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Dyeing of Wool Using Conical Prickles of Bombax Ceiba Bark for Imparting Antioxidant and UV Protection Activities

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ABSTRACT

The popularity of natural dyes is gradually increasing worldwide because of their sustainable and eco-friendly dyeing properties. The potential of natural dyes to replace synthetic dyes is expected to be explored by a new researchers. Bombax Ceiba is a tree widely known for medical purposes. The present work focuses on developing functional wool using natural dye obtained from conical prickles of Bombax Ceiba bark. The colouration of wool using conical prickles of Bombax Ceiba bark was done in a rota-dyer machine. Assessment of the fastness and functional properties of the dyed fabrics was performed. Mordants (aluminum ferric sulphate and chitosan) were utilised to dye the wool. Satisfactory fastness and good colouration properties were achieved by dyed fabric. More than 76 % antioxidant activity and excellent UV protection were acquired by the dyed fabrics. The dyed wool fabric confirmed K/S>1 and wash fastness ratings in the range "good" to "excellent". Thus, the novel combinations of conical prickles of Bombax Ceiba bark and various mordants were explored for the functional colouration of wool fabric. Prog. Color Colorants Coat. 18 (2025), 375-381© Institute for Color Science and Technology.

1. Introduction

Nowadays, sustainable routes in the textile industries for producing functional textiles are primarily in demand. Generally, dyeing is used to produce the coloured fabric, and finishing is performed after dyeing to impart functional effects such as antibacterial, antioxidant, and UV protection. These separate operations need vast amounts of labor, process, and machinery costs; moreover, they produce a large quantity of textile effluent. Modern textile generations are focusing on new routes to minimize the load on effluent treatment plants. Natural dyes originated from nature and are one of the best choices to provide sustainable, functional dyeing effects [1].

The massive use of synthetic dyes for the colouration of textiles is done by textile industries. Synthetic dyes have several advantages, such as easy colouring ability and higher affinity. However, they generate toxic dyeing effluent and produce carcinogenic effects. Due to the harmful effects of synthetic dyes, the textile industries are now onwards shifting to natural dyes. Natural dyes can be a perfect alternative to synthetic dyes due to their non-toxic and eco-friendly nature [1]. The functional effects of some of the natural dyes are of further interest to exploration. The lack of affinity of natural dyes for textiles is a significant concern, which is improved by the bio [2-5] and metal mordanting [6-9]. Mordanting is done by two ways: pre-mordanting and post-mordanting. Pre-mordanting ensures uniform dye adsorption on the fibre, as the present mordant helps achieve even dye

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distribution. However, the presence of the mordant on the fibre can sometimes hinder the dye's ability to diffuse deeply into the fibre. Post-mordanting allows for more even diffusion of dye into the fibre. However, the impact of post-mordanting tends to be more pronounced on the surface of the fibre, which can result in variations in mordant diffusion throughout the substrate.

Bombax Ceiba plant is found in India, Bangladesh, and Malaysia and is also known as the silk-cotton tree. It is widely accepted for medical purposes. It has various properties such as anti-HIV [10], anti-inflammatory [11], antiangiogenic [12], antibacterial activity [13], anti-oxidant activities [14], and hepatoprotective [15]. Sheikh et al. used *Bombax Ceiba* (Kapok) flower extract for the colouration of linen, and the dyed fabric imparted functional effects (antibacterial activity, UV protection, and antioxidant activity) [16]. The natural dye obtained from *Bombax Ceiba* bark was utilised for the dyeing of cotton fabric [17]. Multifunctional linen was prepared using zinc oxide nanoparticles and Bombax *ceiba* flower extract [18]. *Bombax Ceiba* flower extract was used to colour the wool with the help of tannin-based mordants [4].

Ibrahim et al. developed antibacterial and UV-protective cotton using natural dyes (Madder, Curcuma, Onion, Henna) [19]. Natural dyes (pomegranate, curcumin) were used to provide multifunctional protection to cotton [20]. Verma et al. prepared UV protective and antibacterial cotton fabric using chitosan as a mordant and onion skin as a dye [21]. UV protection to cotton was imparted by using mordant-free natural dyeing with the help of natural dyes (onion peel and catechu) [22]. Dyeing of wool using madder dye and bio-mordants (yellow myrobalan and black myrobalan) was explored [23]. Hosseinnezhad et al. used Sumac as a bio-mordant and Reseda green dye to colour silk fibres [24].

Bombax Ceiba's bark has been very limited explored for the colouration of textiles. Conical prickles of *Bombax Ceiba* bark as a source of natural dye for the colouration of wool is not studied till now. The present work investigates the functional dyeing of wool by using aluminum ferric sulphate and chitosan as mordants. The colouration and functional properties provided by dyed fabrics were evaluated. The fastness properties of dyed wool were also studied to confirm the dye-fibre interaction. Thus, the first attempt of dyeing with *Bombax Ceiba's* bark and aluminum ferric sulphate and chitosan as mordants were explored.

2. Eperimental

2.1. Materials

100 % wool fabric having ends/inch of 60/2, picks per inch of 52/2, and grams per square meter of 140 was purchased from Wool Research Association Mumbai. The collection of kapok bark was done from the local campus of the Indian Institute of Technology Delhi.

2.2. Methods

2.2.1. Preparation of kapok dye solution

The 50 g of conical prickles was washed with 300 mL of water, and its drying was done using an oven at 50 °C for 24 h. The dried prickles were crushed using mortar and pestle. The obtained powder was reddish-brown in colour. The aqueous solution of 6 % (w/v) was prepared by dissolving 12 g of the dye powder in 200 mL of distilled water using heating at 90 °C for 75 min. With continuous stirring of 500 rpm. The extract was filtered and was directly used for dyeing.

2.2.2. Dyeing of wool fabric

Dyeing was carried out in a two-step process: pre-mordanting and dyeing.

Mordanting of wool was carried out with two different mordants (aluminum ferric sulphate and chitosan) at different concentrations (1, 3, and 5 %) on the weight of the fabric (owf). 1 % (w/v) solution of aluminum ferric sulphate was prepared in water at 40 °C for 30 min. A Chitosan solution of 1 % (w/v) was made by dissolving it in water with the help of 1 % (w/v) acetic acid. Treatment of fabric with aluminum ferric sulphate and chitosan was done at 90 °C for 1 h at a material-to-liquor ratio of 1:40 in a rota dyeing machine (RBE, India). The mordanted fabric was squeezed and processed for dyeing.

The mordanted wool was dyed with varying concentrations of dye using a liquor ratio of 1:40 at 90 °C for 60 min in the rota dyer machine. After completing the dyeing of the wool fabric, the fabric was washed with tap water to remove the excess dye on the fabric, and later on, the fabric was air-dried.

2.2.3. Evaluation of colouration properties of dyed fabrics

The colouration properties: colour values (L*, a*, b*), and colour strength (K/S) were evaluated using a spectrophotometer (Color-Eye 7000 A, X-Rite, Switzerland).

2.2.4. Evaluation of fastness properties of dyed fabrics

Washing fastness, rubbing fastness, and light fastness of dyed fabric were determined as per ISO 105- C06 A2S:2010, ISO105-X12:2001, and ISO 105 BO2:2013, respectively [25].

2.2.5. Evaluation of functional properties

The antioxidant activity of dyed fabrics was evaluated using DPPH (2,2 Diphenly-1-picryhydrazyl) as per the reported literature [3]. Dyed wool fabrics were dipped in a methanolic solution of DPPH solution and shaken at 37 °C for 30 minutes in the dark. The DPPH scavenging activity (%) was then determined using the formula below.

$$\text{Antioxidant activity } (\%) = [(\text{Ab} - \text{As})/\text{Ab}] \times 100$$

Ab=absorbance. of blank DPPH solution, As=absorbance. AS/NZS 4399 (1996) method was used to obtain the UV protection activity of dyed samples [26].

3. Results and Discussion

3.1. Colour values and colour strength of the dyed samples

The colour values of fabric samples are presented in Table 1. A total of 9 samples were made, out of which 3 were without mordant, and 6 other samples were dyed with varying mordant concentrations. All dyed samples showed good colour values. The term "owf" indicates the amount on the weight of fabric. Even without the application of the mordant, good colour shades were developed. By incrementing the amount of dye and mordant on the weight of the fabric, improvement in colour strength (K/S) was observed (Table 1). Dyed samples showed L* values in the range of 69.07 to 70.158, a* in the range of 2.614 to 5.139, and b* in the range of 9.516 to 12.40. More than 1 colour strength (K/S) was obtained for dyed wool.

Without the application of mordants, good coloured shades were developed, which suggests the colouration ability of the dye for wool in the absence of the mordant. Figure 1 shows the photographs of representative samples. The photos of dyed samples clearly show that the pre-mordanted samples have darker shades than the undyed and unmordanted samples.

Table 1: Colour strength and colour values of the dyed wool.

Sample no	Mordants (owf %)		Kapok Dye (owf %)	Colour values$			Colour strength$
	Aluminum Ferrous Sulphate	chitosan		L*	a*	b*	K/S
1	0	0	10	69.4	2.723	9.803	1.997
2	0	0	20	69.273	3.519	9.516	2.285
3	0	0	30	69.575	4.954	10.886	2.603
4	1	0	10	69.793	2.614	11.068	5.280
5	3	0	20	70.158	3.617	12.40	8.295
6	5	0	30	70.072	4.086	12.264	11.154
7	0	1	10	69.914	3.354	11.614	2.387
8	0	3	20	69.07	3.979	11.079	2.412
9	0	5	30	69.654	5.139	11.066	3.200

$ represents the average value of three replicates

Figure 1: Photographs of undyed and dyed samples.

3.2. Fastness properties of dyed samples

The fastness properties of dyed wool fabric were evaluated, and the obtained results are mentioned in Table 2. In the absence of a mordant, washing fastness of 3-4 and 3 were obtained. With the mordant-assisted dyeing, the fastness rating was improved. The improvements in washing and rubbing fastness ratings with the mordants suggested the role of mordants in the dyeing operation. Lightfastness in the range of satisfactory was obtained. The rubbing fastness of 3 or more than 3 also offered the resistance of the dye during excessive rubbing in both dry and wet conditions. Thus, the role of mordants in improving the colouring ability of the dye to wool was confirmed. The role of mordants to provide enhanced exhaustion and fastness was confirmed.

Figures 2 and 3 depict the interaction of the dye with wool. The wool was treated with mordants (chitosan and aluminum ferric sulphate). Wool is amphoteric in nature, has both charges (positive and negative), and chitosan interacts with wool through electrostatic bonding. Bombax Ceiba's bark contains gallic acid as a component [27]. An electrostatic bonding between gallic acid and chitosan is possible. Thus, chitosan acted as a mordant for the dye and contributed to improving the dyeing ability of the dye to wool. In the mordanting with aluminum ferric sulphate, Fe^{3+} is generated and interacts with gallic acid and wool by coordination bonding. Al^{3+} interacts in a similar manner to Fe^{2+} and contributes to the mordanting process. In this way, aluminum ferric sulphate played a role as a mordant.

Gallic acid can also directly interact with wool through hydrogen bonding, which occurs without a mordant. Mordants serve as an anchoring agent between the dye and the fibre, enhancing both the colouration and the fastness properties, as suggested by the mechanism above. Thus, due to the strong action of the mordants, fastness ratings are improved.

Table 2: Fastness of dyed fabrics.

Sample no	Washing Fastness	Rubbing Fastness		Light Fastness
		Dry	Wet	
1	3-4	3-4	3-4	4
2	3-4	3	3	4-5
3	3	3	3	5
4	5	5	5	4-5
5	5	4-5	4-5	5
6	4-5	4-5	4-5	5
7	4-5	5	5	4-5
8	4-5	5	5	5
9	4-5	4-5	4-5	5

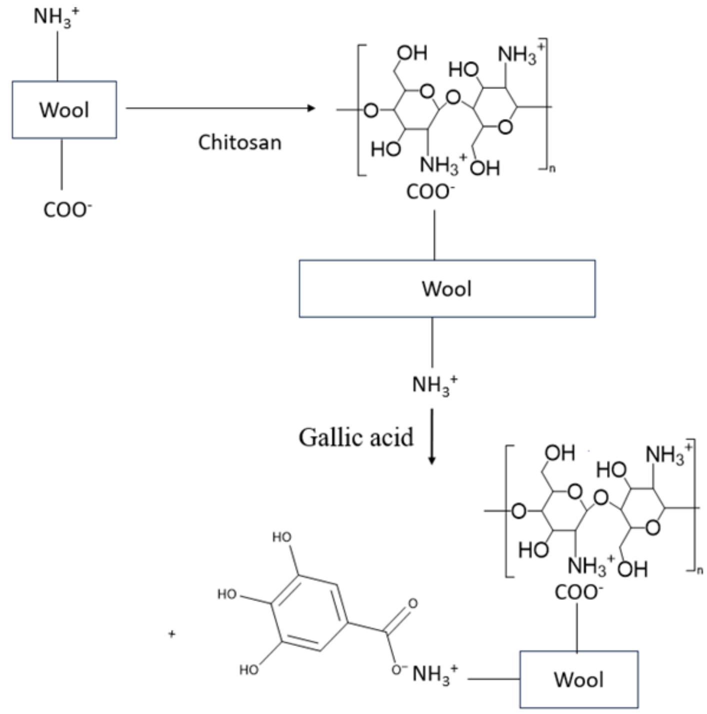

Figure 2*:* Interaction of dye with wool and chitosan.

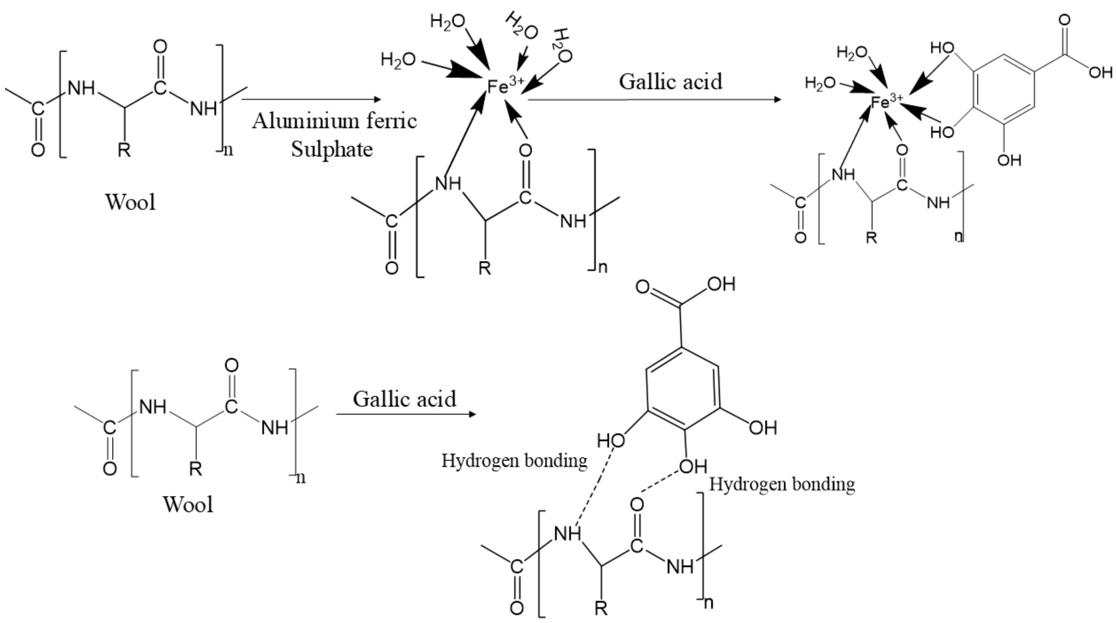

Figure 3*:* Interaction of dye with wool and aluminium ferric sulphate.

3.3. Functional properties of dyed wool fabric

Table 3 indicates the antioxidant activity of dyed wool fabric. Undyed wool showed an antioxidant activity of 10.31 %. The sample dyed with the dye indicated an antioxidant activity of more than 76 %. With increasing mordant and dye concentration, the antioxidant activity was improved. Conical prickles of *Bombax Ceiba* bark contain a higher number of phenolic compounds, such as scopoletin and gallic acid. Phenolic compounds are well known for showing antioxidant activity. The antioxidant activity of phenolic compounds, such as gallic acid, primarily occurs through the stabilisation of the DPPH. Thus, it imparted antioxidant activity.

Undyed wool showed a poor UPF rating and a UPF value of 5.50 (Table 4). Even without using mordants, the UPF values of wool fabric were improved. The excellent rating was shown by the dyed fabrics (sample 3, sample 6, sample 9). Aromatic phenolic compounds absorb UV rays and prevent their transmission through the fabric, thus imparting UV protection properties.

Table 3: Antioxidant activity of dyed wool fabric.

Sample no	Antioxidant Activity$ (%)
1	89.47
2	94.90
3	95.28
4	88.60
5	89.96
6	94.72
7	76.90
8	86.10
9	91.64

$ represents the average of value of three replicates

Table 4: UV protection activity of dyed wool.

Sample no	UPF value$	UPF rating
Undyed wool	5.50	Poor
3	128.3	Excellent
6	150.5	Excellent
9	160.1	Excellent

$ represents the average value of three replicates

4. Conclusions

The dyeing of wool fabric with natural dye (conical prickles of *Bombax Ceiba* bark) was successfully carried out. Wool dyeing was done with the help of mordant chitosan and aluminum ferric sulphate. The dyed wool fabric also shows good washing and rubbing fastness. The dyed fabric showed more than one colour strength. The role of mordants in improving the fastness rating was confirmed. Antioxidant activity of 76 % and excellent UV protection were shown by dyed wool fabric. Washing fastness in the range of 4-5 was obtained in the presence of mordants. K/S>1 indicated the dyeing ability of novel natural dye for dyeing for wool. A sustainable method of dyeing wool fabric with natural dye (conical prickles of *Bombax Ceiba* bark) for obtaining valuable properties was explored.

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