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Natural dye, an innovative eco-friendly approach

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Abstract
Dyes obtained from plants, invertebrates, minerals etc. are known as natural dyes. In the recent past, synthetic dyes had gained popularity due to ease in processing. But in recent times, rebirth of natural dyes as an alternative to the hazardous synthetic dyes has received a worldwide attention. This is virtually a global hunt for eco-friendly dyes as they are non-toxic, biodegradable and safe. Eri silk (*Samia ricini*), is known as ahimsa silk, as it is much more humane to the creatures creating the silk and North-east India is known as the homeland of Eri silk. Combination of different natural dyes produces different shades. Dyeing eri silk with different natural dye enhances the quality and aesthetic value of the fabric. Moreover, it gives a new look to the fabric so as to help in product diversification. And being eco-friendly it can be effectively used for dyeing. With this consideration, a study was undertaken in College of Community Science, Assam Agricultural University, Jorhat-13 on dyeing of eri silk fabric with combination of two natural dyes i.e. annatto (*Bixa orellana*) seed and fresh tea (*Camellia seninsis*) leaves. In this study, the dyeing was carried out in aqueous as well as alkali medium by using different mordant such as Aluminium potassium sulphate [$Alk(SO_4)_{2.}$ 16 H_2O], Copper sulphate($CuSO_4$), Stannous chloride($SnCl_2$) and Ferrous sulphate($FeSO_4$) and also mordanting methods. Combination of different mordants and mordanting methods resulted in beautiful hues. The colour fastness properties of dyed fabrics were recorded to be very fair to good, as per the rating scale. The colour yield obtained from combination of annatto seed and tea leaves on dyed eri silk fabric were dark goldenrod, old gold, sepia and taupe.

Keywords: Natural dye, Eri silk, Combination, Colour fastness, Product diversification

Introduction

Colouring refers to applying a colour producing material onto a fabric physical means, such as by painting, rubbing or pressing it into the fibre. This coating process yielded colours on fabrics that were, in general, not very fast or stable to washing. The process of dyeing a textile refers to the fixing of a colouring substance into the textiles by chemical bonding which is produced between the dye and the fibre.

Eri (*Samia ricini*), also known as endi or erndi, ranks next to tassar in commercial importance. It is described as a silk of incredible durability. The eri silk yarns are generally woven to fabric which is used as winter wrapper. The eri cloth can be an excellent material for shirting, suiting, bed spread, neck tie, curtain, dress materials and other furnishing items [10].

Dyeing eri silk with different dyestuffs enhance the quality of fabric as well as its aesthetic value. Dyeing makes the natural grey or beige colour of eri silk more attractive. Natural dyes have the property of eco-friendliness so it does not create any environmental problems at the stage of production or use and maintain ecological balance. They are non-toxic and non-allergic having soothing effects and aesthetic value. The use of natural dyes on textiles has been one of the consequences of increased environmental awareness worldwide. Use of natural dyes for the colouration of textile has mainly been confined to craft dyers and printers [6]. Natural dyes are mostly subtle and dull colour and bright colours are generally not found in natural dyes. But different shades can be obtained by combining two or more natural dyes or by combining different mordants [8]. While eri silk is a naturally obtained protein fibre and it does not create any health hazards at the stage of production. Combination of natural dyes with eri silk will produce eco-friendly product. Eri silk is dull in colour, so dyeing of eri silk by combining two natural dyes will produce a different shade, so aesthetic value of eri silk will be changed and gives a wider scope for product diversification.

Considering the increasing popularity of using natural dyes as well as the potentiality of eri silk in global market the present study was undertaken with the following objectives: to optimize the dyeing conditions of selected dye for eri silk fabric and to evaluate the colour fastness properties of dyed fabric.

Materials and Method

For conducting the study the Annatto seed (*Bixa orellana*) were collected from Chowldhuwa of Lakhimpur district of Assam. The collected seeds were, cleaned and dried and tea leaves (*Camellia assamica*) were collected from tea garden of Assam Agricultural University, Jorhat. The tender tea leaves from below the third leaves which were not required for manufacturing of tea were selected for the study. The collected leaves were cleaned, washed and dried as the colour obtained from dry leaves are brighter than fresh ones. Eri silk (*Samia ricini*) fabric with plain weave were collected from local market of Jorhat.

Chemicals used

Chemical used for the study were Hydrogen peroxide, Soda ash/ sodium bicarbonate, Aluminium potassium sulphate (metallic salt)/ alum, Sodium chloride and Lactic acid, USP 85% of LR grade.

Mordants used

Four mordants namely Aluminium potassium sulphate (metallic salt)/ alum [$Alk(SO_4)_2.16H_2O$], Copper sulphate ($CuSO_4$), Ferrous sulphate ($FeSO_4$) and Stannous cloride ($SnCl_2$) were used for the study. Mordants are the substance capable of binding a dye to textile fabrics [7]. Mordant helps to produce faster shades by forming an insoluble compound of mordant and dyestuffs within the fiber itself [2, 9].

Methods

Degumming

Before dyeing degumming is done for removing the sericin and uniform dyeing. For this the silk fabric were boiled in washing soda solution (5g/lit) at 60^0 C for 30 min. Fabric were washed properly in running water and dried.

Bleaching

Bleaching improves the whiteness property and also imparts uniform absorbency and high degree of dyeability. For this the silk fabric were boiled in 1% H_2O_2 at 50 ^{0}C for 30 min. Fabric were washed properly in running water and dried.

Optimization of different dyeing conditions

A series of experiments were conducted to optimize the different dyeing conditions namely dye extraction medium, dye extraction time, dye material concentration, combination ratio, dyeing time, concentration of mordants, mordanting time, mordanting methods etc. for dyeing of eri silk fabric with tea(*Camellia seninsis*) and annatto (*Bixa orellana*) dye.

In all the experiments, the values of some of the variables like material to liquor ratio for extraction (1:50), material to liquor ratio (1:30) for dyeing, extraction temperature (100 °C) and dyeing temperature (70 °C) for the study were kept constant based on some research findings (3,4,5).

Extraction of dye

For the selection of extraction medium 3 methods were selected i.e. aqueous, alkaline and acidic and tested, and it was found that aqueous medium was more suitable based on percent dye extraction. In this method 1gm dye material were mixed in 100ml of soft water at 100 ^{0}C for 1 hour.

Dyeing of eri silk fabric

The eri silk fabrics to be dyed were weighed. The extracted dye liquor was taken as per requirement at material to liquor ratio 1: 30. The optical density of the dye liquor was recorded. The eri silk fabrics were placed in the dye liquor and dyed for 45 minutes in the dye bath with occasional stirring. After completion, the fabrics were removed and the optical density of the liquor was recorded. The fabrics were then soaped, washed, rinsed and dried in shade. The percentage of dye absorption (%) of the fabric was estimated by using the following formulae:

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

Optimization of mordanting methods

Optimizations the pre, post and simultaneous mordanting methods. Absorption (%) of dye by the eri silk fabric was calculated from optical density values for different mordanting methods. The method showing the maximum dye absorption was selected as the optimum method for each mordant.

Pre-mordanting method

Pre-mordanting method was done by mordanting the fabric first and then dyed. For this method, an aqueous solution was prepared by dissolving required amount of mordant in water. The fabric were boiled in 70 ºC in this solution for 30 minutes and then entered in the prepared dye solution for dyeing.

Simultaneous mordanting

In this method, the mordants and dye were applied in the same bath. The eri silk fabric were placed in the extracted dye bath and dyed for 15 minutes. The required amount of mordants were added to the dye solution by lifting fabric and mixed properly. The fabrics were then dyed in the solution for 30 minutes.

Post mordanting

In this method the sample 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 a 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.

Optimization of combination ratio of tea and annatto
The combination ratio was optimized by extracting tea: annatto dye at different ratios *viz.,* 10:90, 20:80, 30:70, 40:60, 50:50 and vice versa with both the dye, and optical densities of the extracted dye liquor for different ratios were recorded. Dye extraction ratio was optimized based on maximum optical density value obtained by the dye liquor for different ratios.

Evaluation of colourfastness
The dyed samples of eri 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 [1] procedure.

Findings and Discussion
Optimization of combination ratio
The combination ratio of tea and annatto dye was optimized by dyeing eri silk fabrics in different combination ratio of dye. It was cleared from Table 1, that dye absorption was found maximum (64.44%) in 70:30 ratio (70% tea & 30% annatto) of selected dye.

Optimization of dyeing time
Data obtained from Table 2, revealed that maximum absorption (68.48%) of dye by the fabric was found at 45 minutes of dyeing time. The absorption (%) by the fabric was decreased gradually by the increasing the dyeing time. Hence, 45 minutes duration of dyeing time was considered as optimum time, as it showed maximum absorption by the eri silk fabric.

Optimization of mordant concentration
It was evident from the Table 3, that, the maximum absorption (59.39%) was found in 6 percent concentration of alum. In the case of $CuSO_4$ 3% concentration showed maximum absorption (84.01%). In $FeSo_4$ absorption of dye was found maximum (75.48%) in 3 percent concentration. The concentration of stannous chloride showed maximum absorption (68.74%) in 2 percent concentration. Hence 6%, 3%, 3% and 2% concentration of mordant was selected as optimized for alum copper sulphate, ferrous sulphate and stannous chloride, respectively.

Optimization of mordanting time
From Table 4, it was evident that the highest absorption percentages of dye were obtained in 30 min of mordanting time of each mordant. Therefore, 30 min mordanting time was considered as the optimum time for mordanting for eri silk fabric.

Optimization of mordanting method
From the Table 5, it was clear that all the mordants show maximum absorption in simultaneous mordanting method except the alum. It shows maximum absorption at pre mordanting method. Hence simultaneous mordanting method was considered as suitable mordanting method for copper sulphate, ferrous sulphate and stannous chloride and pre mordanting method is suitable for alum.

Colourfastness properties of dyed eri silk fabric
From the Table 6, it was interesting to know:-

Colour fastness to sunlight
Samples dyed with without mordant alum, ferrous sulphate and stannous chloride mordanted showed very fair colour fastness to sunlight. While copper sulphate mordanted sample showed fair fastness to sunlight.

Colour fastness to crocking
The dyed samples were tested for colourfastness to dry and wet crocking and the result shows good fastness to crocking by all samples dyed without mordant, alum, copper sulphate, ferrous sulphate, stannous chloride and no staining was observed for dry crocking. Colour fastness to wet crocking showed good fastness by all the samples dyed without mordant, alum, copper sulphate, ferrous sulphate and stannous chloride mordanted. And slightly colour stain was observed in without mordant, alum and ferrous sulphate mordanted sample.

Colour fastness to washing
The samples without mordant and mordanted with ferrous sulphate showed very fair fastness to washing while alum, copper sulphate and stanneous chloride mordanted showed good fastness and all the samples do not show any colour stain.

Colourfastness to perspiration
The dyed eri samples were tested for colour fastnesss to perspiration (acidic and alkali). In alkaline perspiration sample without mordant, copper sulphate and ferrous sulphate mordanted showed very fair fastness and slightly stained was observed. While other samples mordanted with alum and stannous chloride showed good colour fastness and no colour stain was observed.
In acidic perspiration, samples mordanted with alum, copper sulphate and stannous chloride showed good colourfastness and no colour stained was observed. While samples without mordant and ferrous sulphate mordanted showed very fair colour fastness and slightly stained was observed.

Colourfastness to pressing
All samples without mordant, mordanted with alum, copper sulphate, ferrous sulphate and stannous chloride showed good fastness to dry and wet pressing and no colour stain observed.

Table 1: Optimization of combination ratio (tea:annatto)

Sl. No.	Combination ratio	Dye absorption (%)
1.	10:90	56.81
2.	20:80	36.03
3.	30:70	32.28
4.	40:60	21.84
5.	50:50	19.49
6.	60:40	46.34
7.	70:30	64.44
8	80:20	40.28
9	90:10	34.13

Table 2: Optimization of dyeing time

Sl. No.	Temperature (ºc)	Wavelength (nm)	Dyeing time (min)	Dye absorption (%) (70:30)
1	70	460	15	45.49
2	70	460	30	51.26
3	70	460	45	68.48
4	70	460	60	50.58
5	70	460	75	39.41
6	70	460	90	28.85

Table 3: Optimization of mordant concentration

Name of mordant	Mordant concentration	Dye absorption (%) (70:30)
Alum	2	36.98
	4	50.42
	6	79.39
	8	62.48
	10	50.62
	12	41.85
Copper sulphate	1	62.86
	2	70.48
	3	84.01
	4	74.28
	5	60.42
	6	51.68
Ferrous sulphate	1	53.26
	2	64.25
	3	75.48
	4	60.98
	5	48.26
	6	36.89
Stannous chloride	1	51.28
	2	68.74
	3	46.34
	4	31.49
	5	28.26
	6	21.89

Table 4: Optimization of mordanting time

Name of the mordant	Mordanting time (min)	Dye absorption (%) (70:30)
Alum	15	52.47
	30	57.61
	45	51.91
	60	32.60
	75	29.81
Copper sulphate	15	38.26
	30	46.51
	45	34.16
	60	29.01
	75	21.20
Ferrous sulphate	15	47.16
	30	56.15
	45	50.51
	60	42.21
	75	34.61
Stannous chloride	15	51.26
	30	62.48
	45	48.29
	60	35.48
	75	30.64

Table 5: Optimization of mordanting method for all the mordants

Mordant	Mordanting method	Dye absorption (%) (70:30)
Alum	Pre	59.68
	Simultaneous	42.26
	Post	33.48
Copper sulphate	Pre	31.54
	Simultaneous	48.26
	Post	24.10
Ferrous sulphate	Pre	43.26
	Simultaneous	60.42
	Post	40.82
Stannous chloride	Pre	58.48
	Simultaneous	71.26
	Post	54.16

Table 6: Ratings for colourfastness properties of dyed samples (70:30)

Sl. No.	Mordant used	Sunlight	Washing		Crocking				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	4	4	5	5	5	5	4	4	4	4	4	5	5	5	5
2.	Alum	4	5	5	5	5	5	4	5	5	5	5	5	5	5	5
3.	Copper sulphate	3	5	5	5	5	5	5	5	5	4	4	5	5	5	5
4.	Ferrous sulphate	4	4	5	5	5	5	4	5	5	4	4	5	5	5	5
5.	Stannous chloride	4	5	5	5	5	5	5	4	4	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

Conclusion

From the above study it has been found that combination of tea and annatto dye enhances the colour of eri silk fabric, which will boost in preparing diversified products. Such efforts are required to improve the qualities and aesthetic value of this poor man's silk to match with new trends in national and international market. On the basis of the findings of this study improvement in dyeing processes of eri silk with natural dyes may be incorporated in order to obtain wide consumer acceptance.

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