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Extraction of dye from eucalyptus bark for dyeing of silk using natural mordant

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Abstract
In recent years due to awareness of ill effects on health and environmental concerns caused by synthetic dyes people are shifting towards natural dyes and there has been a great resurgence of natural dyes. As an application of natural dyes on textile materials it has a significant role in the decline of environmental pollution. In this present study natural dye was extracted from the bark of Eucalyptus *globulus* with the dyeing of mulberry silk yarn and banana pseudo stem was used as a natural mordant. An aqueous medium was employed with an optimum wavelength of 580 nm and was examined under UV-Visible spectroscopy with the highest optical density is 1.743. Optimal results were achieved when dyeing at 60 °C with different mordanting techniques. The dye extraction methods with different dyeing conditions were optimized and optical density values of the dye solution before and after were recorded. The colourfastness of all the dyed samples was found to be satisfactory. SEM and FTIR were also done. Therefore it was found that eucalyptus bark can be effectively used for dyeing mulberry silk yarn.

Keywords: Banana pseudo stem sap, eucalyptus bark, mulberry silk yarn, natural dyes, natural mordant

1. Introduction

The world's rich biodiversity has been created by varied climatic zones, elevations, and geographic diversity, which has given flora and fauna a variety of perennial and shrub plants that generate colours [1]. Since everyone has different preferences and a natural inclination towards colour. By providing an appealing dimension to a surface and conveying thoughts and feelings, the use of colour improves its aesthetic quality, bringing beauty and interest into our daily lives. The impact of colour on human existence has existed since the beginning of mankind. The only source of colour ants available to humans in primitive times was naturally available sources [2]. The necessity for a sustainable "green economy" has gained increased recognition as a result of rising public awareness of environmental problems and health risks [3]. Researchers are still looking into the subject even though natural dyes are unable to fulfill the requirements of the global demand due to their ecological advantages, non-toxicity, and biodegradability [4].

Natural dyes are produced from a variety of plant parts, including roots, bark, leaves, flowers, and fruits. They are available in a wide variety of shades [5]. Shabbir, *et al.* [6] Natural dyes not only add colour, but they also provide UV protection, deodorization, antifeedant, fluorescent, antifungal, antibacterial, and other beneficial finishing features. It improves the product's intrinsic and aesthetic valuation [7].

Silk is referred to as the queen of textiles by Boruah *et al.* [8] because of its intrinsic beauty and luster. Because of the delicate, highly durable, glossy hues that they produce, natural dyes perform very well with silk. Even after a long time, they are still attractive and elegant.

Eucalyptus *globulus* is usually found all over the world; the growth rate of eucalyptus is 35,000 per year [9]. 10% to 12% of the naturally occurring tannins and polyphenols in eucalyptus bark are present in substantial amounts, and the majority are either wasted or utilised as fuel [10]. According to Grattapaglia *et al.* [11], it has a long history of usage in traditional medicine and as a deodorant, antiseptic, and treatment for ailments like allergies, wounds, fevers, asthma, diabetes, malaria, rhinitis, migraines, cough, and tuberculosis. The bark of the eucalyptus species includes a variety of colours, including tannins, polyphenols, and the most vibrant yellow pigment known as quercetin [12]. The objective of this research is to ascertain whether silk may be constantly dyed using other dyeing agents and eucalyptus bark extracts as a natural dye.

2. Materials

Eucalyptus bark was selected and collected from the University of Assam Agricultural University, Jorhat. Processing begins with the removal of contaminants, such as dirt from eucalyptus bark, using brushes before being washed with water [13]. Following processing, eucalyptus bark has been cut into small pieces with the use of a sharp knife, according to Naseer *et al.* [14]. Processed dye was ground into a powder using an electrically powered high-efficiency grinder machine. The screened powder was preserved for future use in an airtight container with identification and kept in a cool, dark place. They are shown in the figures below. Degumming and bleaching were done for selected mulberry silk yarn. The solution for degumming was prepared by adding 5 g of washing soda by maintaining the material-to-liquor ratio at 1:50 for 30 minutes. For bleaching, the yarn samples were treated with 1% of H_2O_2 at 60 ºC for 30 minutes by maintaining the material-to-liquor ratio of 1:50. The silk yarns were taken out and rinsed thoroughly and dried after degumming and bleaching processes.

Eucalyptus bark Dye powder Extracted dye

Fig 1: Eucalyptus bark, dye powder and extracted dye.

3 Methods

3.1 Extraction of dye

Natural dye from eucalyptus bark was extracted by using an aqueous extraction method. About 1g of the processed eucalyptus bark was taken in different beakers containing 100 ml water to keep the dye material fully immersed in this solution. The beakers were then incubated at different temperatures i.e. 40°, 60°, 80°, and 100 °C and were taken out at different periods i.e. 15 min, 30 min, 45 min, 60 min, 75 min, and 90 min. All experiments were performed at least in triplicate and results were expressed as means of standard deviation. The extracts were filtered through a Whatman no 1 filter paper. The extracts were placed in a clean dried pre-weighed flask and then the volume of the extract was determined. The optical density (OD) value of each of the extracted dye liquor was recorded using a spectrophotometer. The highest optical density value determined by using a spectrophotometer from the various parameters was noted.

3.2 Extraction of mordant

Fig 2: Extracted mordant

Banana pseudo stem was extracted from the *Musa chinensis* and has been used as a mordant to dye the selected mulberry silk yarn. After the fruit bunch had been collected, it was removed by scraping with a blunt knife and then being squeezed. After that, a Whatman no. 1 filter paper was used to filter the extracted mordant.

3.3 Method of dyeing

To obtain the most optimal dyeing conditions, silk yarn was dyed by using different dyeing intervals and dye-material concentrations while maintaining a constant temperature and M: L ratio, i.e. 60 °C and 1:50. Dyeing was carried out at different time intervals i.e. 30, 45, 60, 75 and 90 minute and dye material concentration i.e. 1, 2, 3, 4 and 5 gm/100 ml. All experiments were performed at least in triplicate and results were expressed as means of standard deviation. With occasional stirring, the yarns were dyed in the dye bath. After the dyed samples had cooled for an hour, they were rinsed, cleaned, squeezed, and dried under shade [15]. Silk yarns were dyed in a dye bath with optimum time and temperature. The dye baths were prepared by adding 6% banana pseudo stem mordant on the basis of the weight of the yarn. The pre-mordanting method was followed for dyeing.

3.4 Determination of colour co-ordinates

The CIE Lab colour coordinates value of the dyed samples was calculated using a Brightness, Opacity, and Colour Tester (Model No. UEC-1080) with an average of five readings. The colour parameters L* (depth of colour), a* (positive and negative value redness and greenness), and b* (positive and negative value yellow and blueness) were noted. The values obtained from the study's mordant were determined. Higher values of a* and b* indicated brightness of colour, whereas lower values of L value suggested deeper depth of colour.

3.5 Evaluation of colour fastness properties of dyed yarn

All the silk yarn samples that had been dyed were examined by using the ASTM method[16] to determine the way their colours would hold up against washing, exposure to sunlight, crocking (dry and wet), pressing (dry and wet), and acidic and alkaline perspiration.

3.6 Fourier Transform Infrared Spectroscopy (FTIR)

Using Fourier Transform Infrared Spectroscopy (FTIR), the control and dyed silk yam were analyzed. Wave numbers between 600 and 4000 cm-1 are used to describe the frequency range. The resulting absorption spectrum, which additionally reveals the sample's composition and functional groups.

3.7 Scanning Electron Microscopy (SEM)

Using a JOEL JSM-6360 Model Microscope, Oxford, SEM was used to analyse the surface morphology of the samples on the control and coloured silk yam. The samples were visualised in exquisite detail using high-quality, spatially-resolved images.

4 Results and Discussion

Dyeing was performed with M:L ratio of 1:50 at 60°C for 60 minutes with occasional stirring and then allowed for a further 30 minutes as the bath cools down. The pre-mordanting method was followed for dyeing using 6% banana pseudo stem mordant.

(a) Control silk yarn (b) Non- Mordanted silk yarn (c) Mordanted silk yarn

Fig 3: Different shades of colour produced by eucalyptus dye on mulberry silk yarn

4.1 Evaluation of CIE lab colour co-ordinates

Table 1: CIE lab colour co-ordinate values of eucalyptus bark dyed yarn

Dyed yarns	CIE Lab values		
	L*	a*	b*
Non-mordanted	68.84	8.07	10.96
Mordanted	60.67	9.58	15.66

L*= higher depth of colour
a*= redness (positive a*) and greenness (negative a*)
b*= yellowness (positive b*) and blueness (negative b*)

From Table 1, it was found that mordant samples had the lowest L* values—60.67, indicating deeper colours—while non-mordanted dye samples had the greatest L* values—68.84, indicating lighter colours. Therefore, it can be stated that samples that have been mordanted provide a deeper level of colour. The dyed yarn's presence of redness and yellowness suggested that a* and b* had positive values.

4.2 Evaluation of colourfastness properties of dyed yarn

After dyeing in optimum dyeing conditions, the mulberry silk yarns were evaluated for their colourfastness properties by use of the International Gray Scale compared with the controlled sample. According to each test specimen, colour change (CC) and colour staining (CS) ratings were evaluated and ratings were done. CC Ratings: 1=poor, 2=fair, 3=very fair, 4= good, 5= very good
CS Ratings: 1= heavily stained, 2=considerably stained, 3= noticeable stained, 4= slightly stained, 5= negligible or no staining.

Table 2: Ratings for colourfastness properties of dyed samples

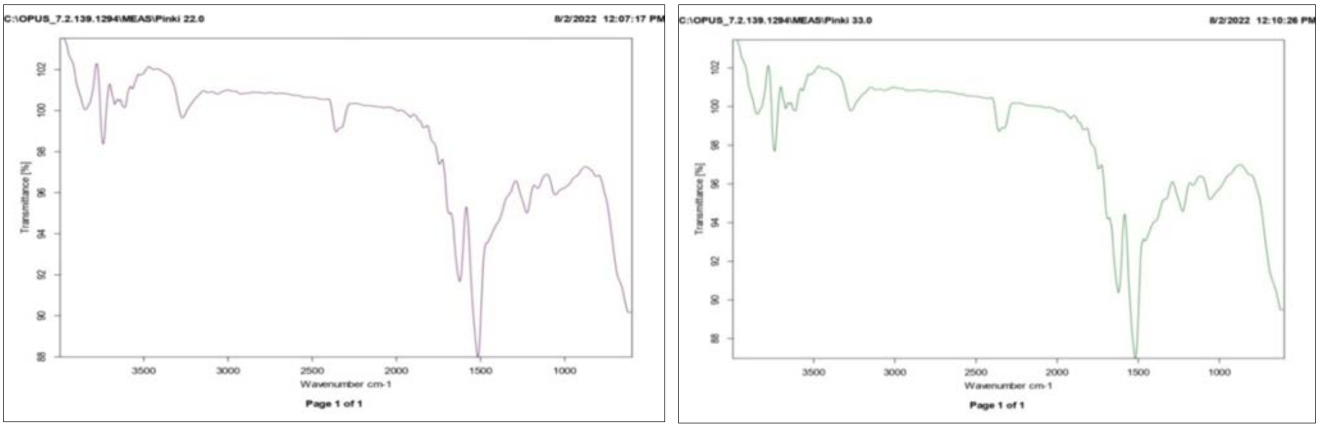
SL. No.	Mordant used	Washing		Crocking				Pressing				Sunlight		Perspiration		
				Dry		Wet		Dry		Wet				Acidic	Alkaline	
		CC	CS	CC	CS	CC	CS	CC	CS	CC	CS	CC	CC	CS	CC	CS
1	Non-mordanted	3	4	4	3	5	4	5	5	4	5	4	5	5	3	4
2	Mordanted	4	5	4	5	5	5	4	5	5	5	5	4	5	4	4

From the table, it could be seen that samples that were non-mordanted had very fair colorfastness to washing but were slightly stained. The mordanted dyed sample, however, exhibited good fastness properties with negligible stain. The non-mordanted samples showed good colorfastness to dry crocking with noticeable stains, while the mordanted samples showed good with negligible stains. The mordanted sample showed very good fastness properties while being slightly stained in wet crocking. Non-mordanted dyed yarn showed no colour stains.
All samples i.e. mordanted and non-mordanted exhibited very good to good fastness properties in dry pressing and no stains. However, all of the samples displayed good to very good fastness and negligible stain during wet pressing. All samples, whether mordanted or not mordanted, showed very good to good colorfastness to dry pressing and no stained. However, all of the samples displayed good to very good fastness with negligible stain during wet pressing.
It was found that both non-mordanted and mordanted samples had well to very good fastness properties to sunlight. The yarn samples were tested for colorfastness to perspiration in both acidic and alkaline mediums. All of the samples were found to have good to very good fastness in an acidic medium, with negligible stain. The non-mordanted sample had very fair

fastness with a slightly stained in an alkaline medium. The mordanted dyed sample showed good fastness properties with slightly stained.

4.3 Fourier Transform Infrared Spectroscopy (FTIR) analysis

Peaks between 1625 and 1650 cm-1 show the presence of an alpha-beta unsaturated carboxyl group, which is a characteristic of the yellow flavonoid pigment[17]. While the band between 1610 and 1570 cm-1 indicates the presence of an aromatic ring[18], the peaks between 3030 and 3010 cm-1, which are commonly regarded as bands for flavonoid pigments, denote the C-H bond. The peaks between 1610 and 1570 cm^{-1} and 3030 and 1010 cm^{-1} in the images were used to identify the presence of flavonoids in the dye pigment. The spectrum showed that from the mordanted silk yarn, there were distinct peaks in each of the dyed samples. The functional group 3640-3610 of alcohols and phenols were found to have the highest peak in Fig 4 (a) and (b). The FTIR graph indicates the presence of the phenolic group, aliphatic amines, alcohols, carboxylic acid, esters, ethers, and phenols.



Non-mordanted Mordanted

Fig 4: FTIR Spectra of non-mordanted and mordanted

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

At a 1000 x magnification, the SEM image of a control silk fibre is seen in (Fig 5). Due to the existence of a sericin layer in the raw silk fibre, the control silk fiber's surface was covered in a gummy-like substance and exhibited irregularities. At a magnification of 1000x, the SEM images of the control, non-mordanted, and mordanted dyed silk fibre were displayed in Fig 5 (a), (b), and (c). Non-mordanted silk has a rough surface and a more cylindrical shape than controlled silk fibre. It might be due to the removal of the sericin layer and other materials during degumming and bleaching procedures. Due to the dye's absorption capacity by the fibre under the dyeing conditions, mordanted coloured silk had a more uniform and smooth cylindrical structure in addition to a slight increase in the diameter of the fibre.

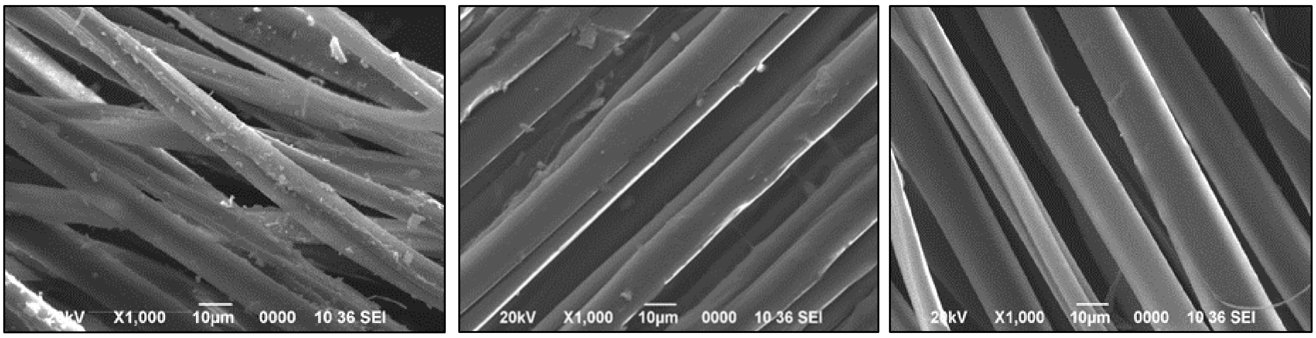


Fig 5: SEM images of control, non-mordanted and dyed silk yarn

5. Conclusion

Eucalyptus bark extract can be used to colour mulberry silk yarn well, and it can also be used to create apparel. The optimised extraction parameters were 45 min of extraction time at 100 °C, 4 g of dye material concentration, 6% of mordant concentration, and 60 min of mordanting duration. While dyeing, the pre-mordanting procedure was chosen. The mordanted sample's Colour Strength (K/S) measurement had the highest value of 9.11 K/S. The non-mordanted dyed sample had the lowest depth of colour and the greatest L* value (68.84) according to the CIE lab results. The presence of redness and yellowness in the dyed yarn was indicated by the positive values of a* and b*.

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