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Research Paper :

Value addition of eri silk with annatto – a natural colourant

NABANEETA GOGOI

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Correspondence to:
NABANEETA GOGAI
Department of Clothing and Textiles, College of Home Science, Assam Agricultural University, JORHAT (ASSAM) INDIA

ABSTRACT

Eri silk, also known as poor man's with natural beige colour ranks next to tassar silk in commercial importance. The beautiful eri fabric can be an excellent material for shirtings, suitings, dress materials, bed spreads and other furnishings. Colouring of eri silk with annatto dye enhance the fabric as well as its aesthetic value and marketability. Various dyeing conditions were optimized with different mordants for dyeing eri silk with annatto and a little decrease was observed in breaking strength and other physical properties of dyed eri silk. But the colours obtained from annatto were very much colourfast, irrespective of mordants.

Key words : Erisilk, Colour, Dress materials, Annatto

Eri silk also known as endi or erandi, ranks next to tassar silk in commercial importance. It is the product of domesticated silk worm, *Philosomia ricini* that feeds mainly on castor leaves. The beautiful eri fabric which is known for its durability is a regular winter wrapper for Assamese people. This lack luster hand spun silk though subdued in shine has the usual suppleness of other silks. Cottonist in texture, eri has the warmth of wool. The eri cloth can be an excellent material for shirtings, suitings, neck tie, bed spreads, curtains and other furnishings. It is felt that product diversification of eri yarn might enhance the marketability of eri silk. Proper attention in some of the properties of eri silk like colour, gloss, feel etc. can be improved so as to utilized this "poor man's silk" for making attractive products.

The revival of the use of natural dyes world-wide is primarily due to the increasing environmental consciousness to-day. Colouring of eri silk with natural dye enhance the fabric as well as its aesthetic values. This silk has potential and wide scope for improvement in its properties, so that eri silk can be utilized for making diversified products, which can play a significant role in improvement of the rural economy as well as its demand in the market. Considering the importance of eri silk and also the eco-friendly dyeing, the present work was undertaken with the following objectives : to optimize the dyeing conditions of selected dye for dyeing eri silk and to evaluate the colour fastness and physical properties of dyed eri silk.

METHODOLOGY

For conducting the study the following materials and methods were selected and used.

Eri silk yarns were used for the study and it was purchased from local source.

Table 1 : Natural dyes used for dyeing eri silk yarn

Local name	English name	Scientific name	Parts used
Phuku guti or Hat-ranga	Annatto	*Bixa orellana*	Seeds

The annatto seeds are coated by a yellowish orange substance, which is the dye. It is one of the very few bright natural dyes available for textiles especially for silk and cotton. Annatto yields an orange dye based on the carotenoid structure. The proportion of the dye varies between 10-12% on the weight of the seed. Annatto dyes behave as an acid dye on protein fibres at an acidic pH. The chief colouring matter is Bixin. The seeds and surrounding pulp of annatto is rich in tannin and contains a mixture of eight colourants of carotenoid group of which the two main carotenoids are Bixin and Cis-Bixin containing vitamin A (Devi *et al*., 2002; Gulrajani, 2001 and Teli *et al*., 2001).

Chemicals used:

The chemicals used in the research work are given in Table 2.

Mordants used :

As regards the selection of mordants, the eco-friendly mordants namely Aluminium sulphate (alum), copper sulphate and ferrous sulphate were selected for the present work. As mordants helps to link between dye stuff and fibre which allows the dye with no affinity for the fibre to be fixed.

Plate 1 : Annatto plant and seeds

Table 2 : Chemicals used

Sr. No.	Name of the chemicals	Molecular formula	Purpose
1.	Sodium carbonate	Na_2CO_3	For extraction of dye
2.	Urea	NH_2CONH_2	For colourfastness test
3.	Acetic acid	CH_3COOH	For colourfastness test
4.	Sodium bicarbonate	$NaHCO_3$	For colourfastness test
5.	Sodium chloride	$NaCl_2$	For colourfastness test

Degumming :

Before deying, degumming has become an important preparatory process for uniform dyeing. For this, silk yarns were boiled in washing soda solution (5 g/lit) at 60°C for 30 minutes. Yarns were washed properly in running water and dried.

Extraction of dye :

For extraction of dye, the three medium namely aqueous, acid and alkali were tried and out of these, alkaline medium, was found to be the best, based on per cent dye extraction. In this method, 1% solution was prepared with the addition of 1 gm sodium carbonate in 100 ml of water. 2 gm dye material was added and heated at 90°-100°C for different time period. The cooled dye solution was then filtered. Extraction time was optimized based on maximum optical density of the extracted dye solution.

Optimization of different dyeing conditions:

For dyeing eri silk yarn with annatto dye, a series of optimization experiments such as extraction of dye, dye material concentration, dyeing time, concentration of mordants, mordanting time, mordanting method etc. carried out to ascertain the best dyeing condition.

Dyeing temperature (70°C) and material to liquor ratio for dyeing (1:30) were kept constant based on some research findings (Gogoi, 2004; Bansal *et al*., 2006).

Mordanting methods :

Three mordanting methods namely pre-mordanting, simultaneous mordanting and post-mordanting methods were used for the present work and is presented as under.

Pre-mordanting :

In this method the yarns were mordanted in the first stage and then dyed in the second stage. Optical density of the dye liquor before and after dyeing was recorded.

Simultaneous mordanting :

In this method, the mordants and the dye were applied simultaneously in the same bath. First, the optical density of the extracted dye liquor was recorded and then the yarn samples were entered in the dye liquor and boiled for 15 minutes. Required amount of mordants were then added to the dye solution and boiled for 30 minutes with occasional stirring. Again optical density of the dye liquor was recorded and samples were then washed, rinsed and dried in shade.

Post mordanting :

In this method the yarns were first dyed and then mordanted. Dye solution was prepared first and then optical density was recorded. Yarns were then dyed in the dye solution for 45 minutes and the optical density of the dye liquor after dyeing was also recorded.

An aqueous solution was prepared by adding required amount of mordant and then boiled the yarn samples in the mordanting liquor for about 30 minutes. Samples were then washed, rinsed and dried in shade.

Dyeing of eri silk :

The extracted dye liquor was taken as per requirement at material to liquor ratio 1:30. The optical density of the dye liquor before dyeing was recorded. The eri silk yarns were placed in dye liquor and dyed for 45 minutes in the dye bath with occasional stirring. After completing, the yarns were removed and the optical density of dye liquor was recorded. The percentage of

dye absorption (%) of the yarn was estimated by using the following formula.

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

Test for colourfastness

The dyed eri silk yarns were evaluated for colour fastness to washing, sunlight, crocking, pressing and perspiration by following standard procedure laid down by BIS No. IS: 3361-1979, IS: 686-1957, IS: 766-1956, IS: 971-1956, respectively.

Evaluation of physical properties

Prior to testing, all the samples were conditioned to attain moisture equilibrium and tested in the standard atmospheric condition of 65±2 per cent relative humidity and temperature 25°±2°C as per ASTM standard (1968). Sample were prepared as per BIS method (IS : 6359-1979).

The dyed yarns were evaluated for different physical properties like breaking load (kg), breaking strength (g/tex), elongation (%) and stress (%) by adopting the BIS method (IS : 6359-1979).

FINDINGS AND DISCUSSION

The findings of the research work are discussed below :

Optimization of dye extraction time:

It was evident from Table 3, that the maximum (0.289) optical density value was found in 60 min of extraction time. Hence, 60 min of extraction time for annatto dye might be considered as suitable extraction time.

Table 3 : Optimization of dye extraction time

Dye	Extraction time (min)	Wavelength (nm)	Optical density
Annatto seed	30	450	0.230
	45	450	0.244
	60	450	0.289
	75	450	0.265
	90	450	0.210

Optimization of alkali concentration for extraction of annatto dye:

From the Table 4, it was evident that highest optical density value (0.305) was found in 1% concentration of Na_2CO_2 and lowest was found in 3% concentration.

Table 4 : Optimization of alkali concentration

Alkali concentration (g/100 ml) (%)	Wave length (nm)	Optical density value
0.5	450	0.288
1.0	450	0.305
1.5	450	0.292
2.0	450	0.285
2.5	450	0.268
3.0	450	0.245

Optical density value showed decreasing trend as the concentration of alkali increased. So, 1% concentration of Na_2CO_2 was considered as optimum for extraction.

Optimization of dyeing time:

Data depicted in Table 5 showed that the maximum dye absorption was recorded (35.57%) in 45 min of dyeing for eri silk yarn. After 45 min of dyeing time, the absorption of dye by the yarns started decreasing gradually as evident from the table. Hence, 45 min was optimized as dyeing time for eri silk.

Table 5 : Optimization of dyeing time

Dying time (min)	Wave length (nm)	Dye absorption (%)
30	450	32.01
45	450	35.57
60	450	32.58
75	450	27.81
90	450	25.68
105	450	22.29

Optimization of concentration of dye:

Table 6 revealed that dye absorption (%) was found maximum (62.55%) in 2% concentration of annatto dye and as the dye concentration were increased the absorption (%) decreased gradually and lowest absorption (12.84%) was found in 6% concentration of dye. Hence, 2% concentration might be considered as the most suitable concentration of dye for dyeing eri silk yarn.

Table 6 : Optimization of concentration of dye

Dyeing time (min)	Concentration of dye (g/100g of yarn)	Wave length (nm)	Dye absorption
45 min	1	450	46.36
	2	450	62.55
	3	450	47.50
	4	450	35.11
	5	450	22.71
	6	450	12.84

Optimization of concentration of mordants:

It can be seen from Table 7 that the per cent dye absorption was maximum (48.38%) at 10% of alum mordants, highest absorption (46.13%) showed in 5% concentration of copper sulphate and for ferrous sulphate mordant 3% concentration showed maximum (46.66%) dye absorption percentage. Hence, 10%, 5% and 3% concentration of mordants was selected as optimized concentration for alum, copper sulphate and ferrous sulphate, respectively.

Table 7 : Optimization of mordant concentration

Name of the mordant	Mordant concentration (g/100g of yarn)	Wave length (nm)	Dye absorption (%)
Alum	2	450	21.90
	4		23.16
	6		26.16
	8		37.95
	10		48.38
	12		29.15
Copper sulphate	1	450	20.47
	2		27.31
	3		31.07
	4		38.01
	5		46.13
	6		33.10
Ferrous sulphate	1	450	26.97
	2		36.65
	3		46.66
	4		30.50
	5		27.05
	6		24.96

Optimization of mordantime time:

From Table 8, it was evident that the highest absorbances (%) of dye were obtained in 30 min of mordanting time for each mordant. Therefore, 30 min of mordanting was considered as the optimum time for mordanting eri silk yarns.

Optimization of mordanting methods:

It was evident from the above table that in pre-mordanting method alum showed maximum (48.33%) absorption of dye by eri silk yarn. In simultaneous mordanting method ferrous sulphate showed the maximum (37.73%) absorption and in post mordanting method, copper sulphate showed the maximum (43.11%) absorption of dye by eri silk yarn.

From the observation, it might be inferred that the yarns mordanted with alum could be better dyed with pre-mordanting, ferrous sulphate with simultaneous mordanting and copper sulphate with post mordanting method.

Table 8 : Optimization of mordanting time

Mordants	Mordanting time (min)	Concentration of mordants (%)	Wave length (nm)	Dye absorption (%)
Alum	15	10%	450	53.47
	30			56.61
	45			54.91
	60			33.06
	75			31.81
Copper sulphate	15	5%	450	38.26
	30			43.51
	45			43.16
	60			33.01
	75			31.20
Ferrous sulphate	15	3%	450	47.16
	30			52.15
	45			51.51
	60			41.12
	75			35.16

The optimum dyeing conditions for annatto dye on eri silk were summarized in Table 10. All the mordants were suitable for application on eri silk. 10% alum, 5% copper sulphate and 3% ferrous sulphate had given different shades of orange colour on eri silk.

Colourfastness properties of dyed eri silk yarns:

From Table 11, it was interesting to note that colour fastness grades rated for all the tests were very good which indicates, that the eri silk yarns dyed with annatto were very much colourfast, irrespective of any mordants.

Effect of dyeing on physical properties of dyed eri silk yarn:

From Table 12 and Fig. 1, it was cleared that breaking loads and breaking strength of dyed yarns were decreased to certain extent in comparison to undyed yarns. On the other hand, elongation and stress of all the dyed samples had increased and maximum elongation (10.70%) and stress (17.06%) were observed in alum mordanted samples. Hence, it might be inferred that dyeing had little affects on the physical properties of eri silk.

Conclusion:

From the above study, it can be concluded that dye

Table 9 : Optimization of mordanting method

Methods of mordanting	Mordants	Mordant concentration (g/100g of yarn)	Wave length (nm)	Dye absorption
Pre-mordanting	Alum	10	450	48.33
	Copper sulphate	5	450	23.20
	Ferrous sulphate	3	450	26.37
Simultaneous	Alum	10	450	22.31
mordanting	Copper sulphate	5	450	21.41
	Ferrous sulphate	3	450	37.73
Post mordanting	Alum	10	450	38.10
	Copper sulphate	5	450	43.11
	Ferrous sulphate	3	450	39.53

Table 10 : Summary of the optimized dyeing conditions for dyeing eri silk with annatto dye

Yarn	Extraction medium	Extraction		Dyeing time (min)	Dye material conc. (g/100g)	Mordanting time (min)	Mordant conc. (%)			Mordanting method		
		Time (min)	Alkali conc. (%)				Alum	$CuSO_4$	Fe_2SO_4	Alum	$CuSO_4$	Fe_2SO_4
Eri silk	Alkali	60	1	45	2	30	10	5	3	Pre	Simultaneous	Post

Table 11 : Fastness properties of eri silk yarn dyed with annatto dyes under optimum condition of dyeing

Sample dyed with	Sun light	Fastness properties																
		Washing			Crocking				Perspiration						Pressing			
		CC	CS		Dry		Wet		Acidic						Dry		Wt	
			S	C	CC	CS	CC	CS	CC	CS		CC	CS		CC	CS	CC	CS
										C	S		C	S				
Annatto dye (without mordant)	4-5	5	4-5	4-5	5	5	5	4-5	5	5	5	5	4-5	4	5	5	5	5
Alum mordant	4-5	5	4-5	4-5	5	5	5	5	5	5	5	4-5	4	4	5	5	5	5
Copper sulphate mordant	4-5	5	4-5	4-5	5	4-5	5	4-5	5	5	5	4-5	4	4	5	4-5	5	4-5
Ferrous sulphate mordant	4	5	4-5	4-5	5	4-5	5	4	5	5	4-5	4-5	4-5	4	5	4-5	4-5	4

CC - Colour change CS - Colour strain
S - Silk C - Cotton

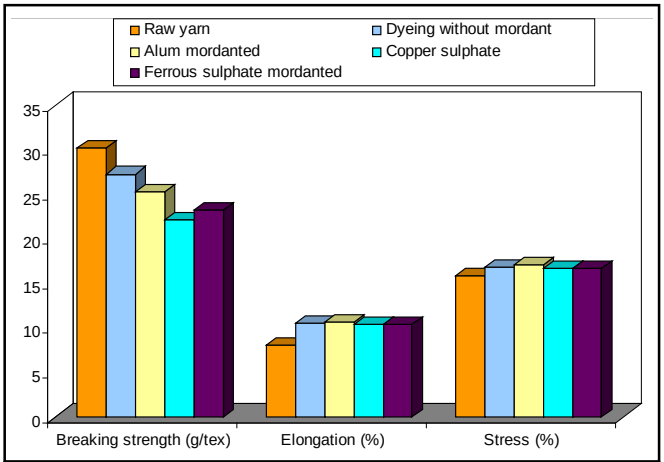

Fig. 1 : Effect of dyeing on physical properties of dye deri silk

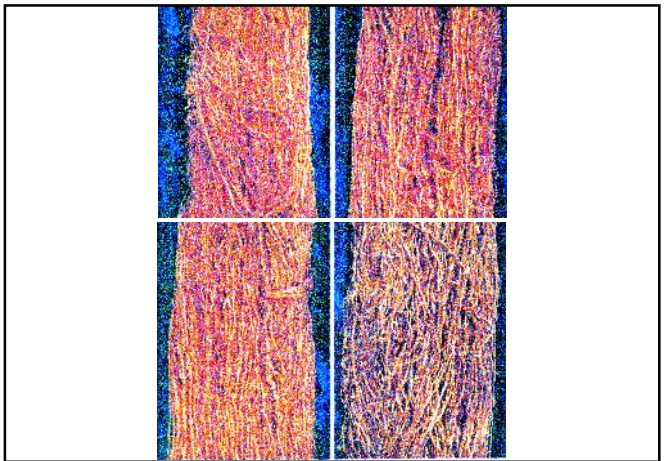

Plate 2 : Different shades obtained from Annatto dye

Table 12 : Effect of dyeing on physical properties of dyed eri silk

Samples	Breaking load (kg)	Breaking strength (g/tex)	Elongation (%)	Stress (%)
Raw yarn	1.77	30.20	8.05	15.90
Dyeing without mordant	1.60	27.30	10.46	16.85
Alum mordanted	1.48	25.26	10.70	17.06
Copper sulphate	1.30	22.18	10.40	16.73
Ferrous sulphate mordanted	1.36	23.21	10.33	16.73

extracted from annatto seed could be used effectively for dyeing eri silk yarn as well as fabric, which enhance the quality of eri silk. Thus, value addition to this "poor man's silk" by natural dyeing improves its aesthetic values and as such this silk can be utilized for making different attractive products so as to improve its marketability.

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