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Investigation of pH influence on madder-dyed cotton blends

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The study aimed to optimise the dyeing pH of Banana/Cotton and Bamboo/Cotton blended fabrics using natural madder dye. In this study, eight fabric samples are dyed under varying pH conditions (pH 7–10) using madder dye extracted through an aqueous process. *Punica granatum* peel is employed as a natural mordanting agent for all samples. The Banana/Cotton blend exhibits the highest colour strength at pH 9 (K/S = 24), while the Bamboo/Cotton blend shows maximum colour strength at pH 10 (K/S = 26). SEM analysis of the optimised dyed samples reveals distinct surface modifications, while FTIR spectra confirm chemical changes among raw, mordanted, and dyed fabrics. Colour fastness to rubbing, washing, and light exposure is found to range from good to excellent. All madder-dyed fabrics demonstrate higher antimicrobial activity than their undyed counterparts, with the Bamboo/Cotton blend exhibiting superior antimicrobial performance compared with the Banana/Cotton blend.

Keywords: Antimicrobial activity, Banana/Cotton, Bamboo/Cotton, Colour strength, Colour fastness, Madder dye

1 Introduction

Growing awareness about sustainability practices in the textile and clothing industry and the fatal implications of synthetic dyes and the items they colour resulted in the revival of natural dyes to a more considerable extent[1]. Natural dyes have no risks; however, synthetic dyes produce a large amount of colour and chemical waste. The causes of hazards to human health and the environment have been contextualised[2]. The world's tremendous biodiversity comprises a wide range of temperate zones and altitudes and a diverse range of animals and flowers that give rise to various vibrant shrubs. Many new plant sources have been researched for natural dyeing, some well-known medicinal plants with a wealth of natural colour potential[3]. The colourants produced from the roots of several plant species in the Rubia genus have been used as dyes for ages and are commonly referred to as madder[4]. Madder is a perennial plant that grows throughout the summer. The upper sections of the plant die throughout the winter, and new twigs emerge from the twig roots in the spring. The plant grows to a maximum height of around 1.5 m. Madder plants produce 150 to 200 kg of dyestuff per acre[5]. Robiquet & Colin observed that *R. tinctorum* root contained two colourants, such as alizarin and purpurin[6]. The peel of pomegranates, one of the oldest fruits, has also been used as a colouring and mordanting agent. Because of its high tannin concentration (19-26 %), this dye works especially well for colouring Cotton, silk, wool, and other natural fibres[7]. Many investigations were done on madder on different fibres with different fibre conductions to improve the dyeing performance. Mozhgan *et al*. investigated the madder dye with wool yarn in the presence of two combinations of agro-based bio-mordants, walnut husk (WH) and oleaster peel. It was observed that combining two mordants resulted in high colour strength and colour fastness values[8]. Nusrat and Datta dyed the madder dye on Cotton and silk using different mordants to investigate the colour effects. Pre-mordanting and post-mordanting processes were followed. They found that silk fabric absorbed more colour than Cotton, and pre-mordanted fabric has yielded a deeper tone than the non-mordanted fabric samples[9]. Yue *et al*. pre-treated Cotton fabric with cetylpyridinium chloride, a citric

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acid and succinic acid combination, then coloured it with madder dye to improve colour fastness and antibacterial activity. It was reported that the pre-treated Cotton fabric had great colour depth and fastness attributes. Furthermore, the antibacterial activity and UV protection factor have been dramatically improved[10]. Amutha *et al*. coloured the Banana/Cotton blend cloth using natural dye derived from onion skin and pomegranate rind. It was observed that using pomegranate rind, the mordant significantly increased the colour strength of the fabric[11]. Kaur *et al*. conducted research on colouring Bamboo with green tea extract. The dyeing process was carried out with and without a mordanting condition. The major dyeing variables were compared, such as dye exhaustion, colour depth in terms of K/S, washing fastness, and light fastness. It was observed that the pre-mordanted sample generated a dark brown tone and had good colour fastness qualities[12]. It was also stated that a few colouration processes were carried out on Bamboo and Banana fibre using natural dye sources. There were a few attempts to colour Banana, Bamboo, and their blended materials with a madder-based dye source. This research attempts to execute the madder dye on Banana and Bamboo blends with Cotton fabrics. This work is also aimed at investigating the antimicrobial and colour-fastness properties of dyed fabric.

2 Materials and Methods

The materials used in this study include the peel of *Punica granatum* (pomegranate), procured from a local farm in Coimbatore, India, and the root of *Rubia tinctorum L.* (madder), collected from the field. *P. granatum* peel served as a natural mordant, while the madder root was used as a natural dye source.

2.1 Extraction Process

The collected *P. granatum* peels were shade-dried for 20 days until they became hard and brown. The dried peels were then ground and sieved to obtain a fine powder (Fig. 1). Since *P. granatum* peel possesses inherent mordanting properties, no additional mordant was required for the dyeing process.

The collected *R. tinctorum* roots were shade-dried, ground, and sieved to obtain a fine powder (Fig. 2). For dye extraction, 500 g of madder powder was soaked in 1000 mL of distilled water (pH 7) and heated at 95 °C for 30 min. Subsequently, it was cooked, filtered, dried, and concentrated to obtain a 50 % dye solution, which was used for dyeing[13].

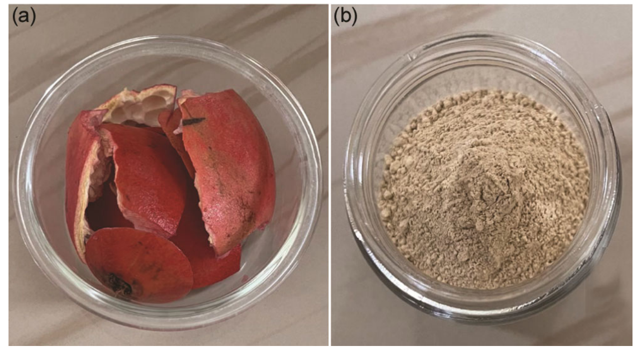


Fig. 1 — *P. granatum* (a) peel and (b) powder

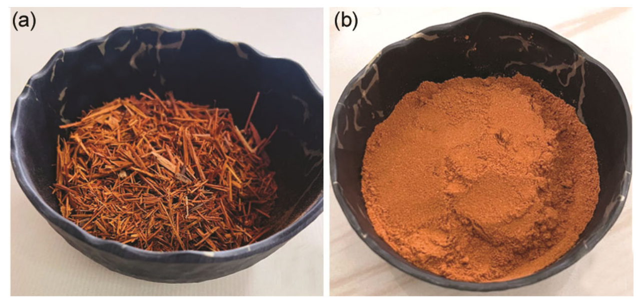


Fig. 2 — *R. tinctorum* (a) root and (b) powder

2.2 Selection of Fabrics

Two fabric blends—Banana/Cotton and Bamboo/Cotton—were selected for natural dyeing. Banana/Cotton fabric specifications were warp and weft count 22 Ne x 22 Ne, 40 EPI and 22 PPI, and 175 GSM. Similarly, the Bamboo/Cotton fabric specifications were a warp and weft count of 40 Ne x 40 Ne, 40 EPI, 22 PPI, and 150 GSM.

2.3 Pre-Treatment Process

Pre-treatment was carried out to remove impurities such as waxes, oils, and natural gums, improving fibre wettability and dye uptake. Scouring was performed using 3 % NaOH (v/w) and 3 % Na_2CO_3 (v/w) at 100 °C for 60 min[14]. The bleaching process was carried out with H_2O_2 at 100 °C for 60 min[15].

2.4 Pre-Mordanting

The pre-treated Banana/Cotton and Bamboo/Cotton fabrics were mordanted using *P. granatum* peel extract at a concentration of 10 % (v/w). Mordanting was performed at 95 °C for 40 min. The treated samples were dried and cold-washed to remove the non-bonded surface particles so that the dyeing process could be done effectively.

2.5 Dye Application Method

Dyeing was carried out on Bamboo/Cotton and Banana/Cotton fabrics using the extracted madder dye under four pH conditions (7, 8, 9, and 10). The dye concentration was maintained at 50 % (v/w) with a material-to-liquor ratio (MLR) of 1:20. The pH was adjusted using 1–3 % oxalic acid The dyeing process was carried out at temperatures of 45 °C to 95 °C for 30 min, and the dyeing process was continued for 60 min at 95 °C. After dyeing, the samples were cold-washed and rinsed thoroughly[13]. The dyed fabric samples are shown in Fig. 3, and the experimental plan is detailed in Table 1.

2.6 Characterisation

2.6.1 Colour Strength

The colour strength (K/S value) of the dyed fabric samples was determined using the Kubelka-Munk equation[3]:

$$K/S=(1-R^2)/2R \quad \ldots(1)$$

where K represents the absorption coefficient of the dye; S, scattering coefficient of the substrate; and R, reflectance.

A Texflash spectrophotometer (Datacolor Co., Switzerland) was used to measure the CIE L*, a*, b*, C*, and h° colour coordinates, as well as the samples' reflectance spectra. All colour properties were tested under illuminant D65 using the CIE 1964 10 °C standard observer, except the specular component. The colour strength was measured in the wavelength range of 400 to 700 nm. The colour coordinates are defined as follows:

L* = Lightness (0 = black, 100 = white)

a* = Red-Green axis (Positive a* = red tones; Negative a* = green tones)

b* = Yellow-Blue axis (Positive a* = yellow tones ; Negative a* = blue tones)

C* = Chroma

h° = Hue angle

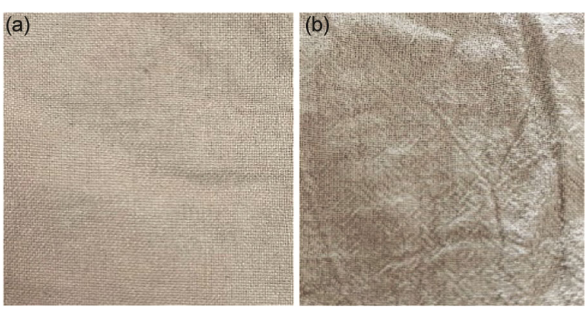


Fig. 3 — Dyed (a) Banana/Cotton and (b) Bamboo/Cotton fabric samples

2.6.2 Colour Fastness

The dyed samples were washed with 5 g/L soap at 40 °C for 30 min before executing this colour fastness test. The samples' wash, light, and rubbing fastness were determined using the ISO 105-C06: 1994 (E), ISO 105-B02:1994 (E), and IS766L1988 standards. The samples were exposed to an air-cooled xenon arc lamp for light fastness for 24 h. Wash fastness was evaluated based on the degree of colour change and staining. Similarly, rubbing fastness determines the amount of colour that can be transferred from the surface of coloured fabrics to other surfaces when rubbed.

2.6.3 FTIR

Fourier Transform Infrared (FTIR) spectroscopy was used to identify the functional groups present in the treated and untreated fabrics. FTIR and Attenuated Total Reflection mode (ATR) (Nicolet 6700) were used within the wavenumber range of 400–4000 cm^{-1} at a resolution of 4.0 cm^{-1}. The following samples were analysed: undyed Banana/ Cotton, undyed Bamboo/Cotton, mordanted Banana/ Cotton, mordanted Bamboo/Cotton, madder-dyed Banana/Cotton, and madder-dyed Bamboo/Cotton fabrics. The spectra were compared to assess chemical modifications due to mordanting and dyeing processes.

2.6.4 Antimicrobial Activity

The antimicrobial activity of the dyed and control fabrics was evaluated using the AATCC 147 method.

Table 1 — Colorimetric data of dyed Banana/Cotton and Bamboo/Cotton fabrics under different pH conditions

Sample code	Fabric	pH	L*	a*	b*	c*	K/S at λ_{max}
S1	Banana/Cotton	7	17.32	12.56	5.34	14.32	20
S2	Banana/Cotton	8	19.53	17.33	8.34	20.23	22
S3	Banana/Cotton	9	21.78	20.43	12.54	25.34	24
S4	Banana/Cotton	10	20.23	17.43	10.43	17.54	21
S5	Bamboo/Cotton	7	18.34	10.43	8.12	14.12	21.2
S6	Bamboo/Cotton	8	19.12	11.20	8.20	14.04	24.3
S7	Bamboo/Cotton	9	20.33	19.43	10.23	19.32	23.4
S8	Bamboo/Cotton	10	22.78	22.12	14.23	25.23	25.5

The test was performed against *Staphylococcus aureus* and *Escherichia coli* through the agar diffusion test method. Approximately 500 µL of each bacterial suspension (1 × 10^8 CFU/mL) was swabbed evenly over the agar surface. The test fabric samples were then placed on the inoculated agar plates and incubated at 37 °C for 24-48 h. After incubation, the zone of inhibition (L) was measured using the following equation[16]:

$$L=(A-D)/2 \quad \ldots (2)$$

where L is the zone of inhibition; D, diameter of the control sample before incubation; and A, diameter of the tested samples with inhibition zone diameter.

3 Results and Discussion

3.1 Colour Strength

The Banana/Cotton and Bamboo blended samples are dyed with *P. granatum* mordanted madder dye under four different pH conditions. The corresponding results are presented in Table 1 and Fig. 4.

Table 1 shows that the colour parameters (a*, b*, and C*) of all dyed samples fall within the first quarter of the CIE L*a*b* colour space, representing reddish-brown hues. Samples S3 and S8 exhibit higher chroma (C*) values, yielding a brownish-red tone, while other samples produce relatively duller brown shades. The samples with lower pH conditions (S1, S2, S5 & S6) have shown lower lightness than others. It is shown in Fig. 4 that the colour strength (K/S) increases progressively under alkaline conditions (pH 9–10), even when other parameters remain constant. The K/S was measured at the maximum wavelength (λ_{max}) value of 400 nm. The Banana/Cotton blend achieves the highest K/S value (24) at pH 9, while the Bamboo/Cotton blend records its maximum (26) at pH 10. This trend indicates that alkaline conditions enhance dye solubility and interaction with the fibre, consistent with the behaviour of purpurin-based colouring compounds that exhibit improved solubility and affinity in alkaline media[17].

3.2 FTIR Analysis

The FTIR spectra confirm the structural and chemical modifications occurring in the fibre after mordanting and dyeing. Figure 5 presents the FTIR spectra of untreated, *P. granatum*-treated, and madder-dyed Bamboo/Cotton fabrics.

The characteristic broad absorption band observed between 3657–3834 cm^{-1} corresponds to O–H stretching vibrations from cellulose hydroxyl groups. The peak at 2916 cm^{-1} is assigned to asymmetric and symmetric C–H stretching, while the bands at 1435 and 1327 cm^{-1} correspond to C–H and O–H bending of cellulose[18]. In comparison to the untreated sample [Fig. 5 (a)], the mordanted [Fig. 5 (b)] and dyed [Fig. 5 (c)] fabrics show noticeable peak shifts and intensity variations. The reduction in O–H stretching intensity at ~3600 cm^{-1} and its shift towards higher wavenumbers (3826 cm^{-1} and 3757 cm^{-1}) indicates hydrogen bonding interactions involving hydroxyl and phenolic groups from *P. granatum* and madder dye. The increased band intensity at 1435 cm^{-1} in the mordanted sample further supports the formation of new O–H and N–H linkages[19].

Similarly, Fig. 6 shows the FTIR spectra of untreated, pomegranate-treated, and dyed Banana/ Cotton fabrics. The spectra show broad O–H

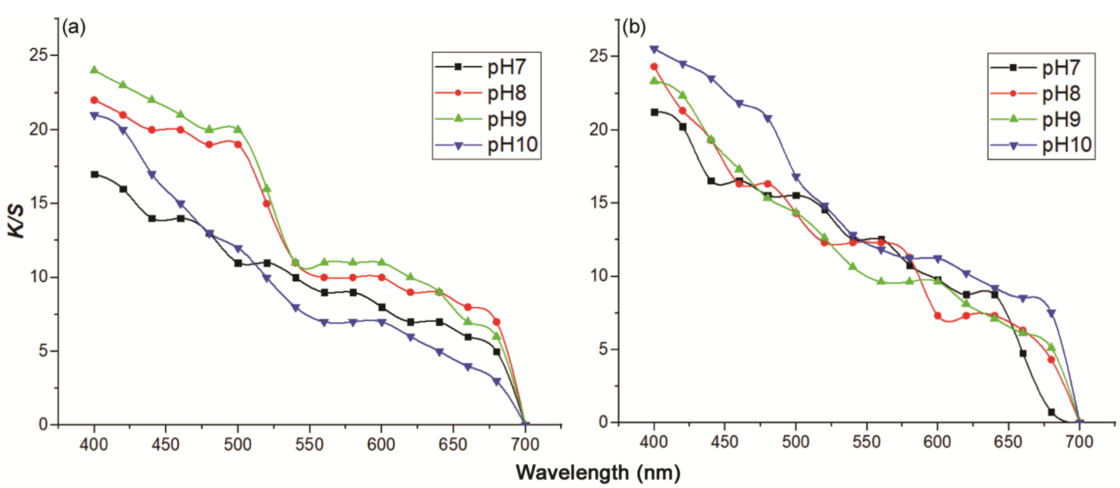


Fig. 4 — Colour strength of the (a) Banana/Cotton and (b) Bamboo/Cotton dyed samples under different pH

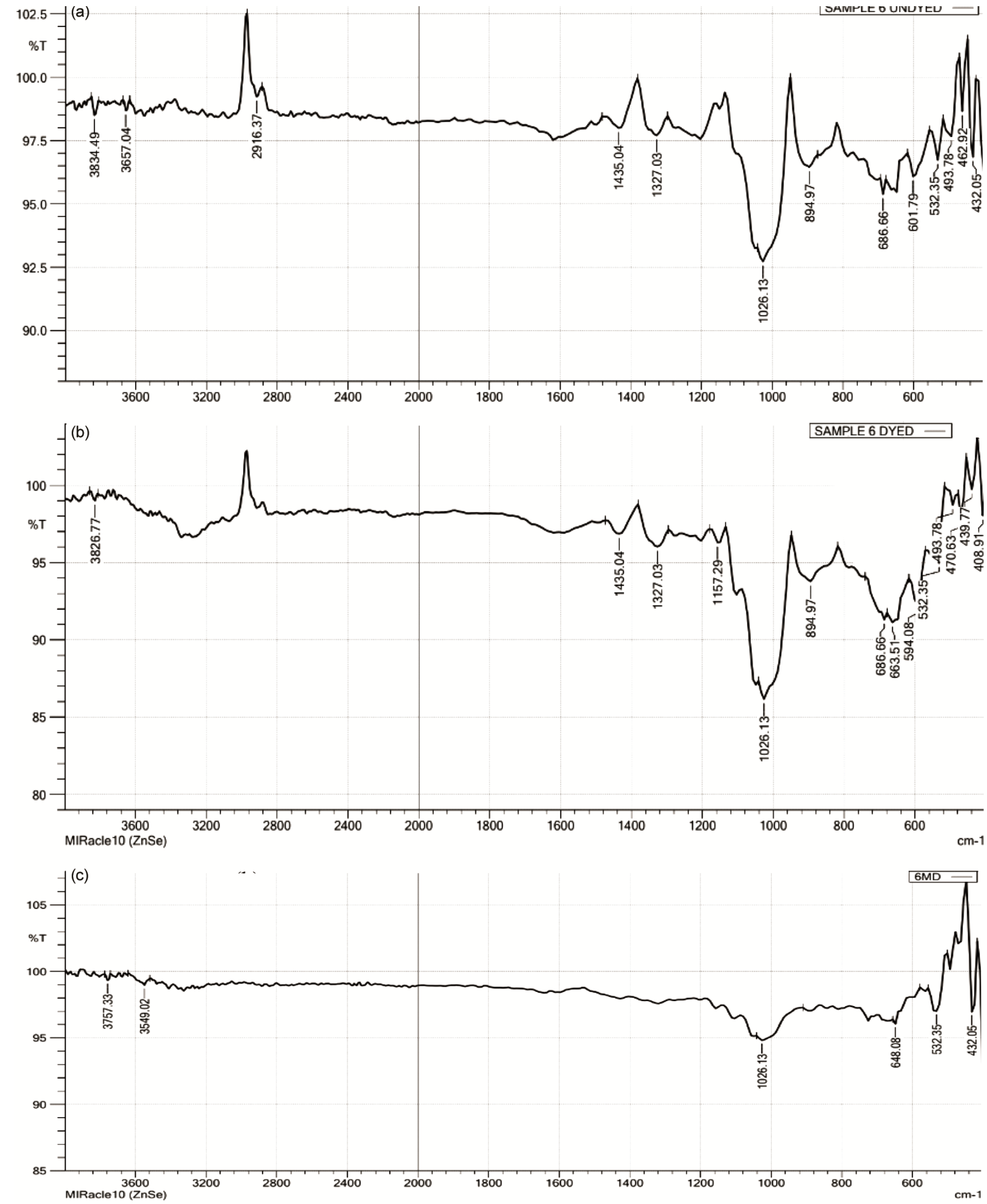


Fig. 5 — FTIR spectra of Bamboo/Cotton fabrics: (a) untreated, (b) *P. granatum*-treated, and (c) madder-dyed

stretching bands at 3749–3649 cm^{-1} and characteristic cellulose bands at 1620 and 1422 cm^{-1}. The treated samples exhibit distinct variations in band shape and intensity, particularly at 1026 cm^{-1} (shifted to 1018 cm^{-1} after mordanting), suggesting the incorporation of phenolic and tannin-based groups from *P. granatum*[19]. These spectral changes confirm the formation of hydrogen and covalent bonds between dye–mordant–fibre systems, enhancing dye fixation and stability.

3.3 Colour Fastness

The colour fastness properties of the dyed Banana/Cotton and Bamboo/Cotton samples against light, rubbing, and washing are summarised in Table 2. Samples S3 and S8 show excellent wash fastness, graded 4–5, while the remaining samples

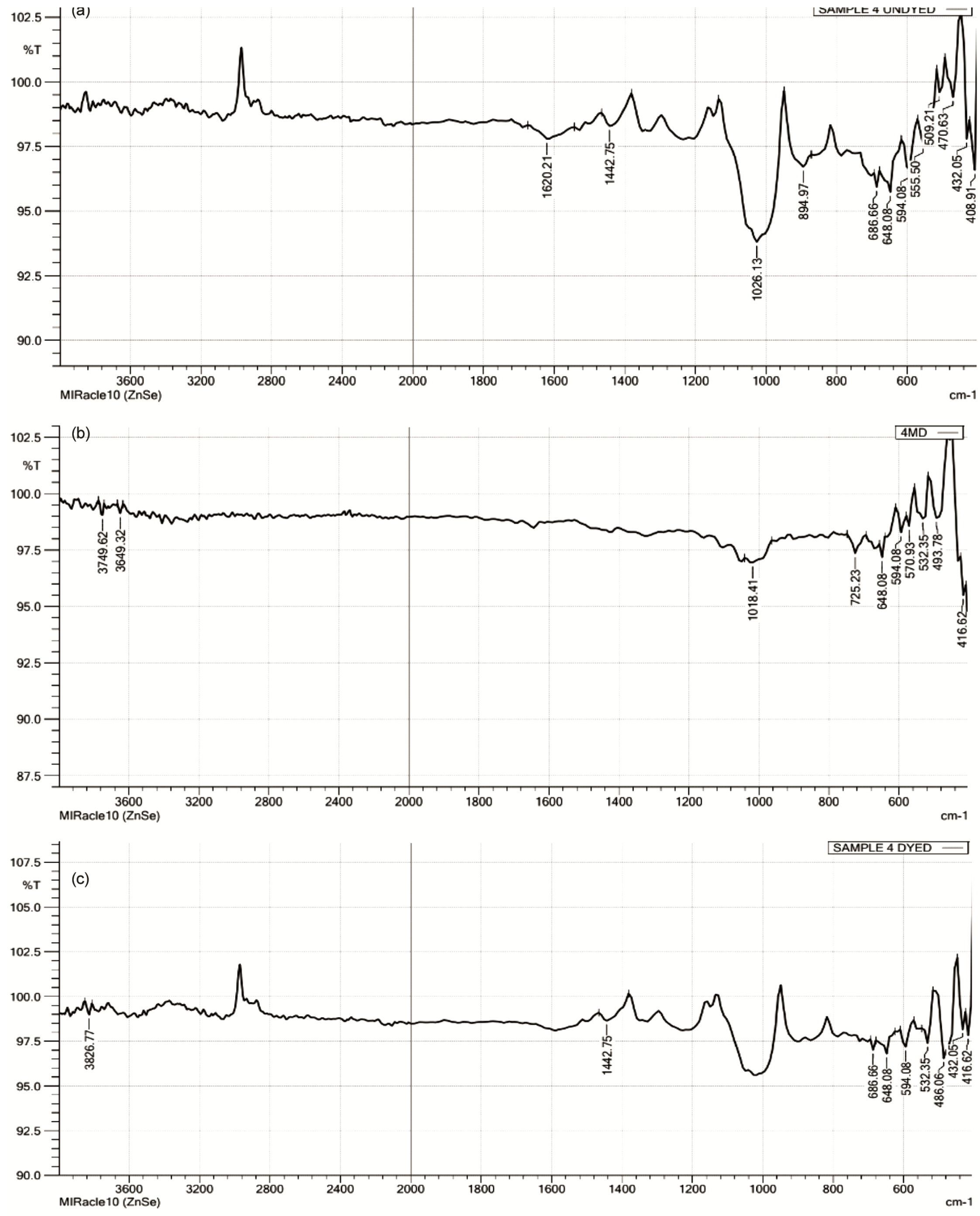


Fig. 6 — FTIR spectra of Banana/Cotton fabrics: (a) untreated, (b) *P. granatum*-treated, and (c) madder-dyed

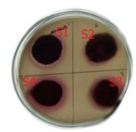
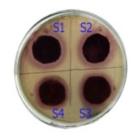
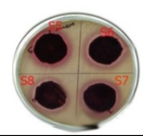
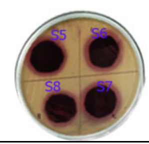
show slightly fair to moderate ratings (2–3 to 4) in both colour change (CC) and colour staining (CS) modes. The rubbing fastness under CC mode is found very good to excellent (4–5) for S3, S7, and S8. Similarly, S2, S3, S5, S6, and S8 also display high performance under CS mode. The light fastness results show that S3 and S8 achieve the highest rating (7, excellent), while other samples range between 5 and 6 (good to very good). The enhanced fastness properties under alkaline conditions (pH 9–10) can be attributed to improved dye–fibre bonding. As both Bamboo and Banana fibres are cellulosic, their hydroxyl groups can form strong covalent or ionic linkages with the dye molecules, leading to better dye fixation and resistance to washing and rubbing[20].

Table 2 — Fastness grades of dyed Banana/Cotton and Bamboo/Cotton fabrics under different pH conditions

Sample code	Light fastness	Rubbing fastness		Washing fastness	
	CC	CC	CS	CC	CS
S1	5	3-4	4	2-3	4
S2	6	2-3	4-5	3	4
S3	7	4-5	4-5	4-5	4
S4	6	3-4	3	4	5
S5	6	3-4	4-5	2-3	4
S6	6	3-4	4-5	3	4
S7	6	4-5	4-5	3-4	5
S8	7	4-5	4-5	4-5	5

CC- colour change; CS - colour staining

Table 3 — Zone of inhibition (ZOI) of dyed Banana/Cotton and Bamboo/Cotton fabrics against different microbes

Sample code	ZOI (mm)		Images	
	S. aureus	*E. coli*	*S. aureus*	*E. coli*
S1	0.9	0.8		
S2	1.1	0.9		
S3	1.3	1.3		
S4	1.2	1.1		
S5	1.3	1.3		
S6	1.4	1.3		
S7	1.4	1.3		
S8	1.6	1.5		

3.4 Antibacterial Activity

The antibacterial activity of the madder-dyed fabrics is shown in Table 3. All dyed samples exhibit higher antibacterial activity compared to their undyed counterparts, with an average inhibition zone of approximately 0.3 mm. This was reconfirmed with the previous finding of madder dyed fabrics[21,22.] It is further noted that the Bamboo/Cotton fabric possesses higher antibacterial activity than the Banana/Cotton fabric because of the higher inherent antimicrobial activity of Bamboo fabric[23,24.] Among the Banana/ Cotton fabrics, sample S3 demonstrates the maximum inhibition zone of 1.3 mm against both *S. aureus* and *E. coli*. Similarly, sample S8 of Bamboo/Cotton shows the highest inhibition zones of 1.5 mm and 1.4 mm against *S. aureus* and *E. coli*, respectively. The antibacterial performance correlates positively with the K/S values, suggesting that higher dye concentration on the fabric surface enhances antimicrobial efficiency. These results are correlated with the previous findings of Faisal *et al*.[25].

4 Conclusion

The study confirms that Banana/Cotton and Bamboo/Cotton blends achieve effective and sustainable dyeing with pomegranate-mordanted madder under alkaline conditions. The Banana/Cotton fabric shows maximum colour strength at pH 9, while Bamboo/Cotton performs best at pH 10. FTIR spectra indicate successful bonding between the dye–mordant complex and fibre hydroxyl groups. The dyed fabrics exhibit good to excellent fastness to washing, rubbing, and light. All samples show enhanced antibacterial activity, with Bamboo/Cotton demonstrating superior inhibition due to its inherent antimicrobial nature. The results highlight that the madder dyed Banana/Cotton and Bamboo/Cotton fabrics may have practical utility as antibacterial textiles for medical applications such as gowns, sutures, implantable goods, etc.

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