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Evaluation of physical properties of Eri Block print fabric with Annatto (*Bixa orellana*) dye

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Abstract
This paper concern with the use of annatto seed as a new dye source for block print on eri silk fabric with alum and ferrous sulphate as mordant. The annatto seeds were collected and extracted by using alkaline medium. Different printing variables such as dye material concentration; dye extraction ph, dye paste and thicker ratio, viscosity of the dye liquor, fixer concentration, mordant concentration were optimized for making the printing paste. Experiment was done to evaluate the colour fastness and physical properties of the eri block printed fabric. The result reveals that annatto can be successfully used for printing of eri fabric and has good colour fastness properties. All printed samples with mordant improve physical properties of annatto dye on eri silk fabric.

Keywords: dye, block print, colour fastness properties, physical properties, optimization, mordant

Introduction
Dyeing and printing of textiles with different natural dye stuffs had been used from time immemorial. Printing is an technique where design and colour forms an artistic expression to embellish beauty (Paul *et al*. 2008) [7]. Colorants obtained from natural sources such as plants, insects/animals and microbes have been scrutinized in recent past for their use in different kinds of applications. Research into new natural dyes sources along with eco-friendly, robust and cost-effective technologies for their processing and application have greatly aided in widening the scope of natural dyes in various traditional and advanced application disciplines. Till 18th century India was the largest exporter of textiles, obviously hand woven and processing with natural dyes in the world. (Paul, 2011) [8] Natural dyes are good biodegradable and are non-toxic, non –allergic to skin non carcinogenic, easily available and renewable. For coloration of fabrics many chemicals are used in textile industry. However, environmental issues in the production and application of synthetic dyes once again revived consumer interest in natural dyes. The use of natural dyes is growing considerably because of the quality of the natural dyestuffs obtained, the environmental compatibility and the subsequent minimizing the processing cost (M.B Kasiri and S. Safapour 2015) [5]. The main aim of this paper is therefore to study the application of natural dyes (annatto dye) on textiles using block printing method. To address the main objective the following specific objectives were included; such as identifying the source and types of natural dyes, understanding the application of natural dyes and identifying the possible extraction and study of colour fastness and physical properties of natural dyes.

Methodology
In the study, the eri silk fabric weighted 43g/inch, thickness 43 mm, tearing strength 76.8 g and fabric count of 34 warp (thread/inch and weft 21 thread/inch) with pale yellow colour is selected for the study. Prior to the printing process the fabrics were degummed to remove the gum that served as protective layer, left on the silk until it is ready to dye (kiron, 2012). For printing annatto (*Bixa orellana*) has been selected as dye source. At first the annatto seeds were boiled in aqueous solution for 15 minutes at 60 C temperatures, and then dried in sunlight to dry the seeds for converting them to powdered form.

Optimization of different values
The dye particle size were evaluated using a siever containing 9 different sizes of about 1000um-45um and allow separation in a sieve shaker machine for 30 minutes.

$$\text{Percentile of particle (\%)} = \frac{\text{Weight of separated particle of particle size}}{\text{Total wt of sample}} \times 100$$

Annatto dye were extracted in alkaline medium by adding sodium carbonate to the extracted bath by following the procedure adopted by Bhuyan ang gogoi, (2016) [12].

Different ph values such as 6,7,8,9, and 10 with mean score of the dye paste were calculated and based on highest mean score ph value was selected. The main component of printing paste is thickener and the dye paste thickener ratio were optimized by using different ratio such as 1:1, 1:2, 1:3 1:4 and 1:5 dye liquor and thickener ratio. Based on wash fastness and clearity of printing design of the paste and thickener ratio was optimized.

Viscosity of annatto was optimized by using viscometer and optimized by taking 1 gram of each ratio ie 1; 11; 21; 31; 4 and 1; 5. Than weighted dye solution is than diluted in 100ml of distill water. Than the viscosity is calculated by passing the solution through viscometer and the count of the time is noted. The fixer concentration were optimized by using the different volume of fixer concentration from 0.5, 1, 1.5, 2 and 2.5 were mixed in the dye paste and printing was done to fix the fixer concentration. The role of fixer is to improve the fastness of prints Use of different types of fixing agents also increased the fastness ratings (Uddin M.G and Islam .M, (2015) [2].

Five different volume of fixture concentration ie 0.5, 1, 1. 5, 2 and 2.5 percent were mixed with the dye paste and printing was done on eri fabric samples. The optimized fixer concentration was determined on washing fastness of the printed samples. For optimization of mordant concentration, different concentration of alum such as 2gm, 4 gm, 6gm, 8gm and 10 gm and ferrous sulphate from 0.5-3 gm was taken and mixed with the printing paste. Printing was done with different mordant concentrations. Printing ample were dried and evaluated for wash fastness. Based on fastness and clearity of design mordant concentration was optimized.

For application of printing paste on eri silk fabric, the dye liquor was first added with the thickner and stirred for five minutes. Than the fixer was added in the dye paste and again stirred for five minutes and the required amount of alum anf ferrous sulphate of mordant concentration was added separately in the dye paste and stirred vigorously for 40 minutes for uniform printing paste. Eri silk fabric was ironed and fixed on the printing table with pins. Block was put on in printing paste which is poured on pads and was made fix on the fabric by giving prominent press and dried in air. Printing sample were stemed for a period of 1 hour at a boiling temperature and than dried in air.

Testing of colourfastness test

Prior to testing the speciments were conditioned to moisture equilibrium and tested in standard atmospheric condition of 65+2 percent relative humidity as per IS method (1971) and preparation test was done as per IS method (1972).

Colourfastness properties like colourfastness to washing, was tested by using sasmira laundro meter. The washfastness rating was rated by using grey scale rating from1-6. Colourfastness to pressing (dry and wet) was done by subjecting to hot iron in both dry and wet condition. It was compared with grey scale to see the both colour change and colour stain. Colourfastness to sunlight was seen by exposing the fabric to the direct effect of sunlight simultaneously from 9 am to 5 pm. Colourfastness to perspiration was assessed by using ASTM (1968) methods.

Testing of physical properties

Physical properties like Fabric weight is determined by using electronic balancr (ASTM 1968). Tearing test were performed according to IS (7702-1975) using elementary tearing testercount was determined by using peak glass and thickness is determined by using ASTM 1968 method.

Findings and Discussion

The present investigation was carried out to study “Block printing on cotton and silk fabric with annatto dye”. The findings of the study were showed in table.1 the size particle distribution of annatto dye from Bixa seed in micron along with the weight percentage and the percentage of its cumulative frequency. To optimize the dye liquor concentration, optical density of the dye liquor was also evaluated through spectrophotometer and the reading is shown in Table. 2. It was evident from the Table.2 the optical density of extracted dye liquor was found maximum (0.292) in 15ml of concentration. As the concentration of dye increases the optical value of extracted dye decreases. According to Ghanipur *et al*, 2014 adding dye structure lead to decrease band gap and increase in the absorption of samples is due to decrease the real part of the refractive index and increasing in imaginary parts. Hence 15ml of dye concentration was selected as optimized dye concentration for extracting the dye for making the paste.

From the Table 3 it can clearly be noted that with five different pH value taken, the weighted mean score of the dye material concentration increased with the increasing pH up to 8 and then decreased as further rise in pH, made the dye and fabric more ionic which repelled each other and cause lesser dye print on higher ph. Similar result was found by Ali *et al*. (2008). Hence the result indicated that slightly alkaline pH was best for annatto dye and pH 8 was selected as optimum extraction pH value for extracting annatto for making printing paste.

To optimize the dye liquor and thickener ratio five different ratios such as 1:1, 1:2, 1:3, 1:4 and 1:5 were taken and printing was done on eri silk fabric and evaluated for the washing fastness, sharpness and clearity of design. From the Table 4. It was cleared that the dye liquor and thickener ratio at 1:2 was found very good in all the respects like sharpness of design, clearity of design and as well as washing fastness.

From Table 5 it was evident that the relative viscosity (gm) of 1:2 dye liquor and thickener ratio was found maximum (1.27gm) as compared to other four dye liquor and thickener ratio which is 1.096 for the ratio 1:3, 1:4 and 1:5 respectively.

Hence, based on sharpness, clearly and washing fastness of printed samples as well as relative viscosity of dye liquor and thickener ratio, 1:2 ratio was elected as optimized ratio for making the printing paste

It was observed from the Table 6 above that washing fastness increased with the increasing fixer concentration up to 1.5 percent. In this percent of fixer washing fastness grades were found excellent comparatively to other concentrations such as 0.5, 1.0, 2.0 and 2.5. Further increase in fixer concentration washing fastness was less comparatively to fixer concentration 1.5 and clearity as well as sharapness of design was found good and very good in the concentration 1.5. Hence, for the study 1.5 percent concentration was selected as optimum fixer concentration. According to Grover. N and Patni V. 2011 mordant and their combination can be successfully used for dyeing. Table 6 showed that in order to optimize the mordant concentration for eri fabric five different concentration of alum (2-22gm) and ferrous sulphate (0.5-3.0gm) were taken with printing paste and printed fabric were evaluated for the washing fastness. On the other hand in concentration of ferrous sulphate, eri fabric showed 2-3 grades in 2.5% both in colour change and colour stain. Hence, 18% alum mordant concentration and 2.5% ferrous sulphate mordand concentration were selected as optimum mordant concentration for eri fabric.

The general appearance of untreated (UT) and treated Eri (AT1) And treated eri (AT2) with mordant alum and ferrous sulphate as shown in graph 1 were rated according to respondent view and found that most of the responded found AT2 sample most preferred . As per graph 2 most responded respond ATI and AT2 as fine printd sample. According to visual response of the respondent colour brilliancy was found in AT2 followed by AT1 as shown in graph 3.

Graph 4 showed that most respondent found At2 and UT sample as mostly clearty of design. Most responded found AT1 sample as nicely penetration of dyes followed by AT2 as stated in graph 5. Colourfastness properties like colourfastness to washing, light, crocking and pressing of dyes shown in table were found excellent in AT1 and AT2 samples. It was concluded that according to visual and grey scale test it was found that mordanted samples shows better performance than untreated sample. Similar result was found by V. Narayana swamy (2013) that dyed with mordant fabrics showed acceptable fastness properties.

It was found from the table 9 that the samples had increased warp and weft direction per inch compared with the original. The fabric count in warp direction was greater than the weft direction. In case of untreated sample the increased count was observed by 11.76% in warp direction and 19.4% in weft direction. The count had increased by 2.63 and 4% in warp and weft direction respectively. It was observed that the fabric count of AT2sample increased in both sides. From the observation it was clear that the fabric has more or less effect on count after printing. This indicate that all the fabric had shrunk and the rate of shrinkage was not equal for all samples. Shrinkage depends on the type of fabric constructon (lyle, 1979). Gogoi (1988) stated that after dyeing or printing the fabric count had increased in both direction.

From the table it was seen that the weight of treated and untreated eri silk fabric increased. The increased weight of untreated eri fabric sample was found 1.88 percent and the increased weight of all the treated samples are 1.86 each. The increased in weight was due to various mordants or due to the absorption of dyes in the fabric. The might be increased in number of size of interstice which may lead to change in weight per unit area.

The thickness of the samples are recorded and presented in table 11. It was revealed from the table that the mulberry printed samples have increased in thickness. The thickness of treated eri printed samples was maximum for AT2 by 6.66 percent followed by AT1 5.55 percent. The increased in thickness was due to consolidation taking place during printing which increased the count and may be due to absorption of dyes.

The tearing strength of the printed fabric is increased in warp and weft direction. Tearing strength in warp direction was greater than that of warp direction.

Table 1: Particle dye distribution of annatto dye from Bixa seed

Sl. No.	Size in μm (micro)	Wt of sample (in gm)	Wt. %	Cumulative %
1.	+1000	14.72	14.69	100.00
2.	+500-1000	27.24	27.18	85.31
3.	+300-500	8.33	8.31	58.13
4.	+150-300	36.57	36.49	49.82
5.	+106-150	9.86	9.84	13.33
6.	+90-106	1.25	0.80	3.49
7.	+75-90	0.95	1.25	2.69
8.	+35-75	0.51	0.94	1.44
9.	+45-53	nil	0.50	0.50
10.	-45			

Table 2: Optimization of dye material concentration

Dye material liquor (ml)	Wavelength (Nm)	Optical density Value
5ml	585	0.246
10ml	585	0.255
15ml	585	0.292
20ml	585	0.264
25ml	585	0.282

Table 3: Optimization of dye extraction pH

Sl. No.	Dye material concentration (%)	Dye extraction pH	Weighted mean score (based on total dye paste)
1.	15	6	1.0
2.	15	7	1.2
3.	15	8	1.4
4.	15	9	1.3
5.	15	10	1.3

*On the basis of total dye paste

Table 4: Optimization of dye liquor and thickener ration

Sl. No.	Fixer concentration (%)	Washing fastness Grades		Clearity of design	Sharpness of Design
		CC	CS		
1.	1:1	2	2-3	Good	Very Good
2.	1:2	2-3	2-3	Very Good	Very Good
3.	1:3	2-3	2	Good	Good
4.	1:4	2-3	2	Good	Good
5.	1:5	2-3	2	Good	Good

CC: Colour changes, CS: Colour stain

Table 5: Viscosity of annatto dye

Dye paste Thicker Ratio	Weight of dye solution (gm)	Weight of distilled water (gm)	Density of dye (gm)	Time flow of dye solution (in sec)	Time flow of distilled water (in sec)	Relative viscosity (gm)
1:1	4.84	4.88	1.030	6.6	5.5	1.20
1:2	4.96	4.88	1.016	6.2	5.5	1.27
1:3	4.94	4.88	1.012	6.0	5.5	1.096
1:4	4.80	4.88	0.980	6.0	5.5	1.096
1:5	4.90	4.88	1.004	6.0	5.5	1.096

*On the basis of the total dye paste

Table 6: Optimization of dye liquor and thickener ratio

Sl. No.	Fixer concentration (%)	Washing fastness Grades		Clearity of design	Sharpness of Design
		CC	CS		
1.	Without fixer	2-3	3	Good	Very Good
2.	0.5	2-3	2-3	Good	Very Good
3.	1.0	3	2-3	Good	Very Good
4.	1.5	2-3	3	Good	Very Good
5.	2.0	3	2-3	Good	Very Good
6.	2.5	3	2-3	Good	Very Good

Graph 7: Optimization of mordant concentration for eri fabric

Sl. No.	Mordant	Mordant concentration (%)	Washing fastness grades	
			CC	CS
1.	Without mordant	-	2	3
2.	Alum	2	3	2-3
		6	3	3
		10	3	2-3
		14	2-3	3
		18	2-3	2-3
		22		
3.	Ferrous sulphate	0.5	2	2-3
		1.0	2-3	2
		1.5	2	2-3
		2.0	2	2-3
		2.5	2-3	2-3
		3	2-3	3

Note: CC: Colour changes, CS: Colour stain

Table 8: Rating effect of printed samples on washing fastness (in percentage)

Sample	UT	AT1	AT2
Preference	**Ea**	**Eb**	**Ec**
Negligible change	65	59	92
Slight change	25	23	4
Noticeable change	10	15	2
Considerable change	-	3	2
Much change	-	-	-

Table 9

Direction of the fabric	O	UT	AT1	AT2
	E	**Ea**	**Eb**	**Ec**
WARP	34	38	39	41
% change in warp		+11.76	+2.63	+7.89
WEFT	21	25	26	28
% change in weft		+19.4	+4.00	+12.00

Table 10

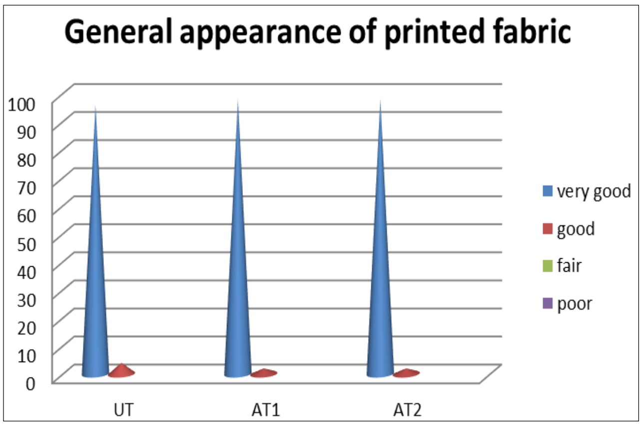
Aspect	O	UT	AT1	AT2
	E	**Ea**	**1.09**	**1.09**
Weight	1.06	1.07	39	41
% change in warp		+1.88	+1.86	+1.86

Table 11

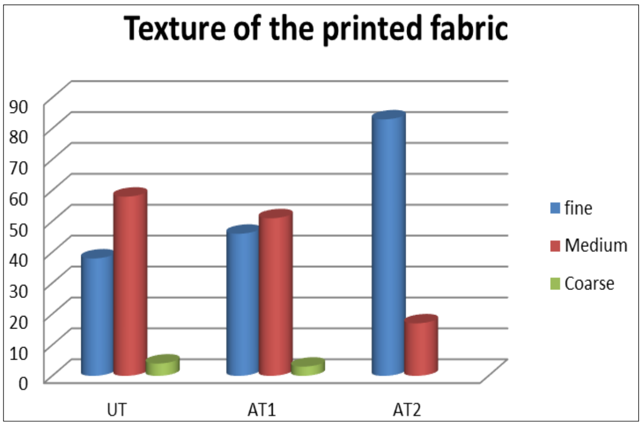
Aspect	O E	UT Ea	AT1 1.09	AT2 1.09
Thickness	43	45	47.5	48
% change in warp		+4.65	+5.55	+6.66

Table 12: Tearing strength are recorded as follows.

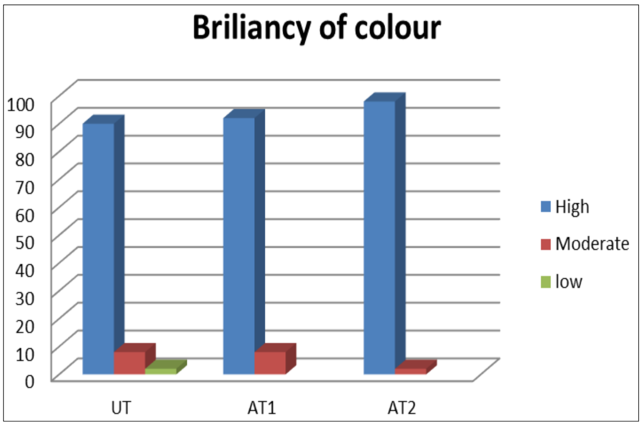
Direction of the fabric	O E	UT Ea	AT1 Eb	AT2 Ec
WARP	76.8	77.6	78.2	78.9
% change in warp		+1.04	+1.77	+1.78
WEFT	80.3	80.9	49.4	50.2
% change in weft		+0.74	+0.86	+1.85



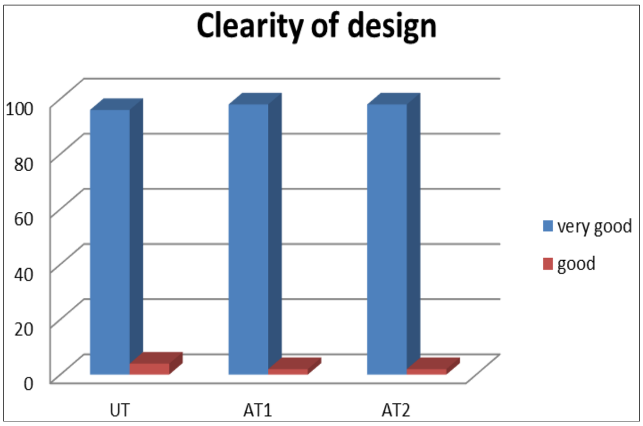
Graph 1



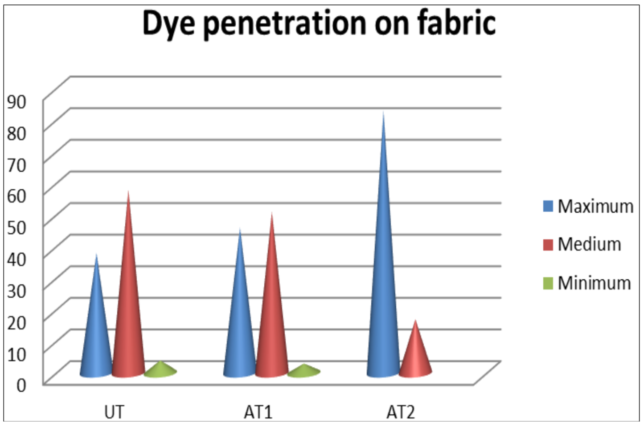
Graph 2



Graph 3



Graph 4



Graph 5

Summary and conclusion

The plant colorant annatto was investigated to determine its potential use as a natural dye for conventional and novel textile applications. Alum was selected as a mordant. Different techniques of mordanting and a broad set of variations in the dyeing recipes were applied to achieve optimization and an improvement in colour fastness properties. It was observed from the study that colourfastness properties of mordanted samples showed more colourfastness than unmordanted samples. From the experiment it has been recorded that the eri printed samples gradually shrunk as a result of which the fabric count of the fabric was increased. This indicated that the eri printed fabric had shrunk and the rate of shrinkage was not equal for all the samples.

Fabric weight and fabric thickness of eri printed samples was increased due to the incrwases in fabric count. All the eri printed samples had been recorded to have an increases in tearing stenght due to the increased in the size of dye molecule after using mordant.

It could be concluded that annatto (*Bixa orellana*) dye can be successfully used for block printing on eri fabric with different mordants. Regarding in physical properties of mordanted annatto printed mulberry samples has more or less change in fabric count, thickness, weight and tearing stenght. Thus it was precipitated from the study that all prined samples with mordant improves physical properties of annatto dye on eri silk fabric. Annatto is locally available, biodegradable which can be utilized for printing of appareals in the textile industry and the use of annatto act as antioxidant.

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