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# Eco-dyeing of wool with *Rubia cordifolia* root extract: Assessment of the effect of *Acacia catechu* as biomordant on color and fastness properties

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## Abstract

In the present study, anthraquinone colorants were extracted from powdered *Rubia cordifolia* roots and applied on wool fiber. *Acacia catechu* is used as a biomordant, a replacement to metallic mordants, for wool dyeing, and the effect on color characteristics and fastness properties was assessed. Shades on wool of red tones with good to excellent color fastness properties were obtained. Pre-mordanting with *A. catechu* improved the overall color and fastness parameters, due to the maximum dye uptake. Auxochromic groups responsible for interaction of dye and wool were studied by Fourier transform infrared (FTIR) analysis of *R. cordifolia* extract. The surface morphology of dyed wool fiber was investigated by scanning electron microscopy (SEM) for any changes on fiber after dyeing.

**Keywords:** Natural dye, Biomordant, Color, Fastness, FTIR, *Rubia cordifolia*

## Background

In the present context of eco-preservation, natural dyes have acquired tremendous commercial potential as some of the synthetic dyes are associated with the release of enormous amount of hazardous chemicals into the environment (Yusuf et al. 2013, 2015). The revival of interest in the use of natural dyes in textile coloration has been gaining incessant popularity all over the world, probably due to environmental concerns, eco-safety, and pollution control. Natural dyes are supposed to be friendlier and exhibit better biodegradability to the environment than their synthetic counterparts (Yusuf et al. 2015; Shabbir et al. 2016a, b). Reproducibility, inadequate availability, cost efficiency, inadequate degree of fixation, and poor color fastness properties are common discernable drawbacks of natural dyes (Cristea and Vilarem 2006; Hill 1997; Khan et al., 2012). In addition, dyes and pigments of natural origin also possess biological and biomedical activities, so they may serve as a

green alternative towards synthetic dyes (Yusuf et al. 2012; Velmurugan et al. 2009).

Low reactivity of natural dyes to wool has been a concern to researchers in the past, and a lot of research has been done to understand the dyeing mechanism of natural dyes on various textile substrates including wool such as adsorption and kinetic studies. Adsorption and kinetic studies suggest the reactivity behavior of wool towards natural dyes via valence forces, H-bonding, etc., as discussed earlier also (Rather et al. 2016; Shabbir et al. 2016a, b). Although metallic mordants are used to enhance the affinity of natural dyes to textile fibers, they generate wastewater containing residual toxic metal ions which leave negative impacts on the environment and cause severe health-related problems and allergic responses (Burkinshaw and Kumar 2009; Zheng et al. 2011). In this regard, researchers searched for a greener substitute from flora and fauna. Consequently, green alternatives have been employed which have high tannin and/or metal-hyperaccumulating contents (Prabhu and Teli 2014; Cunningham et al. 2011; Vankar et al. 2008; Shahid et al., 2012; Vankar and Shanker 2009). Mansour and Heffernan (2011) used *Acacia catechu* (cutch) as a green substitute to metal salt with impressive results also.

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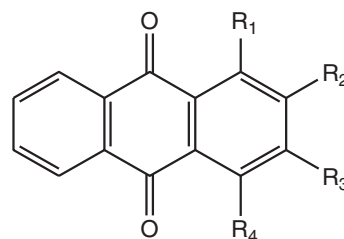
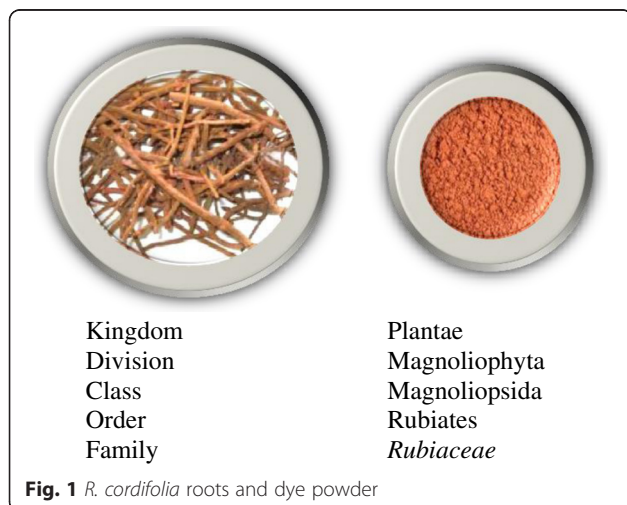
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*A. catechu* is found in most of the Indian sub-Himalayan zone. The powdered heartwood of *A. catechu* contains tannin phyto-constituents and is used for textile coloration from ancient times to impart brown tint shades on textile materials (Bhattacharya and Shah 2000; Bhattacharyya and Lohia 2002; Vankar et al. 2008; Perkin & Everest 1918). Pharmacological studies have demonstrated that *A. catechu* used in traditional medicines shows anti-inflammatory and anticancer activities (Anonymous 2006; Khan et al. 2011).

Madder (*Rubia cordifolia*) is a perennial and herbaceous climber plant having very long, cylindrical, and flexuous root with thin red barks. Its stems often have a long, rough, grooved, woody base. Its old roots are richer in color than the young ones. The roots contain dye present in the free or bound glucosides which are anthraquinone derivatives, mainly purpurin (CI-75410) and munjistin (CI-75370). The roots (Fig. 1) also contain a small amount of xanthopurpurin (CI-75340), pseudopurpurin (CI-75420), nordamncanthal, and rubiadin (Fig. 2.) (Color Index 1971; Mayer and Cook 1943; Anonymous 1972). Madder has been a promising plant for dyeing from the ancient times and worked upon to produce red dyed textiles of varying characteristics with the help of advance techniques and mordants (Gupta et al. 2001; Montazer and Parvinzadeh 2004; Parvinzadeh 2007). Since the modern people are more aware about eco-friendliness along with their comfort, demand for the greener textiles was raised and the evolution of bio-mordants gained popularity.

This study discusses the dyeing process of wool with *R. cordifolia* root extract as a natural dye, mordanting the ability of *A. catechu* and their effect on color in terms of CIEL\*a\*b\*, color strength and fastness properties with respect to light exposure, washing, and rubbing. The structural morphology of wool fibers was evaluated by using scanning electron microscopy.



Compounds	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	R <sub>4</sub>
Purpurin	OH	OH	H	OH
Munjistin	H	OH	COOH	OH
Xanthopurpurin	H	OH	H	OH
Pseudopurpurin	OH	COOH	OH	OH
Nordamncanthal	OH	CHO	OH	H
Rubiadin	OH	CH <sub>3</sub>	OH	H

**Fig. 2** Color compounds in *R. cordifolia* roots

## Materials and Methods

### Materials

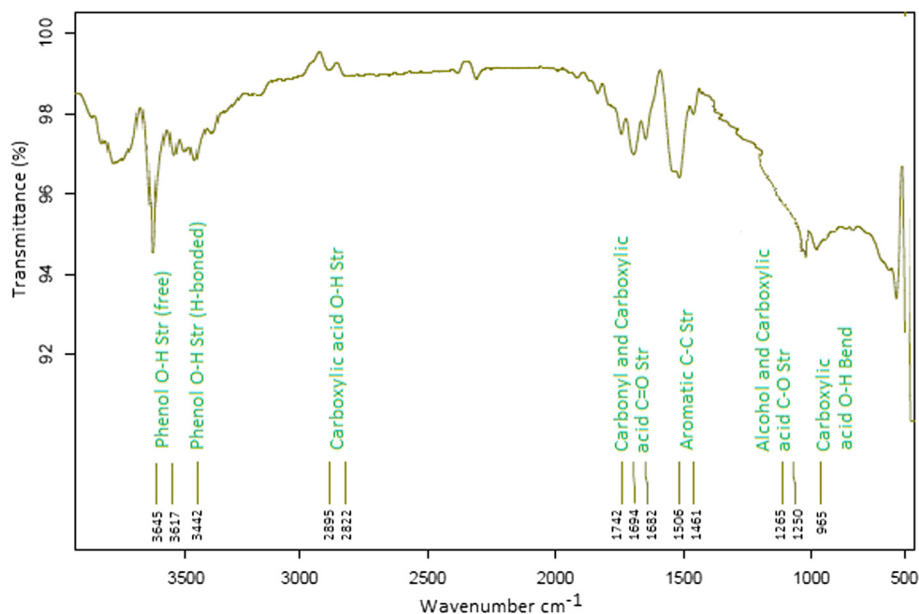
Wool yarn (100 % semi-worsted 60 counts) was purchased from MAMB Woollens Ltd. Bhadohi, UP, India. *A. catechu* extract in powder form was purchased from Sir Biotech India Ltd. Kanpur, UP, India. Powdered madder roots were obtained from SAM Vegetables, Moradabad-244001 (UP), India. All other chemicals used were of laboratory grade.

### Instrumentation

FTIR (Fourier transform infrared) spectra of *R. cordifolia* root powder were recorded on a PerkinElmer Spectrum RXI FTIR system in order to investigate the auxochromic groups responsible for wool-dye interactions (with the resolution of 4 cm<sup>-1</sup>). Bands in the FTIR spectra were analyzed in accordance with the literature data. A PerkinElmer Lambda-40 double-beam UV-visible spectrophotometer was employed for recording absorbance values of dye solutions to assess the dyeing absorptivity. A pH/mV meter (BD 1011) from Decibel digital technologies was used for measuring pH of dye solutions. Scanning electron microscopy (SEM) technique was used to investigate the surface morphology of the wool samples before and after the application of the biomordant and dye. The samples were glued to aluminum stubs with colloidal silver paint for conductivity and sputter coated with gold for 3 min in an argon atmosphere. The samples were observed and digitally imaged on a LEO 435VP Scanning Electron Microscope at 10-kV accelerating voltage.

### Mordanting

Three methods of mordanting were carried out named pre-, meta-, and post-mordanting.



**Fig. 3** FTIR spectra of *R. cordifolia* dye

#### Pre-mordanting

Prior to mordanting, the wool yarns were immersed in an aqueous solution containing non-ionic detergent (5 mL/L) for 30 min for removing unwanted dust from the surface of the wool. Powdered *A. catechu*, 5.0–0.5 % o.w.f. (on weight of fiber), was added to water in separate baths, and the temperature of the mordant solutions was raised to 30 °C to make bio-mordant completely soluble. Then, water-soaked wool yarns were added to the baths. The temperature of the mordanting baths was brought to 90 °C for 1 h with constant stirring. Inherent pH (6.7–7.1) of the mordanting bath was used for mordanting. After the mordant baths were cooled, mordanted wool samples

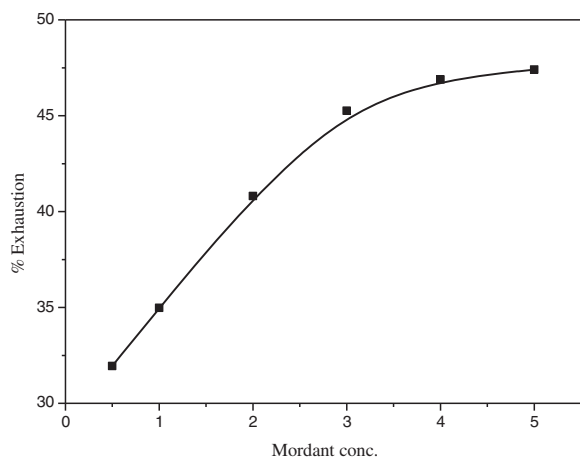
were removed from the mordanting baths and rinsed with tap water.

#### Meta-mordanting

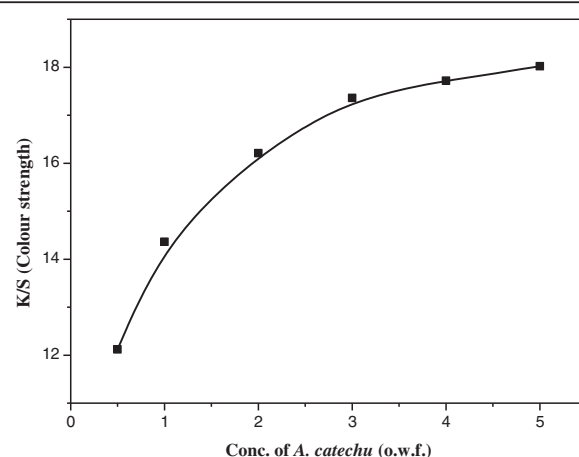
For meta-mordanting, *A. catechu* powder was dissolved in water and the solution was mixed with *R. cordifolia* extract in dye bath for simultaneous mordanting and dyeing. The temperature was raised to 90 °C and kept for dyeing for 1 h with constant stirring.

#### Post-mordanting

*R. cordifolia* extract-dyed wool yarn samples were rinsed with tap water and immersed to the mordant solutions at 30 °C. The temperature of the solution was raised to



**Fig. 4** Optimization of mordant concentration (% dye exhaustion vs concentration of mordant)



**Fig. 5** Optimization of mordant concentration (color strength (K/S) vs concentration of mordant)

**Table 1** Color characteristics and color fastness properties of wool pre-mordanted with *A. catechu* and dyed with *R. cordifolia*

Mordant (%)	L*	a*	b*	c*	h°	K/S	Light fastness	Wash fastness			Rub fastness	
								c.c.	c.s.	c.w.	Dry	Wet
Nil	48.1	40.0	36.2	53.9	42.1	11.02	4/5	3/4	4/5	4/5	4/5	3/4
0.5	46.8	39.2	30.2	49.5	37.5	12.12	4/5	4	4/5	4/5	4/5	3/4
1.0	42.1	38.8	25.2	39.1	32.9	14.36	5	4/5	4/5	4/5	4/5	4
2.0	38.0	36.0	21.9	42.2	31.3	16.21	5	4/5	5	5	5	4
3.0	34.0	32.2	17.1	36.4	27.9	17.36	5	5	5	5	5	4/5
4.0	33.5	30.2	15.6	34.0	27.2	17.72	5	5	5	5	5	4/5
5.0	32.3	30.0	14.0	33.2	25.0	18.02	5	5	5	5	5	4/5

c.c. color change, c.s. color staining on cotton, c.w. color staining on wool

90 °C with constant stirring and kept for 1 h. Post-mordanted dyed wool yarn samples were washed with non-ionic detergent and dried in shade at room temperature.

#### Optimization of concentration of *A. catechu*

The mordant used in this study was optimized, for pre-mordanting, for its better performance on wool in terms of achieving higher color strength and better fastness properties, and the optimized concentration was then used further for meta- and post-mordanting. Concentration of *A. catechu* mordant was optimized on the basis of absorbance values at maximum wavelength (Prabhavathi et al. 2014), recorded before and after dyeing with a UV-vis spectrophotometer, and percentage dye exhaustion was calculated by using the following equation:

$$\% \text{ Dye exhaustion} = [(A_0 - A_1) / A_0] \times 100 \quad (1)$$

where  $A_0$  and  $A_1$  are the absorbance of the dye bath solution before and after dyeing, respectively.

Analysis of the influence of different mordanting methods, namely pre-, meta-, and post-, on the color shades of wool yarns dyed with *R. cordifolia* dye was executed through comparison with the un-mordanted ones.

#### Extraction of colorants

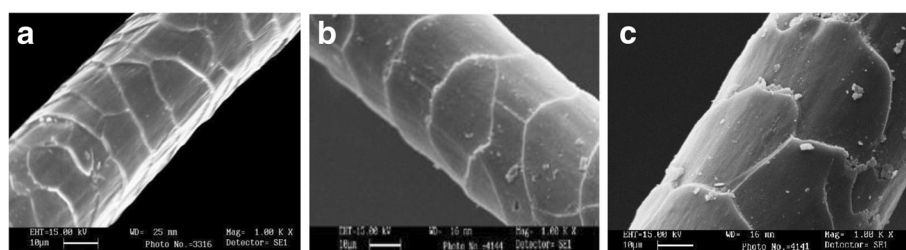
Before dyeing, the colorants were extracted from the powdered madder roots. Fifty percent o.w.f. of powdered madder roots were taken in an acidic (HCl) aqueous solution (pH 3) using material-to-liquor (M:L) ratio 1:20, kept overnight, boiled for 1 h with occasional stirring the next day, and then cooled and filtered through a clean cotton cloth. The dye bath of the extracted reddish yellow madder dye was adjusted to pH 4 ready for dyeing of mordanted and un-mordanted wool yarn samples as previously reported in our paper (Yusuf et al. 2015).

#### Dyeing

The wool yarns were dyed at pH 4 in a bath containing extracted madder dye solution at the liquor ratio of 1:40. The dyeing was started at 30 °C, the temperature was gradually raised till 90 °C, and dyeing was continued for 1 h at this temperature with stirring at regular intervals. The dyed wool yarn samples were washed with 5 mL/L non-ionic detergent and thereafter rinsed with tap water and dried in shade at room temperature.

#### Color measurement

The colorimetric properties of the dyed wool yarn samples were obtained with Gretag Macbeth Color-Eye 7000 A Spectrophotometer integrated with a computer in terms of CIEL\*a\*b\* color coordinates ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $c^*$ ,  $h^\circ$ ) and color strength values ( $K/S$ ). The color strength value ( $K/S$ ) in the visible region of the spectrum (400–



**Fig. 6** SEM images showing the surface morphology of **a** raw wool, **b** *A. catechu*-mordanted wool, and **c** *A. catechu*-mordanted and *R. cordifolia*-dyed wool

**Table 2** Color characteristics and color fastness properties of wool dyed with *R. cordifolia* and (pre-, meta-, and post-) mordanted with 3 % o.w.f. *A. catechu*

Mordant	L*	a*	b*	c*	h°	K/S	Light fastness	Wash fastness			Rub fastness	
								c.c.	c.s.	c.w.	Dry	Wet
Nil	48.1	40.0	36.2	53.9	42.1	11.02	4/5	3/4	4/5	4/5	4/5	3/4
Pre-mordanted	34.0	32.2	17.1	36.4	27.9	17.36	5	5	5	5	5	4/5
Meta-mordanted	35.2	31.6	16.1	35.5	26.9	14.33	5	4/5	5	5	5	4
Post-mordanted	34.8	31.9	16.8	36.0	27.7	16.81	5	4/5	5	5	5	4

c.c. color change, c.s. color staining on cotton, c.w. color staining on wool

700 nm) was calculated based on the Kubelka–Munk equation:

$$\text{Color strength } (K/S) = \frac{(1-R)^2}{2R} \quad (2)$$

where  $K$  is the absorption coefficient,  $R$  is the reflectance of the dyed sample, and  $S$  is the scattering coefficient.

Chroma ( $c^*$ ) and hue angles ( $h^\circ$ ) were calculated using the following equations:

$$\text{Chroma } (C^*) = \sqrt{a^2 + b^2} \quad (3)$$

$$\text{Hue angle } (h^\circ) = \tan^{-1}\left(\frac{b}{a}\right) \quad (4)$$

### Color fastness tests

#### Light fastness

The light fastness of the dyed wool yarn samples was conducted on Digi light NxTM, having water-cooled Mercury Blended Tungsten lamp as per test method AATCC 16e-2004 similar to ISO 105-B02:1994 (Amd.2:2000), which has the nearest approach to that of the sunlight. Light fastness ratings have been done on 1–8 scale as per ISO 105-B02:1994 (Amd.2:2000).

#### Wash fastness

The wash fastness of the dyed wool yarn samples was tested in Digi wash SSTM (Lauder-o-meter) as per the ISO 105-C06:1994 (2010) specifications. The changes in color of the dyed specimens were assessed, both in terms of alteration of shades and the degree of staining on white adjacent fabrics (cotton and wool) against the standard five-point gray scale. A grade of 5 is excellent, and a grade of 1 is very poor. Color fastness rating of less than grade 3 indicates considerable alteration in color after washing.

#### Rub fastness







Dry and wet rub fastness of the dyed wool yarn samples was tested using a Digi crockTM (Crockmeter) as per Indian standard IS 766:1988 similar to ISO 105-X12:2001 by mounting the fabric on a panel and giving ten strokes for both dry and wet rub fastness tests.

## Results and discussion

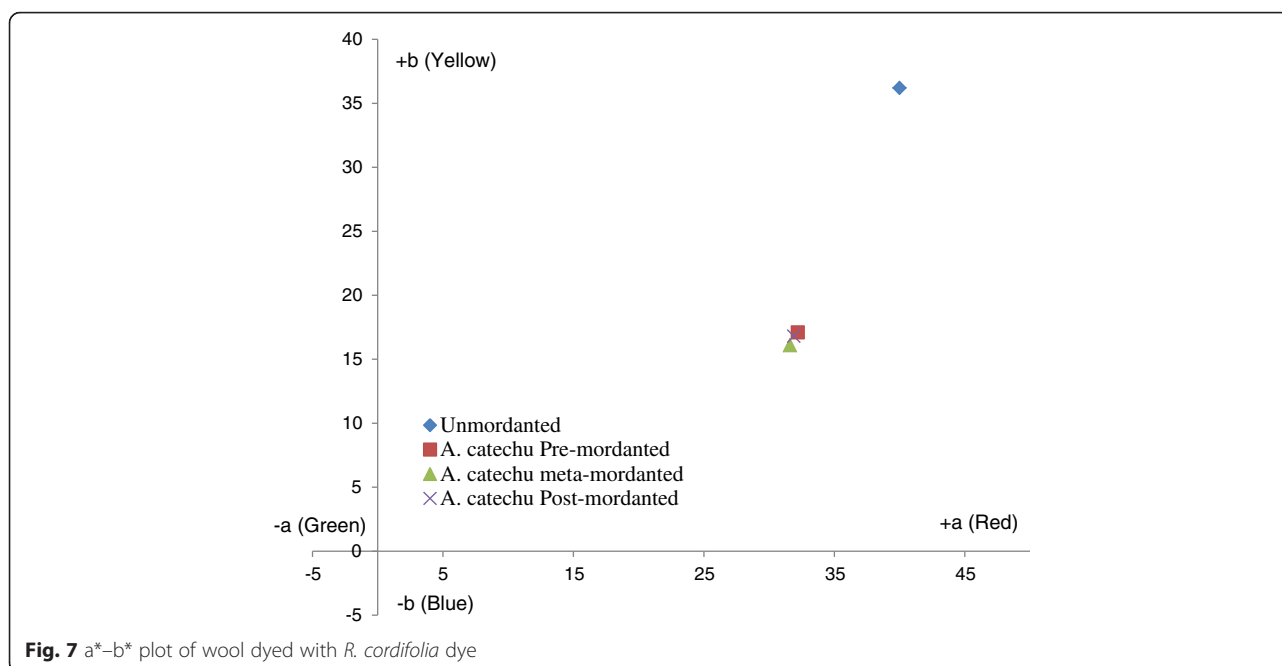
### FTIR spectra of *R. cordifolia* dye

FTIR analysis was used to identify the possible auxochromic functional groups responsible for the dyeability of *R. cordifolia* dye. Figure 3 represents the FTIR spectra of *R. cordifolia* dye which showed characteristic bands at 3645 and 3617  $\text{cm}^{-1}$  corresponding to free phenolic

**Table 3** Shade card for *R. cordifolia* (50.0 % o.w.f.) dye with 3.0 % *Acacia catechu* biomordant

Unmordanted	Pre-mordanted	Meta-mordanted	Post-mordanted
			
Naked wool		Mordanted wool	
			





O–H stretching;  $3442\text{ cm}^{-1}$  for H-bonded phenolic O–H stretching;  $2895$  and  $2822\text{ cm}^{-1}$  for carboxylic acid O–H stretching;  $1742$ ,  $1694$ , and  $1682\text{ cm}^{-1}$  for carbonyl and carboxylic acid C=O stretching;  $1506$  and  $1461\text{ cm}^{-1}$  for aromatic C–C stretching (in ring);  $1265$  and  $1250\text{ cm}^{-1}$  for alcohol and carboxylic acid C–O stretching; and  $965\text{ cm}^{-1}$  for carboxylic acid O–H bending. These results demonstrate the existence of anthraquinone compounds in *R. cordifolia* dye (Nakahara and Fukuda 1979; Richardson et al. 1988).

#### Optimization of biomordant concentration

Figures 4 and 5 depict the optimization of *A. catechu* as a biomordant on the basis of % dye exhaustion and color

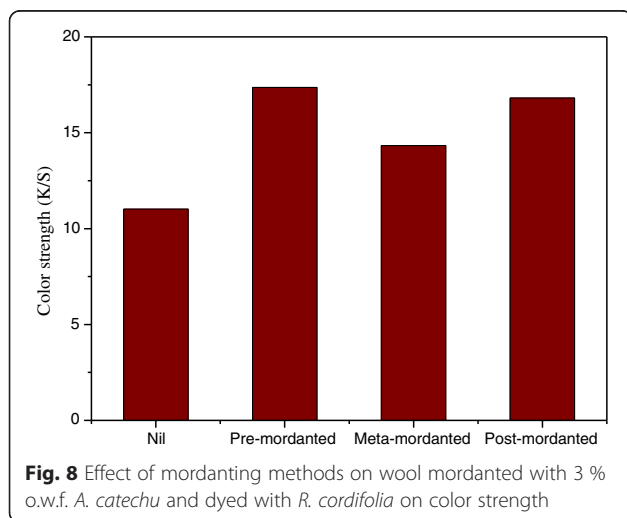
strength ( $K/S$ ), respectively. It can be observed that there is a gradual increase in % dye exhaustion and color strength ( $K/S$ ) of pre-mordanted wool yarn samples when the concentration of *A. catechu* reached to 3 % o.w.f., and thereafter, a little increment occurred. Thus, 3 % biomordant is believed to be the optimized mordant concentration for subsequent dyeing experiments with *R. cordifolia* natural dye.

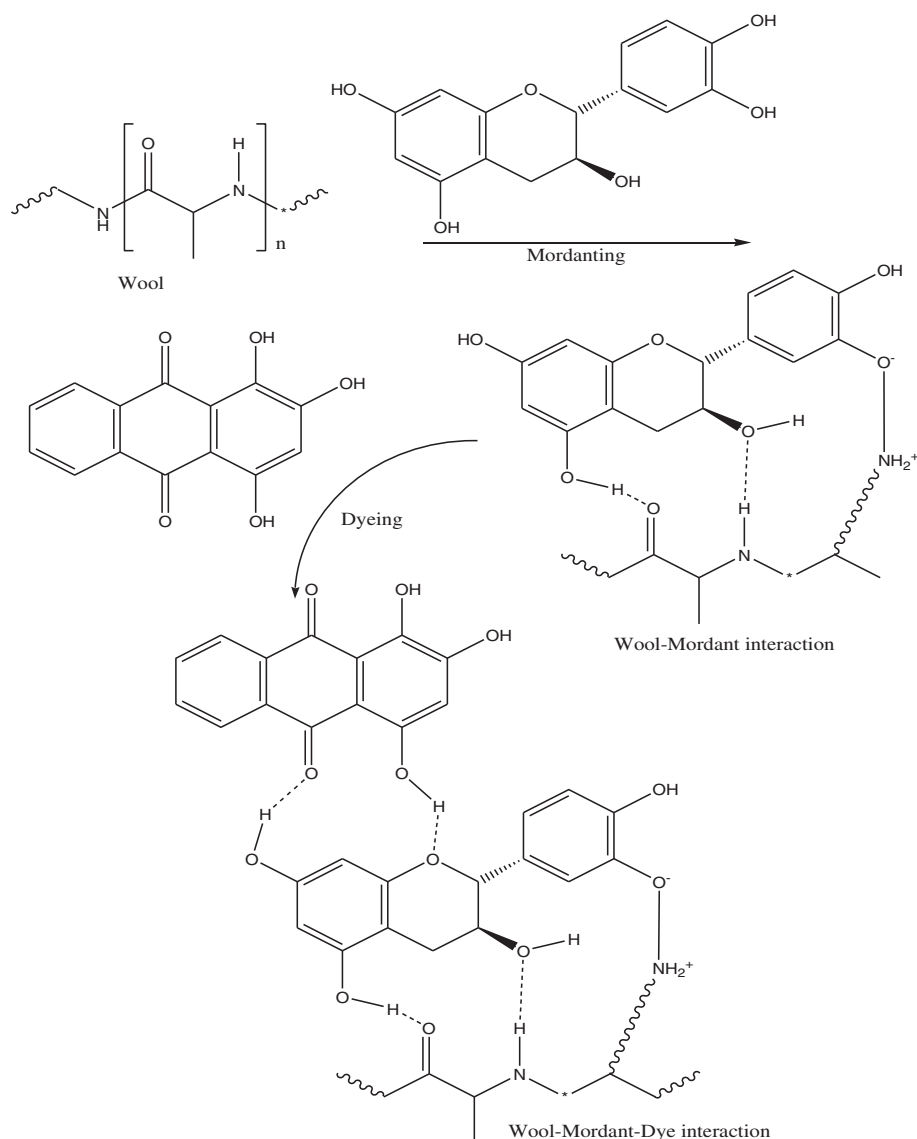
#### Scanning electron microscopy analysis

The morphological features of wool fibers are depicted in Fig. 6, and it can be seen clearly that a wool fiber consists of scaled and fibrillar structures (Fig. 6a). Figure 6b, c represents the surface of mordanted and dyed wool fibers which were seen without any physical changes such as cracks on surface, responsible for alteration of wool fiber properties.

#### Color characteristics

Colorimetric characteristics (CIEL\*a\*b\* and  $K/S$  values) of wool yarn mordanted with *A. catechu* and dyed with *R. cordifolia* were analyzed and presented in Tables 1 and 2. Orange red and dark brown red shades are obtained by *R. cordifolia* dye of different hue and tones, with or without mordant (*A. catechu*). A drastic decrease in lightness values is observed in the case of biomordanted samples in comparison to the control dyed samples (Table 3). All the dyed wool samples appeared in red-yellow zone of CIEL\*a\*b\* color space (Fig. 7).





**Fig. 9** Schematic representation of probable dyeing mechanism

### Effect of mordanting methods

In comparison to the control dyed wool yarn, *A. catechu*-mordanted, dyed samples were found to be darker. It was found that the darkening effect reached to maximum in the case of pre-mordanting method in comparison to the post- and meta-mordanting methods. From Fig. 8, it can be seen that the trend of color strength is found to be decreasing from *A. catechu* pre-mordanted to *A. catechu* post-mordanted and *A. catechu* meta-mordanted. Meta-mordanted dyed wool samples have the lowest color strength owing to the interaction of dye and biomordant molecules in dye bath solution and low amount of dye interacts to fiber.  $a^*-b^*$  plot shows shifting of the coordinates towards the red-green axis in all cases of mordanting methods.

### Color fastness properties

Tables 1 and 2 show the fastness properties of control and mordanted dyed with *R. cordifolia* wool samples. It can be seen from the data that mordanting markedly enhances the overall fastness properties, and the best results are observed in the pre-mordanting ones to a significant extent that are commercially acceptable.

Wool fiber is a polypeptide and has many active sites in which a dye can bind to the fiber. It may form covalent or ionic bonding to the  $\text{---NH}_2$  and  $\text{---COOH}$  groups on the ends of the polymer or form similar interactions with the amino acid side chains resulting in significant colorimetric and fastness properties. The chemistry of bonding of dyes to fiber is very complex. In fact, bonding occurs in different ways such as direct bonding

(covalent), H-bonding, and hydrophobic interactions (van der Waals forces). Mordants help in binding of dyes to fiber by forming a chemical bridge between the dye and the fiber, thus improving the substantivity of a dye along with increase in fabric's color fastness properties and color depth as well (Fig. 9). Recently, Vankar et al. (2008) observed that *Eurya acuminata* when used as a mordant in conjunction with *R. cordifolia* dye was found to enhance the dyeability of silk fabric by chelation due to the high Al content present in its leaves. In another study, Cu-hyperaccumulating plant *Pyrus pashia* has been reported to provide better dye adherence due to stronger and useful chelation to the flavone/flavonol dyes extracted from *Delonix regia* (Vankar and Shanker 2009). Mansour and Heffernan (2011) have found that being a tannin-rich species, *A. catechu* is an instrumental tool in the enhancement of dyeability of dye used for textile fabric. In addition, the use of plants as a natural green alternative to metallic mordants is a very promising concept which may help to reduce the enormous environmental risks associated with metal salt mordants.

## Conclusions

This study evaluated the effect of color and fastness properties of wool fibers dyed with *R. cordifolia* as a natural dye and *A. catechu* as an anchoring agent. The colorimetric (CIE L\*a\*b\*) and fastness properties were considerably improved using *A. catechu* as a biomordant and *R. cordifolia* as a natural dye which may be profitably acceptable in industrial and commercial spheres. The pre-mordanting method found more overdriven effects than the meta-mordanting and post-mordanting methods overall. In order to obtain more eco-friendly and biocompatible dyeing, the current study demonstrated a cleaner approach by using *A. catechu* as a biomordant to minimize the pollution with substitution of metallic salts to fulfill the demand of the world in contemporary scenario.

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## Authors' contributions

MY and FM designed the experiment. MY conducted all of the experimental procedures and interpreted the tentative data. MS drafted the manuscript, and MAK provided technical help. All authors read and approved the final manuscript submission.

## Competing interests

The authors declare that they have no competing interests.

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