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VALUE ADDITION OF ERI SILK YARNS WITH *ALLAMANDA GRANDIFLORA* - A NATURAL COLOURANT

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Abstract:

Eri silk is one of the four varieties of silk mostly available in northeastern region of India. Recent trends in the world market of silk revealed that eri silk had tremendous potential as it was gifted with unique properties of protecting human body from some aliments as well as its use as fashionable dress and furnishing materials besides providing comfort of warmth. To suit the norms of the present national and international market, dyeing eri silk with natural dyes contributes to its value chain. The information on dyeing eri silk with natural dyes is very meager and requires to be established. New source such as ***Allamanda grandiflora*** was not used for dyeing textiles as per the available literature. Effort was made to standardise variables involved in extraction of the dyes and dyeing eri silk yarns. The standardisation procedures described under materials and methods were used and the evaluation was based on colour strength (K/S values) measured using colour flex spectrophotometer. Among the three mordanting methods, Pre mordanting method exhibited better results compare with simultaneous and post mordanted samples. Among the five mordants, alum and chitin mordanted samples exhibited good fastness followed by tartaric acid, ferrous sulphate and stannous chloride mordanted samples.

Key words: Eri silk, Natural dyes, *Allamanda grandiflora*, Mordants, Dyeing methods

1. Introduction

Eri and muga are the native silk varieties of Assam. In Arthasastra, Kautilya mentioned that several regions of ancient Kamarupa (Assam) had produced three varieties of silk fabrics, viz, Dakula, Khauma and Patrorna. Historians have identified Dakula as Muga

(*Antheraea assama*). Khauma as Eri (*Philosamia ricini*) and Patrorna as patar or patta (*Bombax mori*) (Baishya, 1998). William Roberson revealed that plantation of mulberry was taken up by only few families for their own use. For the members of the royal families and the grandees, every cultivator had a patch of land in his homestead, full of *Ricinus Communis* (castor) for rearing eri, the fabric of the common. The word eri is derived from the castor plant, in Sanskrit i.e. eranda. Castor leaf is the main food for the eri silk worms and so named as eri. It is also known as endi or errandi. Use of natural dyes for dyeing eri silk further promotes its eco friendliness of the textiles and create boom in exports. To suit the norms of the present national and international market, dyeing eri silk with natural dyes contributes to its value chain.

2. Materials and Methods

2.1. Selection of materials:

Allamanda grandiflora is a common rapid growing shrub for the tropics. It belongs to the family Apocynaceae. It is also a climber. The flowers are large and purple in colour, scented and bloom almost round the year. Mordants form the link between dye stuff and fiber that allows the dye with no affinity for the fiber to be fixed. Among all the mordants alum, stannous chloride, ferrous sulphate, tartaric acid and chitin were selected for the study as they were eco-friendly and did not cause any pollution problem. Sodium carbonate, alum, stannous chloride, ferrous sulphate, tartaric acid, chitin, sodium bi carbonate, sodium chloride, urea, acetic acid and non ionic detergent were selected for testing.

2.2. Selection of methods

Experimental research design was selected for the study as it involved experiments and laboratory tests.

2.2.1. Optimization of dye extraction:

Dye extraction methods such as aqueous, alkaline and acidic were experimented. Among the three extraction methods alkaline method was found suitable for extraction of the flowers of *Allamanda grandiflora* dye sources. Dyeing performance of various dyed samples such as depth of shade, tone etc were measured with colour flex spectrophotometer; computer colour matching system in terms of K/S values and CIELAB co- ordinates L*,a*,b* values with illuminant D 65 and 10 observer. Absorbency of different dye solutions were measured, using colour flex spectrophotometer. A wave length of 420 nm was found to give maximum optical density for *Allamanda grandiflora*. To optimize the dye extraction time the dyes were extracted and the optical density was noted after 30, 45 and 60 minutes of boiling. The time, at which optical density recorded maximum, was selected.

2.2.2. Optimization of mordanting procedures:

Silk yarns were subjected to different methods of mordanting viz, premordanting, simultaneous mordanting and post mordanting. To optimize the time for mordanting, pre mordanting method was selected. The samples were mordanted at 60^0C for 30, 45 and 60 minutes. The mordanted samples were then dyed and the colour strength of these samples were recorded. The optimize the mordant concentration suitable for the dye, four concentration levels were taken into consideration. For alum 5, 10, 15 and 20 per cent, for ferrous sulphate, stannous chloride and tartaric acid and chitin 1,2,3,4 and 5 per cent solutions were prepared separately.

2.2.3. Optimization of dyeing methods:

To determine the dye uptake of eri silk yarns, the colour strength (K/S) values were calculated using computer colour matching system i.e. Colour flex spectrophotometer**.** To optimize the concentration, separate containers with 200ml of water in each were taken to maintain material to liquor ratio of 1:50. The dye material from 1% to 10% was weighed and placed in the container and boiled at 95^0C for the optimized time. Yarn weighing 4g was then placed in the dye liquor and dyed for 30 to 45 min. For optimizing the dyeing time pre mordanted yarns were immersed in separate dye liquors and dyed for 30, 45, 60 minutes respectively. Finally the dyed samples were washed in a luke warm detergent solution to remove the loose dye on the yarn and then rinsed thoroughly in water and dried.

2.2.4. Evaluation of colour fastness tests:

Colour fastness refers to the resistance of the colour textiles to different agents such as washing, sunlight, perspiration and rubbing to which the yarn or fabric is exposed to during manufacturing and subsequent use. It is important because it directly affects the serviceability of fabrics.

3. Results and Discussion

3.1. Dye extraction parameter optimization:

The colour strength (K/S) value (taken at suitable wavelength 420 nm) was found to be maximum for alkaline method followed by acidic and aqueous methods indicating the suitability of alkaline method for *Allamanda grandiflora* dye. Maximum pigment strength was found at concentration of 8 g/ 100 ml of water. The appearance of the shade was also appealing at this concentration. Hence it was (K/S value λ =8.0086) selected. Dyeing time was found 60 minutes appropriate for extraction of dye from the flowers of *Allamanda grandiflora.*

3.2. Mordant optimization:

Based on K/S values, depth of shade and appearance, two concentrations per each mordant were selected. Incase of alum, 15 and 20 per cent concentrations, 2 and 3 per cent concentrations were selected for ferrous sulphate, stannous chloride and tartaric acid mordants. Similarly 4 and 5 concentrations were selected for chitin.

3.3. Shade variations in *Allamanda grandiflora* dyed eri silk after mordanting

Among the alum mordanted samples, pre mordanted samples showed green shades. The depth of shade increased with the increase in mordant concentration. Dull green shades were obtained by post mordanting. Dark shades of olive green were obtained with ferrous sulphate mordanted eri silk by varying the methods of mordanting, dyed samples produced better shades. Stannous chloride mordanted samples, pre mordanted samples had produced golden yellow shades. Very light, dull colours were produced by simultaneous and post mordanted methods. Chitin mordanted samples showed very bright and dark shades. Pre and post mordanted samples produced very good green shades. Slight differences in shades were observed in simultaneous mordanted samples.

3.4. Optimization of dyeing time for *Allamanda grandiflora*:

The data on the density of dye liquor by varying the time of extraction is presented in Table 1.

Table 1: Optimization of dye extraction time for *Allamanda grandiflora* dye

S. No	Extraction time (Minutes)	K/S values at λ(max)
1	45*	6.4928
2	60	5.9356
3	90	5.3498

*Indicates selected dyeing time

As per the data presented in Table 1 increase in dyeing time above 45 minutes for silk resulted in decreased absorption. Moreover, boiling silk for longer time decreases its luster also. Hence 45 minutes dyeing time was selected for the dye source *Allamanda grandiflora*.

3.5. Colour fastness tests of *Allamanda grandiflora*:

The eri silk samples dyed with the pigment of *Allamanda grandiflora* flowers were subjected to laboratory tests to assess their colour fastness to washing, crocking, perspiration and sunlight. The fastness grades of eri silk mordanted with alum are given in Table 2.

Table 2: Fastness grades of the A*llamanda grandiflora* dyed and alum mordanted eri silk yarn at optimum dyeing conditions.

Mordanting Method	Mordant concentration g/100g silk	K/S values at λ(max)	Fastness properties													
			Sun light	Washing			Dry crocking		Wet crocking		Acidic perspiration			Alkaline perspiration		
				CC	CS		CC	CS	CC	CS	CC	CS		CC	CS	
					C	S						C	S		C	S
Pre mordanting	15	4.3243	**7**	3	4.5	4.5	4	5	3.5	3.5	3.5	3	2.5	3	3.5	3
	20	8.4314	6	3.5	4	5	4.5	4	4.5	4	3.5	3.5	2.5	4	3.5	3
Simultaneous mordanting	15	2.8834	7	3.5	4	4.5	4	4.5	3.5	4.5	3	4	3.5	3	4.5	3.5
	20	3.6362	7	3	3.5	4.5	4.5	4.5	4.5	4.5	3.5	4	4	3	4	4
Post mordanting	15	2.1382	6	2.5	4	5	4.5	4.5	4.5	3	3	4	3.5	4.5	4.5	3.5
	20	3.6092	6	3	4	4.5	3	3	3.5	4	2.5	4	3.5	3	4	4

Note: CC- Colour change CS- colour stain

Very good to excellent fastness was observed for sun light. Simultaneous mordanted samples and 15 per cent alum pre mordanted samples showed excellent fastness and other samples exhibited outstanding fastness to sun light. The fastness grades of dyed eri silk samples exhibited fair to excellent fastness to serviceable conditions such as crocking and perspiration. The fastness to washing in terms of colour change was found to be fair to good and absolutely no colour change was observed in case of 20 per cent pre mordanted samples and 15 per cent simultaneous mordanted samples. No colour staining was found in washed samples. In dry crocking, 20 per cent post mordanted sample exhibited good fastness in terms of colour change and colour stain. In wet crocking, except 15 per cent post mordanted sample all exhibited good resistance in terms of colour change and colour stain. The fastness to acidic perspiration was fair to good with noticeable staining. The resistance to colour change

due to alkaline perspiration was found to be fair to good and resistance to colour staining was found good.

Table 3: Fastness grades of the A*llamanda grandiflora* dyed and ferrous sulphate mordanted eri silk yarn at optimum dyeing conditions.

The fastness grades of ferrous sulphate mordanted samples are given in Table 3.

Mordanting Method	Mordant concentration g/100g silk	K/S values at λ(max)	Fastness properties													
			Sun light	Washing			Dry crocking		Wet crocking		Acidic perspiration			Alkaline perspiration		
				CC	CS		CC	CS	CC	CS	CC	CS		CC	CS	
					C	S						C	S		C	S
Pre mordanting	2	6.8526	**7**	4	4.5	4.5	4.5	4.5	4.5	3.5	3.5	3.5	3	4	3.5	3
	3	7.4068	6	4	4.5	4.5	4.5	4	4	4	4	4	3.5	4.5	4	3.5
Simultaneous mordanting	2	3.9397	6	3.5	3.5	4	4.5	3.5	4	4	3	4	3.5	3.5	4.5	4
	3	3.0571	7	3.5	4.5	5	4	4	4	3.5	3.5	4	3.5	3.5	4.5	4
Post mordanting	2	3.9509	6	3	4.5	4.5	4.5	4	3.5	2.5	3.5	4	4	4	4.5	4.5
	3	2.2564	7	3.5	4	4.5	4	4.5	4	4.5	3.5	4	4	4.5	3	4.5

Note: CC- Colour change CS- colour stain

It was evident from the data that the fastness to various serviceable conditions such as washing, crocking and perspiration varied from very fair to excellent. Very good to excellent fastness was shown to sun light. The wash fastness of all samples was good in terms of colour change and colour stain, except 2 per cent post mordanted sample. The fastness for dry crocking and wet crocking was found good except in 2 per cent post mordanted sample. In case of acidic and alkaline perspiration good fastness was observed in all samples except in 3 per cent post mordanted sample.

Table 4: Fastness grades of the A*llamanda grandiflora* dyed and stannous chloride mordanted eri silk yarn at optimum dyeing conditions.

The fastness grades of stannous chloride mordanted samples are given in Table 4.

Mordanting Method	Mordant concentration g/100g silk	K/S values at λ(max)	Fastness properties													
			Sun light	Washing			Dry crocking		Wet crocking		Acidic perspiration			Alkaline perspiration		
				CC	CS		CC	CS	CC	CS	CC	CS		CC	CS	
					C	S						C	S		C	S
Pre mordanting	2	1.5989	**6**	4	5	4.5	4.5	4	4	4.5	3.5	4.5	3.5	3	4.5	4
	3	1.6672	6	3	5	5	4.5	5	4	4.5	3.5	4.5	3.5	3	4.5	3.5
Simultaneous mordanting	2	2.1002	8	4	4.5	5	3	4.5	2.5	4.5	3	4	3	2.5	4.5	3.5
	3	2.2175	7	3	5	5	4.5	4.5	4	4	3.5	4.5	4	3.5	4.5	3.5

Post mordanting	2	1.8758	7	3.5	4.5	4.5	4.5	4.5	4	4.5	4	5	3.5	3.5	4.5	3
	3	2.1103	7	3	4.5	5	4.5	4.5	4.5	4.5	3.5	4.5	3.5	3.5	4.5	3.5

Note: CC- Colour change CS- colour stain

Pre mordanted stannous chloride samples exhibited very good sun light fastness. Post mordanted samples showed excellent fastness to sun light. Outstanding fastness was found in simultaneous mordanted samples. The resistance to colour change due to washing was found to be good for lower concentrations and fair for higher concentrations of mordant. Pre mordanted samples exhibited excellent resistance to colour staining due to washing. In dry crocking, there was no colour change except for 2 per cent simultaneous mordanted sample. The colour change after wet crocking was slightly noticeable with meager staining and the fastness was rated as good except 2 per cent simultaneous mordanted sample. For acidic perspiration, good to excellent fastness was found with very slight staining. The fastness to alkaline perspiration was found to be fair for pre mordanted samples and 2 per cent simultaneous mordanted sample. All the post mordanted and 3 per cent simultaneous mordanted samples were found to possess good resistance to colour change. However, good resistance was observed for colour staining in all samples.

Table 5: Fastness grades of the *Allamanda grandiflora* dyed and tartaric acid mordanted eri silk yarn at optimum dyeing conditions

Mordanting Method	**Mordant concentration g/100g silk**	**K/S values at λ(max)**	**Fastness properties**													
			Sun light	**Washing**			**Dry crocking**		**Wet crocking**		**Acidic perspiration**			**Alkaline perspiration**		
				CC	**CS**		**CC**	**CS**	**CC**	**CS**	**CC**	**CS**		**CC**	**CS**	
					C	**S**						**C**	**S**		**C**	**S**
Pre mordanting	2	2.861	**6**	3.5	4	4.5	4	5	4.5	4.5	4	3	3	3	3	2.5
	3	2.5052	6	4	4.5	4.5	4.5	5	4	4.5	4.5	3	3	4.5	3	2.5
Simultaneous mordanting	2	2.1203	8	3	5	5	4.5	4	3.5	4	3.5	4	3.5	4	4	3.5
	3	2.481	8	3.5	4	4.5	5	4	5	4	3.5	4	3	4	4	4
Post mordanting	2	1.6117	6	3.5	5	5	5	5	4.5	4	3.5	4	3.5	3.5	4	3.5
	3	2.4513	8	3	4.5	4.5	4.5	4.5	4.5	4	3.5	4	2.5	3.5	4	3.5

Note: CC- Colour change CS- colour stain

The fastness grades of tartaric acid mordanted samples are given in Table 5. Out standing sun light fastness was exhibited by simultaneous mordanted samples and 3 per cent post mordanted sample. Others showed very good sun light fastness. The fastness to washing was fair to good without any staining in case of simultaneous mordanted and post mordanted samples. In pre mordanted samples, the fastness was good to washing. In case of dry crocking, simultaneous mordanted samples exhibited good to excellent resistance with noticeable staining. The fastness to wet crocking was found to be very good resistance to colour change and colour staining. Three per cent simultaneous mordanted sample exhibited

excellent resistance to colour change. For acidic and alkaline perspiration, the samples exhibited good resistance with noticeable staining. Pre mordanted samples showed fair resistance to colour staining.

Table 6: Fastness grades of the A*llamanda grandiflora* dyed and chitin mordanted eri silk yarn at optimum dyeing conditions

Mordanting Method	Mordant concentration g/100g silk	K/S values at λ(max)	Fastness properties													
			Sun light	Washing			Dry crocking		Wet crocking		Acidic perspiration			Alkaline perspiration		
				CC	CS		CC	CS	CC	CS	CC	CS		CC	CS	
					C	S						C	S		C	S
Pre Mordanting	4	5.5067	**7**	3.5	4.5	5	3.5	4.5	3	3.5	3	3.5	2.5	3	4	2.5
	5	10.9617	6	3	5	4.5	4	4.5	3.5	3.5	3.5	3.5	4	3.5	4	3
Simultaneous Mordanting	4	8.0592	7	3.5	4	4.5	5	3.5	4.5	3.5	2.5	3	2.5	2.5	4	3
	5	8.2444	6	2.5	5	4.5	5	4.5	5	3	3	3.5	3	2.5	3.5	3.5
Post Mordanting	4	8.4478	7	2.5	4.5	4.5	4.5	5	3.5	3.5	3.5	4	2.5	3	4	3
	5	6.1219	7	2.5	5	4.5	4.5	4.5	3	4	3.5	3.5	3	3	4	3

Note: CC- Colour change CS- colour stain

The fastness grades of chitin mordanted samples are given in Table 6. Except 5 per cent pre and simultaneous mordanted samples, all mordanted samples showed excellent resistance to sun light. The resistance to colour change due to washing was fair to very fair. However, the resistance to staining was found to be very good to excellent. In case of dry crocking, the simultaneous mordanted samples exhibited excellent resistance followed by pre mordanted and post mordanted samples which exhibited good fastness. Very slight colour staining was noticed in all samples to dry crocking. The fastness to wet crocking was found to be fair to excellent resistance with slight staining. For acidic perspiration, pre mordanted and post mordanted samples exhibited good resistance. Simultaneous mordanted samples were found to possess fair resistance to colour change. Except 4 per cent simultaneous mordanted sample, all other mordanted samples exhibited good resistance to colour staining. The resistance to alkaline perspiration was fair to good. Except 5 per cent pre mordanted sample, all samples exhibited fair resistance to colour change. Very slight colour staining was found in alkaline perspiration on cotton specimen. However, noticeable staining was found on silk composite fabric.

Conclusions:

- The dyed shades of *Allamanda grandiflora* dye were subjected to colour fastness tests for sun light, washing, crocking, and perspiration.
- In case of sun light, very good to excellent fastness was found in all mordanted samples. Lower concentrations of alum, ferrous sulphate and chitin exhibited excellent fastness.
- All five mordanted eri silk samples showed very fair to excellent fastness to washing. After washing the colour was deepened. Staining was not found on both cotton and silk

samples. Increase in depth of shade is an added advantage to the consumer as the shades usually fade on washing.

- The rating of dry and wet crocking on cotton and silk samples showed very fair to excellent fastness. Slight colour staining was observed in both the samples.
- The findings of colour fastness to acidic and alkaline perspiration showed that all samples had very fair to excellent fastness, with slight colour staining. The colour was depended on exposure to alkaline perspiration. This ensures the sustainability of colour on yarn after subjecting them to several washes containing alkaline soaps.
- Among the three mordanting methods, Pre mordanting method exhibited better results compare with simultaneous and post mordanted samples.
- Among the five mordants, alum and chitin mordanted samples exhibited good fastness followed by tartaric acid, ferrous sulphate and stannous chloride mordanted samples.

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