet	
					CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CS
1	Non-mordanted	4	5	4	5	5	5	5	3	4	3	5	5	5	5	5
2	Tea leaves mordanted	5	5	4	5	5	5	4	4	4	4	4	5	5	5	4
3	Myrobalan mordanted	5	5	4	5	5	5	4	4	4	4	4	5	5	5	4

The results of colourfastness properties of dyed eri matka silk yarns are shown in Table 5. Sunlight fastness as shown in Table 5 was found that the dyed yarns mordanted with tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) showed good colour fastness while non-mordanted dyed sample showed very fair fastness property. Washing fastness was found that non-mordanted and the dyed yarns mordanted with tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) showed good colour fastness and colour change. Slightly colour stained in all the dyed yarn samples during washing. Crocking fastness was found that non-mordanted and the dyed yarns mordanted with tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) showed good colour fastness and negligible stained in dry crocking. On the other hand, in wet crocking, all the samples were found good fastness. No colour stained was occurred in non-mordanted dyed yarns and slightly stained in mordanted samples. All the dyed yarn samples were evaluated for colourfastness to perspiration in both acidic and alkaline medium. In acidic medium, all the samples were found fair to very fair fastness in perspiration. Slightly stained was obtained in all the dyed samples. In alkaline medium, all the dyed yarn samples were obtained very fair to fair colourfastness to perspiration and negligible colour stained in non-mordanted dyed yarn while tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) mordanted dyed yarn showed slightly stained.

All the samples of non-mordanted and mordanted with tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) were observed good fastness and no stained to dry pressing while in wet pressing all the samples showed good fastness and slightly stained except non-mordanted sample showed no stained to wet pressing.

Conclusion

From the present study, it was concluded that dyeing of eri matka silk with mango bark dye in presence of natural mordants tea leaves (*Camellia sinensis*) and myrobalan (*Terminalia chebula*) obtained fair to good ratings with slightly to negligible stained. The highest K/S value 25.75 and the darker shades with L* value of 49.53 found in Myrobalan (*Terminalia chebula*) mordanted dyed silk yarn. It is recommended from the study that mordant Myrobalan (*Terminalia chebula*) is more suitable as natural mordant for dyeing eri matka silk yarn with natural dyes.

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