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Optimization and application of "*Rubia cordifolia*" in Eri silk with alum and myrobalan mordant

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Abstract
The *Rubia cordifolia* L. species, sometimes referred to as Majith and Indian Madder, is one of the most prominent and useful species of the genus Rubia on the Indian subcontinent. *Rubia cordifolia* was selected as natural dye for the study. Eri Silk fabrics and Natural mordant myrobalan and Metallic mordant alum was selected for the study. The non-mordanted fabric gave the greater depth of colour and myrobalan mordanted fabric had highest 16.26 K/S value. According to the FTIR spectra, all of the dyed samples showed different peaks from the raw fabric. It was also noted that the peaks of the dyed samples varied somewhat because of various mordants. Compared to alum-mordanted fabric, myrobalan-mordanted fabric has a smooth, uniform surface.

Keywords: Alum, color palette, Eri silk, manjistha, mordants, natural dye

Introduction
There are countless shades of colour in the world, and each one is important to human existence. It makes an enormous impact in people's life by bringing glamour, glitz, joy, happiness, and beauty while expressing emotions and thoughts. Natural colours are produced by various plants, vegetables, animals, or insects which are eco-friendly and suitable for use in the colouring of textile products (Chanu, 2022) [2].
Madder is known to be the queen of the reds which is an oldest used dyes (Jahan and Datta, 2015) [3]. Red colours can be created with madder or manjistha. This originates from the plant's root and is similar to turmeric, which obtains its colour by washing, drying in the sun, and cooking in water. The main fabrics on which madder root pigments are applied are cotton, silk, and wool. Alizarin is a colouring agent found in madder (Khattak *et al.*, 2019) [5].
Mordants and additives are needed because natural dyes often produce poor colour and have insufficient fastness. In addition to providing shade, it also has antibacterial, fungal, and UV protective properties. Waste water from the manufacturing of natural dyes can be utilised as a fertiliser in agriculture. Natural colours offer incredibly uncommon, calming, and delicate colours in contrast to synthetic dyes (Karthikeyan, 2020) [4].
Silk clothing that was dyed in dazzling hues was seen as a sign of royalty in the community. Natural dyeing of silk produces beautiful glossy tones that add value to the fabric. Even after a long time, they are still incredibly beautiful and charming (Boruah *et al.*, 2019) [1]. The objective of the study was to optimize the dyeing parameter and application of Manjistha dye on Eri silk fabric.

Materials and Methods
Natural Dye: Indian madder, or *Rubia cordifolia*, was chosen as the natural dye and was bought from Guwahati, Assam.

Eri silk fabric: Eri Silk fabric was chosen and collected from Department of Textiles and Apparel Designing, AAU, Jorhat-13.

Mordants
Natural mordant: Myrobalan are washed with water, sun dried, then grounded into powder form and kept it in tight container.

Metallic mordant: Metallic mordant alum was chosen for the study.

Chemicals and Reagents
Chemicals and reagents used are Acetic Acid, Aluminum potassium sulphate (metallic salt), alum, Disodium hydrogen phosphate anhyd, Hydrochloric acid, Histidine monohydrochloride, Hydrogen Peroxide, Lactic acid, USP 85%, Sodium Chloride, Sodium Carbonate, and Surf.

Preparation of fabric for dyeing
For degumming, required quantity of washing soda along with Eri silk fabric were added to water and started heating. After 30 minutes the fabric were taken out and washed properly and air dried. For Bleaching, Eri silk fabric was added to water along with1% hydrogen peroxide. Sodium silicate was added to the bleaching bath as a stabilizing agent in the middle of the process. Fabric were taken out after 30 minutes and washed properly and air dried.

Mordanting and Dyeing
Pre-mordanting method
The fabric was mordanted first in the mordanting solution and then dyed for required period of time.

Simultaneous method
In this method, the mordant and dye were added in the dye bath simultaneously. The fabric was dyed for 15 minutes and then mordants were added to the dye bath and mixed properly. The fabric was again dyed in the solution for required period of time.

Post mordanting method
The Eri silk fabric was dyed first in the dye solution and then placed in the mordanting solution for required time period.

Dyeing of Eris ilk fabric
The Eri silk fabric was dyed for 45 minutes at material to liquor ratio 1:30. The optical density of the dye liquor before and after dyeing was recorded. The percentage of dye absorption (%) of the fabric was estimated by using the following formula:

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

Evaluation of Dyed samples
Colour strength and colour co-ordinates
By measuring surface reflectance of the samples using a Computer Colour Matching System spectrophotometer (CCM), using the Kubelka Munk equation, the K/S value of the yarn was determined. The CIE Lab colour co-ordinate value of all the dyed samples were determined by Brightness, Opacity and Colour Tester (Model no UEC-1080).

FTIR Analysis
FTIR was done to indicate the presence of chemical bonds and functional groups for control and dyed silk fabric using a double-beam Fourier Transform Infrared Spectroscopy (FTIR) spectrophotometer.

SEM Analysis
To assess the surface morphology of the samples using field emission Scanning Electron Microscopy (SEM) (make-Carl Zeiss), model-Sigma, Scanning electron microscopy (SEM) was done for the fabric.

Results and Discussion
The absorbance pattern of Manjistha dye showed the highest absorbance at 430 nm and considered as optimum wave length. The Eri silk fabric was dyed with 2% Manjistha dye at 75 minutes of time. The fabric was pre mordanted with 2% alum mordantat 30 minutes of mordanting time and the fabric was post mordanted with6% myrobalan mordant at 60 minutes of time period.

Color co-ordinates value of dyed fabric

Table 1: CIE Lab values

L*	a*	b*
Non-mordanted	31.24	
45.21	21.48	
Alum mordanted	33.79	
45.89	33.14	
Myrobalanmordanted	15.13	
56.28	35.95	

Where, L* indicates (depth of colour) lower value of L* indicate higher depth of colour, a* indicates redness (positive a*) and greenness (negative a*) and b* indicates yellowness (positive b*) and blueness (negative b*).
Table 1 indicated that myrobalan- mordanted sample showed highest L* value 56.28and showed lowest depth of colour. The lowest L* value 45.21 showed by non-mordanted fabric. Therefore, non-mordanted fabric gave the greater depth of colour. The redness and yellowness present in the dyed fabric indicated the positive values of a* and b*.

Colour Strength (K/S) values of Manjistha dyed fabric

Table 2: Colour Strength (K/S) values of Manjistha dyed fabric

Dyed fabric		Colour Strength (K/S value)
Non-mordanted	6.14	
Alum mordanted	9.66	
MyrobalanmordantedEri		16.26

Table 2 showed that myrobalan mordanted fabric had highest 16.26K/S value and non-mordanted fabric showed lowest 6.14 K/S value. This might be due to the alkaline natureof alum which removed the colour and yielded a lighter shade with natural dye (Vankar, 2009).

Fourier Transform Infrared Spectroscopy (FTIR) analysis of control and dyed fabric
Fig. 1, 2, 3 and4 showed the spectrum of the raw, non-mordanted and mordanted fabric. The presence of functional groups are indicated by the peaks of all the samples. Peaks between 1625-1650 cm-¹ indicated the presence of alpha beta unsaturated carboxyl group which has a characteristics of yellow flavonoid pigment (Nissankararao, 2011) [8]. Stretches band around 1610-1570cm-¹ indicates the presence of aromatic ring and stretches between 3030-3010cm-¹ represents C-H bond which usually considered as band for flavonoid pigments (Aksoz and Ertan, 2012) [7]. The peaks between 1610-1570 cm-¹ and 3030-3010cm-¹ in all the figures determined the presence of flavonoid in the dye pigment. The spectrum revealed that all the dyed samples showed different peak from the raw fabric and also observed a slightly variation in the peak among the dyed samples due to different mordants.

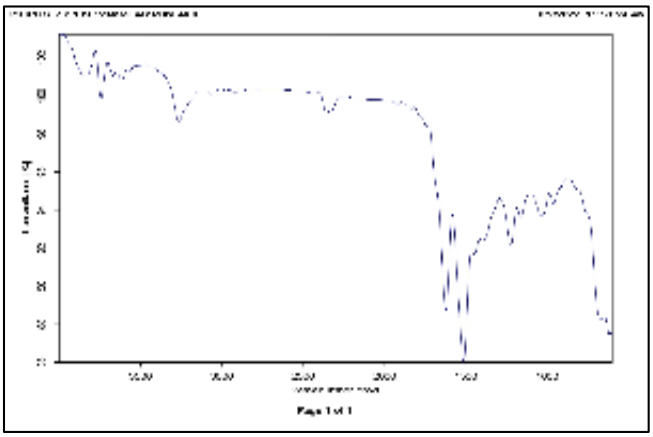


Fig 1: FTIR spectrum of raw Eri silk fabric

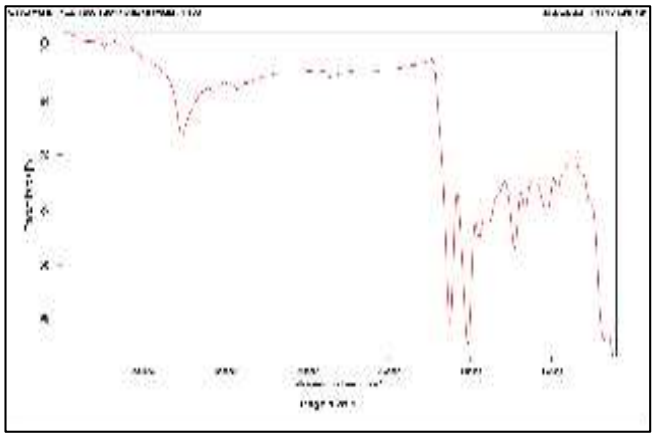


Fig 2: FTIR spectrum of non-mordanted Eri silk fabric

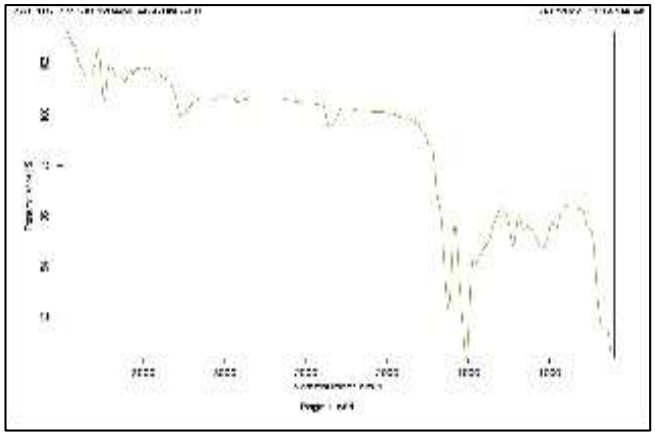


Fig 3: FTIR spectrum of alum mordanted Eri silk fabric

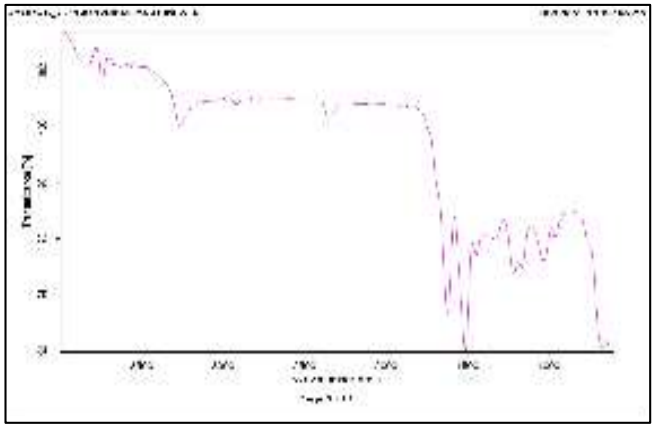
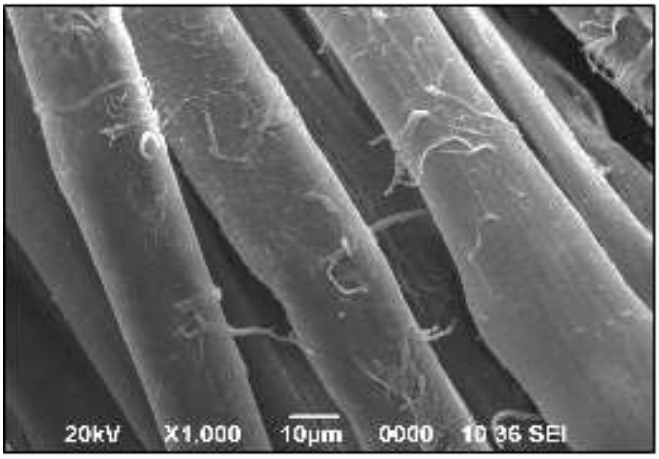


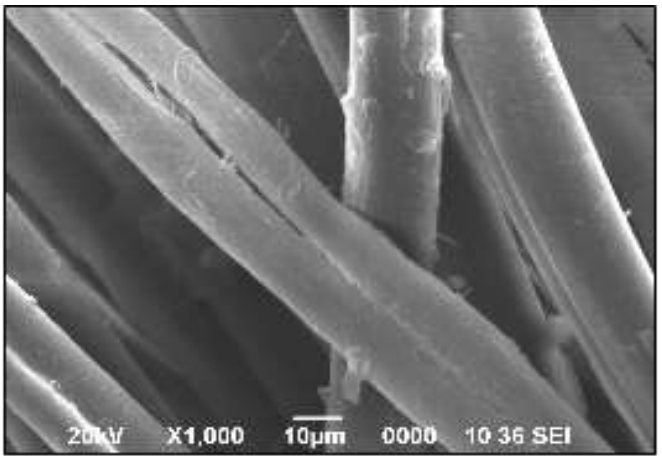
Fig 4: FTIR spectrum of myrobalanmordantedEri silk fabric

Scanning Electron Microscopy (SEM) analysis of control and dyed silk fabric

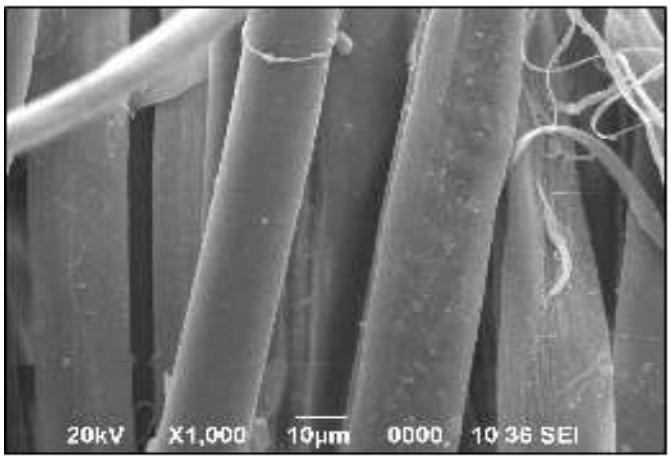
The SEM images of raw Eri silk fabric (a) showed rough and wrinkles surface may due to the presence of sericin and other gummy substances present in silk fibre. Non-mordanted Eri silk fabric (b) shows the inner structure of dye entrapped surface which was less rough and cracks compared to raw eri silk fabric. The alum mordanted sample (c) and myrobalanmordantedEri sample (d) were showing the relatively smooth surface of the dyed silk fabric. From the SEM image, smooth and uniform surface was observed in case of myrobalanmordantedEri fabric as compared to alum mordanted fabric.



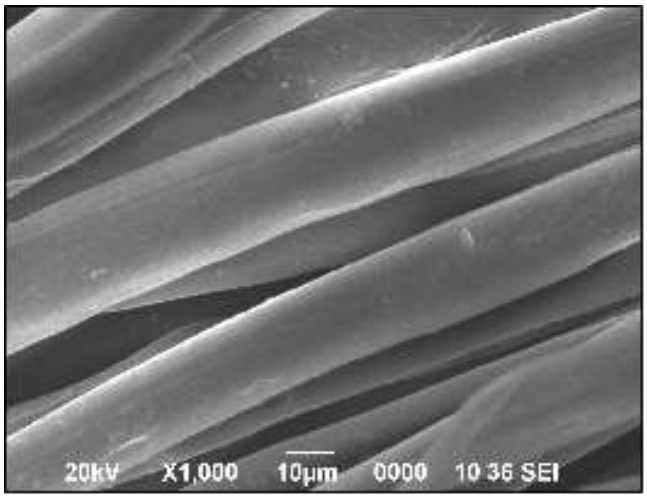
(a) Raw Eri silk fabric



(b) Non-mordanted Eri silk fabric



(c) Alum mordantedEri silk fabric



(d) MyrobalanmordantedEri silk fabric

Fig 5: SEM images of raw and dyed Eri silk at ×1000

Conclusion

Natural dyes are those which are used in eco-friendly dyeing of textile products. The demand for natural dyes recently increasing throughout the world due to awareness of health, environment as well as to reduce the toxic, hazardous substances produced by the synthetic dyes. On the basis of the findings, it was concluded that use of natural mordants resulted in a greater depth of colour and higher colour strength (K/S) values as compared to metallic mordant. Since the dye is easily available in the North-East Region, it will be very much useful for the people who are interested to do work with natural dyes and can create ample of opportunities for generating income.

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