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Dyeing of Cotton Fabric Using *Beta vulgaris* Peel Extracts and Bio-Mordants for Enhanced Ultraviolet Protection and Antioxidant Activity—A Comparative Approach

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Highlights

What are the main findings?

- Beetroot peel waste is valorized as a natural dye for cotton using four food-waste bio-mordants.
- Three extraction solvents and three dye concentrations were systematically compared on the same substrate.
- Color strength, fastness, antioxidant activity, and UV protection are integrated into one framework.

What is the implication of the main finding?

- Bio-mordants can replace toxic metal salts, reducing effluent hazards. Mordant choice depends on target property; no single combination optimizes all the properties.
- Beetroot peel dyeing yields colored, UV-protective, antioxidant cotton from agro-waste.

Abstract

Natural dyes extracted from agricultural waste are increasingly valued due to their biodegradability and minimal ecological risks. This study explores the extraction of dyes from *Beta vulgaris* (beetroot) peels in different media (water, ethanol, and methanol) using a Soxhlet apparatus. The extracted dyes were applied to cotton fabric at 2%, 3%, and 4% concentrations on the weight of fabric through the exhaust method at a liquor ratio of 1:30, maintaining a temperature of 90 °C for 90 min, following a simultaneous mordanting technique using orange peel, lemon peel, *Terminalia chebula* (myrobalan), and gum acacia as bio-mordants. The dyed fabrics were assessed for color strength (K/S) at maximum wavelength (λ_{max} = 537 nm) and colorfastness properties, including washing, light, rubbing, and perspiration fastness, following standard testing protocols. The antioxidant and ultraviolet protection properties were established to determine the functional performance of the dyed fabrics. The K/S values obtained on cotton fabric with the extracts in organic solvents (methanol and ethanol) were higher than the K/S values obtained with the aqueous extract. The maximum K/S value was 4.57 for cotton dyed with 4% methanolic beetroot extract using orange peel as a bio-mordant, followed by 4.44 for the corresponding ethanolic extract; the maximum K/S value obtained with the aqueous extract was 2.81. The mordant type

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also has an influence on the color strength and colorfastness performance. Washing fastness ratings ranged from 3–4 to 4–5 on the gray scale, dry rubbing fastness ranged from 3–4 to 4–5, and light fastness ranged from 2–3 to 4–5 on the blue wool scale, depending on extraction medium, dye concentration, and bio-mordant. The samples dyed in the presence of bio-mordants, particularly orange peel and lemon peels, rendered excellent ultraviolet protection and antioxidant activities, followed by myrobalan and then gum acacia. The maximum ABTS radical inhibition was 88.20 ± 8.5%, and the maximum ultraviolet protection factor (UPF) was 88; both were recorded for cotton dyed with 4% ethanolic beetroot extract using orange peel as a bio-mordant.

Keywords: natural mordant; natural dye; colorfastness; *Beta vulgaris*; myrobalan; lemon peel; orange peel

1. Introduction

The textile materials are mostly dyed with synthetic dyes, which pose serious environmental threats because the effluents from dyehouses are often toxic, mutagenic, and potentially carcinogenic. These synthetic dyes also decrease light transmission and the oxygen content in the receiving water and consequently affect the aquatic ecosystem [1–4]. In recent years, agro-waste utilization has gained researchers' attention for valorization as a natural dye in the textile dyeing and finishing industry [5–7]. Agro-based and food processing wastes can still contain pigments and other components that can be extracted and applied to dye textiles. Singhee discussed the utilization of agricultural and industrial wastes as natural dyes, as this approach can help in reducing waste disposal and also can be used as an alternative raw material for dyes [8]. Natural textile dyes are relevant to sustainable textile development as they can provide alternatives to conventional colorants and promote the utilization of renewable biological resources. Sutlović et al. explored the contribution of natural dyes to textile creativity and sustainability, which gives a broader context to their ongoing research in the modern application of natural dyes in textiles. In this respect, the conversion of beetroot peel waste into a colorant is a good alternative that not only utilizes waste in the production of colored cotton fabric but also provides functional fabric [9]. Natural dyes are being rediscovered as sustainable alternatives to their synthetic counterparts [10]. Unlike synthetic dyes, which generate environmental hazards and health concerns, natural dyes are biodegradable, renewable, and non-toxic [11]. They carry an additional richness, and many contain vitamins, fibers, and bioactive compounds that impart antimicrobial, antibacterial, and even UV-protective properties to textiles [12]. Remarkably, some natural colors deepen and improve with age, adding a timeless quality to fabrics [13]. Yet, despite these advantages, natural dyes were overshadowed in the past century due to lower availability, limited colors, and relatively poor colorfastness in comparison to synthetic dyes [14].

The properties and structure of the cellulose substrate can be changed by scouring, bleaching, and mercerization, and therefore can have a profound impact on the degree of colouration that can be produced using natural dyes for cotton. Botteri et al. studied cotton dyeing with onion and pomegranate peel extract and concluded that color appearance and fastness performance were influenced by the method of pretreatment, and mercerization pretreatment helped in the absorption and bonding of the dyestuff. These results indicate that the substrate preparation must be taken into consideration while evaluating the dyeing performance of natural colorants [15]. Čorak et al. studied the interaction of cotton and cochineal dye, and the effect on dyeing performance by modification of cotton, mordanting, and addition of electrolyte. Their results show that the properties of the textile

material and dyeing system should be considered simultaneously while devising natural dyeing processes [16]. A research group explored the coloration of cotton with thyme and pomegranate peels and established their functional properties [17]. Bouaziz et al. have successfully demonstrated the use of pomegranate peel extract as a dye for polyamide fabrics, and they recorded good washing and rubbing fastness properties, in addition to antibacterial activity. Their results show the potential of natural dyes extracted from waste materials as a source of color and functional effects for textile materials [18]. Recently, neem wood waste was utilized for the biofunctionalization and coloration of jute fabric [19]. Wool dyeing with henna and yarrow was also explored and optimized to achieve better colorfastness results [20]. The performance of natural dyes is influenced by regulating the dyeing conditions such as concentration of dyestuff, pH of dye baths, addition of electrolyte, and mordanting. The influence of these variables in the dyeing of cotton by cochineal colorant has been investigated by Sutlović et al., who showed that the dyeing conditions and pretreatment had an impact on the achieved shades and colorfastness [21]. *Candelariella reflexa* was applied as a sustainable colorant for the dyeing of mohair fibers [22].

Rising environmental awareness about eco-friendly textiles has renewed global interest in plant-based colorants. Among these, *Beta vulgaris* (beetroot) stands out as a vibrant, underutilized dye source [23]. Beetroot produces striking red shades due to its red violet betacyanin and yellow-orange betaxanthins pigments and provides an excellent way to valorize agricultural waste. These functional compounds render potent antioxidant and antimicrobial activities [24–27]. This plant represents promising, sustainable dyeing options that can merge color performance with ecological responsibility [28]. Since natural dyes needs mordants which facilitate binding with fibers polymeric system and most of the researchers utilize heavy metal salts posing serious environmental threats and effluent loads. To address this challenge, bio-mordants have the potential to serve as mordants and facilitate the dye–fiber binding mechanism. Orange peels, lemon peels, myrobalan, and gum acacia are natural mordants that can be applied in natural dyeing [29].

There have been several recent studies investigating the use of beetroot as a colorant for textiles, but each only covers a fraction of the solvent–mordant–substrate space needed for its industrial use. Mia et al. [19] have evaluated bio-mordants, such as orange peel and lemon peels, in natural dyeing of cotton. Rotich et al. [30] dyed cellulosic fabric with beetroot peel/pomace dye and compared the natural mordants (alum, tannic acid) to the metallic mordants (potassium dichromate, ferrous sulfate, copper sulfate. Al-Amir et al. [31] utilized beetroot waste for dyeing cotton, wool, and silk, but only one aqueous extraction was used without adding any mordant. Hudu et al. [25] have established the dyeing and antimicrobial activity of beetroot colorant on cotton; however, they did not study the colorant in other extraction media and bio-mordants. None of the above studies have investigated both the extraction solvent and bio-mordant type in combination on the same substrate and assessed the functional response (e.g., antioxidant activity and UV protection) apart from color strength and colorfastness.

This study addresses that specific gap, which is a systematic paired comparison between three different extraction solvents, namely aqueous, ethanol, and methanol, and four bio-mordants, namely orange peel, lemon peel, myrobalan, and gum acacia, performed on a single cotton substrate along with the functional characterization (antioxidant activity, UV protection) and the standard colorfastness testing. Such an integrated solvent–bio-mordant–functional performance matrix has not been reported for the cotton system dyed with beetroot dye and is proposed to be helpful for practical solvent and mordant selection for sustainable cotton coloration and not further proof of concept.

2. Materials and Methods

2.1. Materials

The fabric used in this study was a 100% cotton (plain weave) purchased from Sapphire Textile Mills Limited (Lahore, Pakistan), Pakistan (130 GSM, 76 ends per inch, 75 picks per inch). The fabric was pretreated by a normal treatment process prior to dyeing. Scouring was performed using 4 g/L sodium hydroxide (NaOH) and 2 g/L non-ionic wetting agent at 95 °C for 60 min at a liquor ratio of 1:20. Bleaching was carried out using 5 mL/L hydrogen peroxide (H_2O_2, 35%), 2 g/L sodium silicate, and 1 g/L NaOH at 90 °C for 90 min at a liquor ratio of 1:15. After bleaching, the fabric was rinsed with hot water and then neutralized in an aqueous acetic acid solution (1 g/L) at 50 °C for 20 min. It was subsequently rinsed thoroughly with distilled water until the rinse water reached near-neutral pH (approximately 6.5–7.0). After each treatment, the fabric was thoroughly rinsed with distilled water. The pretreated fabric was air-dried at room temperature (25 ± 2 °C) and conditioned at 65 ± 2% relative humidity for 24 h prior to dyeing experiments.

In all mordanting experiments, the mordant concentration on the weight of fabric (o.w.f.) was kept at 3%. This percentage is based on the dry powder of the corresponding mordant material, either orange peel powder, lemon peel powder, myrobalan powder, or gum acacia powder, directly added to the dye bath.

2.2. Preparation of Orange and Lemon Peel Bio-Mordants

Fresh orange and lemon peels were separated from the fruits, washed thoroughly with tap water followed by distilled water, and cut into small pieces. The pieces were oven-dried at 60 °C until a constant mass was obtained and ground to a fine powder. For aqueous extraction, 10 g of each dried peel powder was mixed separately with 100 mL of distilled water (solid-to-liquid ratio 1:10, *w*/*v*) and heated at 80 °C for 60 min with continuous stirring. Each mixture was cooled to room temperature and filtered through Whatman No. 1 filter paper. The filtrate was concentrated using a rotary evaporator under reduced pressure and dried in an oven at 50 °C to constant mass. The dried extract was ground gently to obtain a uniform powder and stored in airtight, labeled containers in a dry place until use. The resulting orange- and lemon-peel-derived powders were applied separately at 3% on the weight of fabric (o.w.f.) in the simultaneous dyeing bath. These plant-derived materials are metal-free, bio-based auxiliaries rather than conventional metal-salt mordants.

2.3. Extraction Method

For dye extraction, beetroot was thoroughly washed and peeled. The peels were cut into small pieces and air-dried at room temperature. For dye extraction, 12 g of dried beetroot peels were placed into a thimble and inserted into a Soxhlet apparatus. In total, 300 mL of each solvent, such as distilled water, ethanol, and methanol, was used for extraction under identical conditions. The apparatus was heated at the boiling temperature of the respective solvent; the extraction process continued until the material inside the thimble became colorless, which was considered the endpoint of extraction. After that, the extracts were allowed to cool and were filtered to remove remaining solid particles. The dye liquor was collected in the round-bottom flask and concentrated using a rotary evaporator to remove the solvent under reduced pressure at around 40 °C to 50 °C. The resultant concentrated extracts were subsequently dried and stored in airtight containers preferably protected from light at 4 °C temperature. Table 1 shows the beetroot crude extraction yield, which was in the range of 27.70% to 35.06% depending upon the type of solvent used and was calculated according to Equation (1). The extraction yields were 27.70% for water, 30.00% for methanol, and 35.06% for ethanol. Ethanol produced the highest color yield because of its suitable polarity to extract a wide range of compounds from Beta vulgaris, including

phenolic compounds and pigments. Ethanol has the ability to penetrate plant matrices and dissolve polar and moderately polar compounds and results in greater recovery of extract [32]. A schematic representation of the extraction process is provided in Figure 1.

$$\text{Extraction yield (\%)} = [\text{Mass of dry dye extract (g)}/\text{Mass of dry raw material (g)}] \times 100 \quad (1)$$

Table 1. Assessment of extraction yield from Beta vulgaris peel crude material using different solvents.

Solvent	Dry Dye Extract (g)	Dry Raw Material (g)	Extraction Yield (%)
Water	3.32	12.00	27.70
Ethanol	4.21	12.00	35.06
Methanol	3.60	12.00	30.00

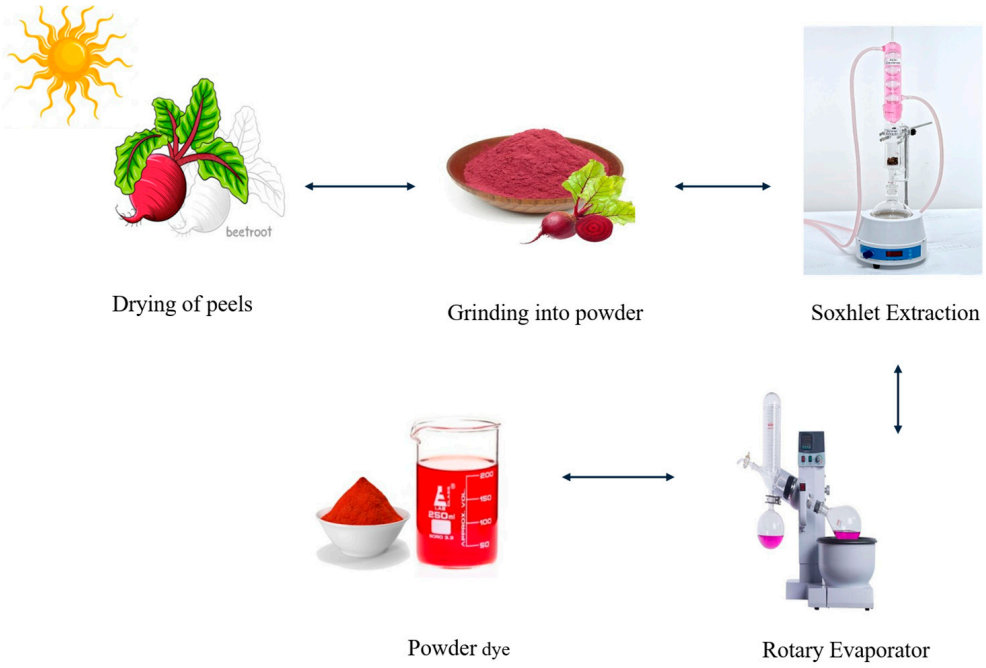

Figure 1. Schematic representation of dye extraction from beetroot peels using Soxhlet Extractor and rotary evaporator.

2.4. Application Method

For fabric dyeing, the simultaneous mordanting technique was employed. The dye bath was prepared with a material-to-liquor ratio of 1:30, and cotton fabric samples were immersed in the bath. Dye concentrations of 2%, 3%, and 4% (o.w.f.) were applied, while the mordant concentration was maintained at 3%. In simultaneous mordanting, the dye and mordant were mixed before introducing the fabric into the dye bath.

During dyeing, the bath temperature was kept at 90 °C. Once the dye bath temperature reached 90 °C, the dyeing process continued for 90 min with continuous stirring to improve dye–fabric contact. For improved dye exhaustion, 1% NaCl was added in two portions; one half was added after the fabric had been completely immersed in the dye bath, and the second half was added after approximately 10–15 min. The bath pH was adjusted to 4.5 ± 0.1 with dilute acetic acid before the fabric was introduced and was verified at the end of dyeing. After dyeing, the bath was cooled to 50 °C, the samples were rinsed in cold distilled water until the rinse liquor was colorless, and dried flat in the dark at room temperature. All specimens were then conditioned at 20 ± 2 °C and 65 ± 4% relative humidity for 24 h before any measurement was taken. A single dyeing time of 90 min

and a single dyeing temperature of 90 °C were applied throughout the study, and Table 2 corresponds exactly to this procedure, with all dye and bio-mordant concentrations in it being expressed as percentages on the weight of fabric (% o.w.f.). The design of experiments (DOE) for dyeing with simultaneous mordanting is shown in Table 2. All the samples were developed and subsequently tested according to standard operating procedures. The design of experiments (DOE) for dyeing with simultaneous mordanting is shown in Table 2. All the samples were developed and subsequently tested according to standard operating procedures.

Table 2. Design of experiments for natural dyeing and simultaneous mordanting of cotton fabric using the exhaust method of dyeing.

Dye Concentration (% o.w.f.)	Extraction Medium	Bio-Mordants Tested (Each 3% o.w.f.)
2	Aqueous	Orange peel; lemon peel; myrobalan; gum acacia
3	Aqueous	Orange peel; lemon peel; myrobalan; gum acacia
4	Aqueous	Orange peel; lemon peel; myrobalan; gum acacia
2	Ethanol	Orange peel; lemon peel; myrobalan; gum acacia
3	Ethanol	Orange peel; lemon peel; myrobalan; gum acacia
4	Ethanol	Orange peel; lemon peel; myrobalan; gum acacia
2	Methanol	Orange peel; lemon peel; myrobalan; gum acacia
3	Methanol	Orange peel; lemon peel; myrobalan; gum acacia
4	Methanol	Orange peel; lemon peel; myrobalan; gum acacia

2.5. Experimental Design, Controls and Replication

The study follows an experimental design with three factors such as extraction medium at three levels (distilled water, ethanol, methanol), dye concentration levels (2, 3 and 4% o.w.f.) and bio-mordant at four levels (orange peel, lemon peel, myrobalan, gum acacia) (Table 2).

2.6. Characterization

2.6.1. Color Strength (K/S) and Color Coordinates (CIELAB) Assessment

Reflectance measurements were obtained using a CHNSpec CS-826 spectrophotometer (CHNSpec Technology (Zhejiang) Co., Ltd., Taizhou, China) in SCI mode with D65 illumination and a 10° standard observer. The instrument software was used to report the CIELAB coordinates (L*, a*, b*) and color strength (K/S) calculated from the measured reflectance data. Five readings were taken at different positions on each sample using four fabric layers. Samples were conditioned before measurement.

The K/S measurements were recorded for all samples, and the results were subsequently plotted in graphical form for clearer interpretation, as discussed in the results and discussion section. The color strength (K/S) of the dyed cotton samples was evalu-

ated at 537 nm, corresponding to the absorption maximum "λ_{max}" of the beetroot extract determined by UV–Vis spectrophotometry (PerkinElmer, Waltham, MA, USA).

2.6.2. Colorfastness Testing Methods

Bleached cotton fabrics were placed in stainless-steel containers of a launder-o-meter (Roaches, Washtec model, Batley, UK) at a liquor ratio of 50:1. The machine was operated at 60 °C for 30 min with continuous rotation to ensure uniform washing conditions. After completion, the samples were rinsed, dried, and assessed for staining against a standard cotton fabric according to the gray scale (rating 1–5) to determine washing fastness ratings. The washing fastness of the dyed fabrics was evaluated according to ISO 105-C10:2006. A detergent solution was prepared using 4 g/L detergent and 1 g/L sodium perborate in distilled water. The pH was adjusted to 10.5 ± 0.1 with approximately 1 g/L sodium carbonate. The washing test used a liquor ratio of 50:1 at 60 °C for 30 min in one washing cycle. The specific detergent type or trade name was not recorded in the available experimental details. Instrumental color measurements were obtained using the spectrophotometer described above, where paired unwashed and post-washing CIELAB measurements were available. The total color difference (ΔE*ab) was calculated using the unwashed sample as the reference.

The perspiration fastness of the dyed cotton fabrics was assessed according to ISO 105-E04 [33]. Fabric specimens were stitched along one short edge to bleached cotton fabric of equal dimensions. The composite samples were immersed separately in acidic and alkaline l-histidine monohydrochloride monohydrate solutions for 30 min at a liquor ratio of 50:1. After soaking, the specimens were mounted in perspirometer kits, and the prescribed pressure was applied. The loaded kits were then placed in an oven at 37 °C for 4 h, then dried separately at ambient temperature. Following exposure, the samples were removed, the stitches were carefully opened (leaving one edge intact), and the fabrics were air-dried at 60 °C. Color change in the cotton fabrics and staining on the adjacent multifiber fabrics were evaluated against the standard gray scale (rating 1–5).

Dry rubbing fastness of the dyed fabrics was determined using a crock meter in accordance with ISO 105-X12 [34]. Cotton specimens measuring 5 × 14 cm were subjected to to-and-fro rubbing against a standard white test cloth under a pressure of 9 N. The test was performed on samples both before and after washing. The degree of color transfer to the rubbing cloth was evaluated using the standard gray scale (rating 1–5).

Wet rubbing fastness was evaluated under the same conditions as the dry rubbing test, using a crock meter in accordance with ISO 105-X12 [34]. The only difference was that the standard rubbing cloth was soaked in deionized water prior to testing. Dyed samples were subjected to rubbing under a pressure of 9 N, and the extent of color transfer and staining was assessed using the standard gray scale (rating 1–5).

Light fastness of the dyed fabrics was evaluated using a Weather-O-meter (Atlas, Mount Prospect, IL, USA). A xenon arc lamp, simulating natural daylight (illuminant D65), was employed as the light source. Fabric specimens were exposed to the xenon arc light under standard testing conditions for 24 h. Following exposure, the degree of color change in the dyed fabrics was assessed against the standard blue wool scale (rating 1–8).

2.6.3. Antioxidant and Ultraviolet Protection Testing Methods

To investigate the antioxidant property of the beetroot extract dyed cotton samples, the colorimetric 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) radical reduction assay was applied. The $ABTS^{+\bullet}$ radical solution was prepared by taking 2.45 mM potassium persulphate ($K_2S_2O_8$) and 7 mM ABTS. Both reagents were added to deionized water. The prepared solution was incubated for 16 h in the dark, and phosphate buffer at

0.1 M concentration was added to achieve an absorbance of 0.700 at the maximum wavelength of 734 nm. For the antioxidant assay of the developed dyed cotton samples, 2 mL of the prepared ABTS solution was added to 50 mg of fabric specimen and incubated for 30 min in the dark. As the colorimetric reaction completed, the absorbance was measured at 734 nm using an EZ400 Microplate Reader. To calculate the percentage of $ABTS^{+\bullet}$ radical inhibition by the test specimen, Equation (2) was employed.

$$ABTS^{+\bullet}\ radical\ inhibition\ (\%) = \left(\frac{A_{ABTS^{+\bullet}} - A_S}{A_{ABTS^{+\bullet}}}\right) \times 100 \quad (2)$$

The antioxidant assay was performed in triplicate (n = 3) for each sample. Results are expressed as mean ± standard deviation, and the mean values with their standard deviations are presented for comparison.

The fabric specimens were cut into 50 mm × 50 mm dimensions to cover the illumination window of the equipment. The UPF value is achieved by spectral transmittance measurements using the following Equation (3) according to ASTM D6603.

$$UPF = \frac{\sum_{\lambda=280}^{400} E(\lambda).\ S(\lambda).\ \Delta\lambda}{\sum_{\lambda=280}^{400} E(\lambda).\ S(\lambda).\ T\ (\lambda).\Delta\lambda} \quad (3)$$

In Equation (3), $E(\lambda)$ is the CIE reference erythema, $S(\lambda)$ is the solar radiation intensity, $T(\lambda)$ is the spectral diffuse transmittance of the fabric, and $\Delta\lambda$ is the wavelength interval. According to this standard, the test specimens are categorized into the following UVR categories.

(a) UPF values = 15–24: Good UV Protection;
(b) UPF values = 25–39: Very Good UV Protection;
(c) UPF values = 40–50+: Excellent UV Protection.

3. Results

3.1. Color Strength (K/S) and CIELAB Color Coordinates Assessment

Table 3 presents the CIELAB color coordinates (L, a*, and b*) of cotton fabric using beetroot dye extracted through an aqueous medium. The results show that lightness (L) values are inversely proportional to K/S, which is evident from Table 3 and Figure 2. From the CIE a* and b* values, it is clear that the samples are on the redder and yellower side. Four bio-mordants were applied, while the dye concentrations of 2%, 3%, and 4% were used throughout the experiment with a fixed mordant concentration of 3% (o.w.f.). The dye extracted in distilled water imparted different colors to the cotton fabric when applied with 2%, 3%, and 4% dye shade in the presence of bio-mordants. Orange peel, myrobalan, and lemon peels produced higher color strength by increasing the dye shade percentage [19]. However, gum acacia produced lower shade depth in terms of K/S on the cotton fabric (Figure 2).

Table 3. CIELAB color coordinates of the cotton fabrics dyed with beetroot dye extracted using an aqueous medium and applying four bio-mordants.

Shade (%)	Orange Peel			Lemon Peel			Myrobalan			Gum Acacia		
	L	a*	b*	L	a*	b*	L	a*	b*	L	a*	b*
2	62.96	7.41	11.6	71.86	7.42	14.16	72.43	7.27	13.36	76.87	8.17	11.01
3	64.45	6.64	10.05	72.22	7.49	13.56	74.70	7.95	11.05	78.23	8.92	12.18
4	69.33	7.52	11.66	70.94	7.22	14.04	71.09	8.63	12.17	77.96	9.04	12.83

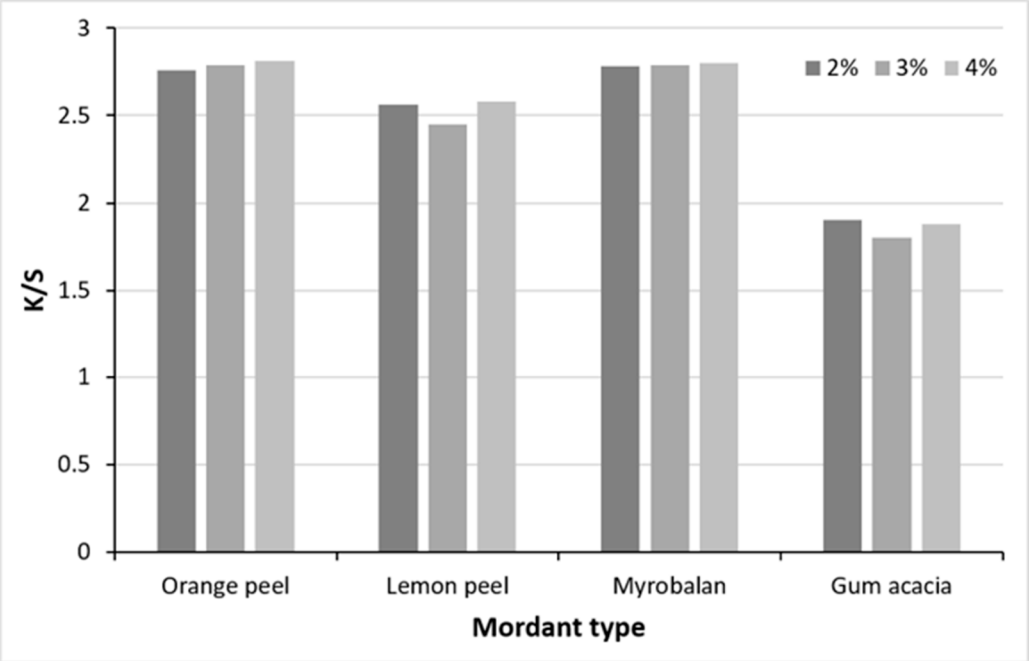

Figure 2. K/S values of cotton fabrics dyed with beetroot dye (2, 3, and 4% shade depths) extracted using an aqueous medium and applying four bio-mordants.

The highest color strength was observed for myrobalan (K/S = 2.78–2.80), followed by orange peel (K/S = 2.76–2.81) and lemon peel (K/S = 2.56–2.58), which can be attributed to the strong mordanting efficiency of these bio-mordants that enhanced dye uptake due to stronger coordination complexes between bio-mordants, cotton fabrics, and dye particles. The increased concentration of bio-mordant can cause increased binding points on the fabric surface, allowing greater dye uptake and dye fixation [35]. However, the increase in the concentration of beetroot dye has no significant effect on the K/S values.

Table 4 presents the CIELAB color coordinates of the cotton fabrics dyed with beetroot dye extracted through an organic medium (ethanol). The CIE L values observed in Table 4 are slightly less compared to those in Table 3, which confirms that the samples dyed with ethanolic extract are slightly darker. For instance, the CIE a* and b* values are all positive, which shows that the samples' tone is redder and yellower. At a 2% dye concentration with 3% mordant, the dye imparted noticeable color to the cotton fabric with orange peel, myrobalan, lemon peels, and gum acacia as bio-mordant. Increasing the dye concentration to 3% resulted in a deeper shade on the cotton fabric compared to the 2% dye shade. Further increasing the dye concentration to 4% enhanced the overall color depth on the fabric. The gradual increase in shade depth by increasing the dye concentration (from 2% to 4%) is due to increased diffusion and adsorption of dye molecules because of the high availability of dye particles in the dye bath. Higher concentration causes more betacyanin dye molecules to react with more hydroxyl (–OH) groups of cellulose and increases the dye uptake [31].

The effects of mordant have a great impact on color strength; orange peel exhibited the highest color strength (K/S = 3.74–4.44), showing a steady increase with the rise in dye concentration (Figure 3), indicating strong dye and fiber interaction [36,37]. Myrobalan and lemon peels also demonstrated appreciable color strength values (K/S = 3.70–3.75) and (K/S = 3.60–3.67), respectively [38], followed by the gum acacia, which produced moderate K/S values (2.10–2.11).

Table 4. CIELAB color coordinates of the cotton fabrics dyed with beetroot dye extracted using an organic medium (ethanol) and applying four bio-mordants.

Shade (%)	Orange Peel			Lemon Peel			Myrobalan			Gum Acacia		
	L	a*	b*	L	a*	b*	L	a*	b*	L	a*	b*
2	53.21	8.34	11.9	60.90	7.03	10.71	56.22	7.45	13.36	69.37	7.41	13.92
3	46.42	8.41	11.76	57.12	7.72	14.58	54.97	7.28	13.22	70.11	8.03	11
4	41.33	7.3	10.94	58.01	7.04	14.11	54.07	7.73	13.58	71.75	8.2	11.37

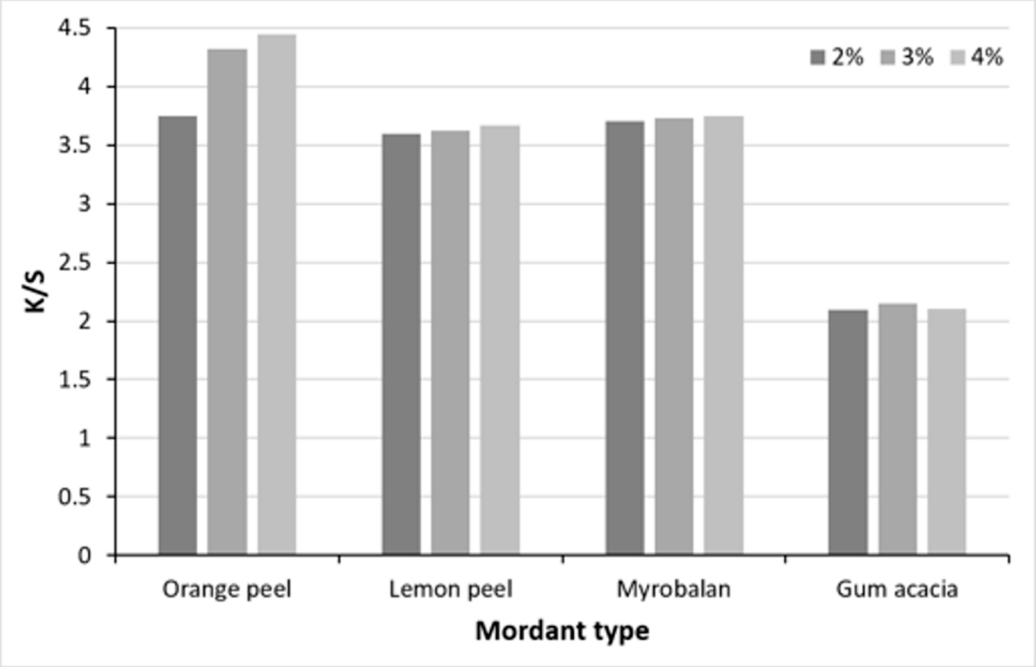

Figure 3. K/S values of cotton fabrics dyed with beetroot dye (2, 3, and 4% shade depths) extracted using ethanolic medium and applying four bio-mordants.

Table 5 presents the CIELAB color coordinates of the cotton fabrics dyed with beetroot dye extracted through an organic medium (methanol). At different dye concentrations and keeping the mordant percentage constant at 3%, the dye imparted color to the cotton fabric. The CIE L values given in Table 5 (methanol) are lower than those in Table 4 (ethanol) and Table 3 (aqueous), which confirms that the samples dyed with methanol are darker than those dyed with ethanol and aqueous extracts. In addition, CIE a* and b* values are positive, which shows that the samples are redder and yellower in tone. Lemon peels, myrobalan, and orange peels as mordant showing the higher color strength by increasing the dye concentration [19] whereas gum acacia showing less coloration as compared to other mordant. Meanwhile, Table 4 shows the higher color yield in case of using methanol as extraction medium as compared to ethanol and water, which were used earlier.

Figure 4 shows the K/S values of cotton fabrics dyed with beetroot extract obtained using methanol as the extraction medium. Orange peels exhibited the highest color strength (K/S = 3.81–4.57), showing a consistent increase with higher dye concentrations, which indicates strong dye and fiber interaction and effective mordanting behavior. Myrobalan and lemon peels also demonstrated a gradual increase in K/S values ranging from 3.90 to 4.00 and 3.75 to 3.77, respectively, as the dye percentage increased. The gum acacia produced moderate K/S values (K/S = 2.15–2.19).

Table 5. CIELAB color coordinates of the cotton fabrics dyed with beetroot dye extracted using an organic medium (methanol) and applying four bio-mordants.

Shade (%)	Orange Peel			Lemon Peel			Myrobalan			Gum Acacia		
	L	a*	b*	L	a*	b*	L	a*	b*	L	a*	b*
2	47.35	8.05	11.72	53.75	8.77	11.8	52.06	7.42	13.86	67.23	7.28	13.21
3	45.04	8.76	11.86	55.04	7.4	13.85	51.56	7.53	13.74	62.95	7.97	10.96
4	41.02	9.02	12.47	59.23	7.22	13.62	49.87	7.26	14.26	63.45	8.79	12.2

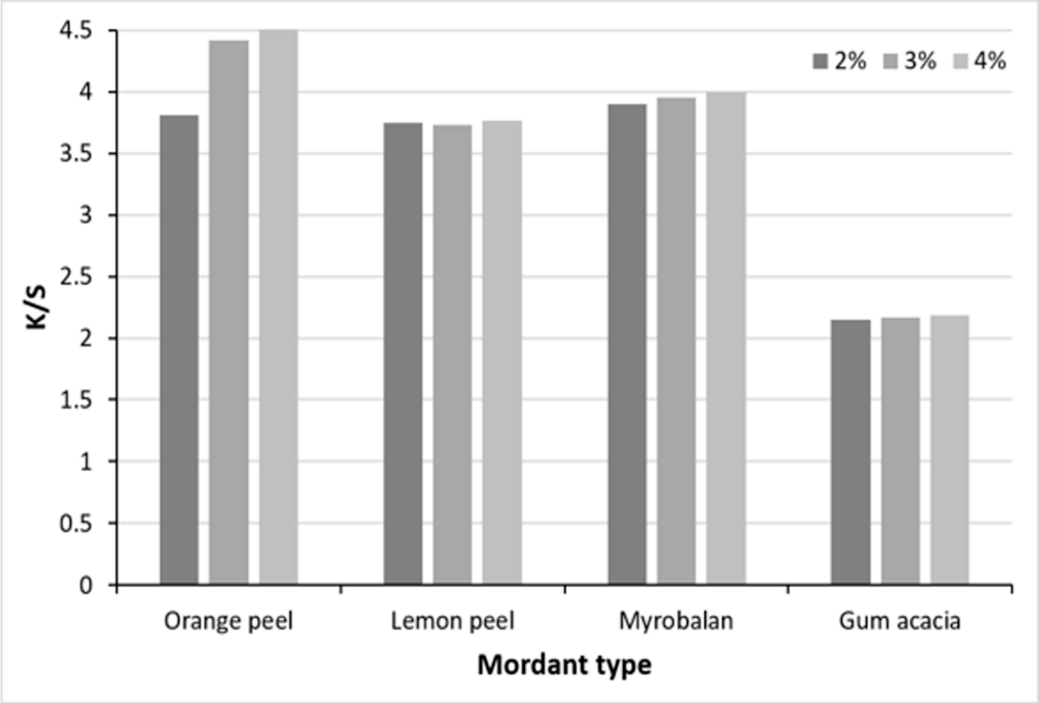

Figure 4. K/S values of cotton fabrics dyed with beetroot dye (2, 3, and 4% shade depths) extracted using methanolic medium and applying four bio-mordants.

3.2. Colorfastness Assessment

The fastness results reported in Tables 6–11 are for cotton fabrics dyed in the presence of the four bio-mordants. Unmordanted dyed cotton was not included as a control in the original experimental design. Therefore, the absence of unmordanted controls hinders quantitative assessment of baseline fastness and mordant-specific improvements. The results are thus limited to comparisons among bio-mordant treatments.

Table 6. Washing fastness (staining on cotton) of beetroot-dyed cotton fabrics extracted through different media and using four bio-mordants. Visual gray-scale rating: 1–5.

Dye Shade (%)	Mordant (3%)	Aqueous	Ethanol	Methanol
2	Orange peel	4–5	3–4	4–5
	Lemon peel	4–5	4–5	4–5
	Myrobalan	3–4	3–4	3–4
	Gum acacia	4–5	4–5	4–5

Table 6. *Cont.*

Dye Shade (%)	Mordant (3%)	Aqueous	Ethanol	Methanol
3	Orange peel	4–5	4–5	4–5
	Lemon peel	4–5	4–5	4–5
	Myrobalan	4–5	4–5	4–5
	Gum acacia	4–5	4–5	4–5
4	Orange peel	4–5	4–5	4–5
	Lemon peel	4–5	3–4	3–4
	Myrobalan	3–4	3–4	3–4
	Gum acacia	4–5	4–5	4–5

Table 7. Perspiration fastness (alkaline and acidic) of beetroot-dyed cotton fabric extracted through different media and using four bio-mordants. Visual gray-scale ratings: 1–5.

Dye Shade (%)	Mordant (3%)	Aqueous		Ethanol		Methanol	
		Alkaline	Acidic	Alkaline	Acidic	Alkaline	Acidic
2	Orange peel	4	3–4	4–5	4	4–5	4
	Lemon peel	4–5	4	4–5	4	4–5	4
	Myrobalan	4–5	3–4	4–5	4–5	4–5	3–4
	Gum acacia	4–5	4	4–5	4	4–5	4
3	Orange peel	3–4	3–4	3–4	3–4	3–4	3–4
	Lemon peel	4–5	4	4–5	3–4	4–5	4–5
	Myrobalan	3–4	3–4	3–4	3–4	3–4	4
	Gum acacia	4–5	4	4–5	4	4–5	4–5
4	Orange peel	3–4	3	3–4	4	3–4	3–4
	Lemon peel	4–5	3–4	4–5	4–5	4–5	4
	Myrobalan	3–4	3	3–4	3–4	3–4	3–4
	Gum acacia	4–5	3–4	4	4	4–5	4

Table 8. Rubbing fastness of beetroot-dyed cotton fabric extracted through aqueous medium and applied using four bio-mordants. Visual gray-scale ratings: 1–5.

Dye Shade (%)	Mordant	Dry Rubbing Fastness	Wet Rubbing Fastness
2	Orange peel	4–5	4–5
	Lemon peel	4–5	4–5
	Myrobalan	4–5	4–5
	Gum acacia	4–5	4–5
3	Orange peel	4–5	4–5
	Lemon peel	4–5	4–5
	Myrobalan	4–5	4–5
	Gum acacia	4–5	4–5
4	Orange peel	4–5	4–5
	Lemon peel	4–5	4–5
	Myrobalan	3–4	3–4
	Gum acacia	4–5	4–5

Table 9. Rubbing fastness of beetroot-dyed cotton fabrics extracted through organic medium (Methanol) and using four bio-mordants. Visual gray-scale ratings: 1–5.

Shade (%)	Mordant	Dry Rubbing Fastness	Wet Rubbing Fastness
2	Orange peel	4–5	4–5
	Lemon peel	4–5	4–5
	Myrobalan	4–5	4–5
	Gum acacia	4–5	4–5
3	Orange peel	4–5	3–5
	Lemon peel	4–5	3–4
	Myrobalan	4–5	3–4
	Gum acacia	4–5	4–5
4	Orange peel	4–5	3–4
	Lemon peel	4–5	3–4
	Myrobalan	4–5	3–4
	Gum acacia	4–5	4–5

Table 10. Dry rubbing fastness of beetroot-dyed fabric extracted through organic medium (ethanol) and using four bio-mordants. Visual gray-scale ratings: 1–5.

Shade (%)	Mordant 3%	Dry Rubbing Fastness	Wet Rubbing Fastness
2	Orange peel	4–5	3–4
	Lemon peel	4–5	3–4
	Myrobalan	4–5	3–4
	Gum acacia	4–5	4–5
3	Orange peel	4–5	3–4
	Lemon peel	4–5	3–4
	Myrobalan	4–5	3–4
	Gum acacia	4–5	4–5
4	Orange peel	4–5	3–4
	Lemon peel	4–5	3–4
	Myrobalan	4–5	3–4
	Gum acacia	4–5	4–5

Table 6 presents the washing fastness results of cotton dyed samples in terms of staining on cotton using a gray scale rating (1–5). In the case of aqueous extraction, fabrics dyed with the 2% dye shade using orange peel, lemon peel, and gum acacia as bio-mordants exhibited no noticeable staining on cotton after washing, indicating good to excellent washing fastness. Meanwhile, Myrobalan as a bio-mordant showed good wash fastness results in the range of (3–4). At the 3% dye shade, all samples retained their original shade, demonstrating excellent wash fastness. However, at the 4% dye shade, orange peel, lemon peel, and gum acacia showed excellent wash fastness ratings (4–5), while myrobalan showed a good wash fastness rating (3–4). In the case of organic extraction (ethanol) at the 2% dye shade, fabrics mordanted with lemon peel and gum acacia showed excellent washing fastness (4–5), while myrobalan and orange peel showed good fastness ratings (3–4). Methanol at the 2% dye shade, mordanted with orange peel, lemon peel, and gum acacia, showed excellent fastness, and myrobalan showed good fastness (3–4). At the 3% dye shade, all samples exhibited excellent fastness. At the 4% dye shade, orange peel and gum acacia again retained their

shades after washing and showing fastness in the range of (4–5), which is excellent, while myrobalan and lemon peel show fastness in the range of (3–4) as good fastness performance.

Table 11. Light Fastness of beetroot-dyed cotton fabrics dyed using different extraction media and using four bio-mordants (Rating 1–8 according to blue wool scale). Blue wool scale rating: 1–8.

Shade (%)	Mordant 3%	Aqueous	Ethanol	Methanol
2	Orange peel	3–4	3–4	3–4
	Lemon peel	4–5	4–5	4–5
	Myrobalan	4–5	4–5	4–5
	Gum acacia	3–4	3–4	3–4
3	Orange peel	3–4	3–4	3–4
	Lemon peel	4–5	4–5	4–5
	Myrobalan	4–5	4–5	4–5
	Gum acacia	3–4	3–4	3–4
4	Orange peel	2–3	2–3	2–3
	Lemon peel	4–5	4–5	4–5
	Myrobalan	4–5	4–5	4–5
	Gum acacia	3–4	3–4	3–4

The better washing fastness of samples mordanted with orange peel, lemon peel, and gum acacia is because of better dye and fabric connections and good dye fixation through hydrogen bonding and film-forming mechanisms, while myrobalan showed a slightly lower value of fastness but still good wash fastness because of the development of partially surface-bound tannin dye complexes [39]. These results clearly indicate that the choice of mordant significantly influences the wash fastness properties of dyed cotton fabrics.

Table 7 presents the perspiration fastness results of cotton fabrics dyed with beetroot extract using different extraction media and mordants. At 2% dye shade percentage, all mordants showed good to excellent wash fastness through all extraction media. Fabrics mordanted with lemon peel, gum acacia, and myrobalan mainly showed ratings of 4–5, indicating minimum to no color change after washing; this proves the effective dye fixation even at lower dye concentration too. At 3% dye concentration, minimum reduction in perspiration fastness was observed for some mordants. Orange peel and myrobalan showed ratings of 3–4, whereas lemon peel and gum acacia maintained 4–5 ratings irrespective of the extraction medium. This is because of stronger hydrogen bond formation between dye and fiber. At the highest concentration (4%), the same pattern continued. The alkaline perspiration fastness is slightly higher than those of acidic medium, and there is no specific trend observed neither in alkaline nor in acidic fastness rating. It may be due to the complex nature of simultaneous dyeing and mordanting processes.

Table 8 shows the dry and wet rubbing fastness of dye extracted through an aqueous medium. The results show excellent color stability for cotton fabrics dyed at 2% and 3% shade levels, with all mordants indicating ratings of 4–5, which represent strong dye and fiber fixation and minimal unfixed dye on the surface. At the 4% shade, orange peel, lemon peel, and gum acacia similarly showed excellent rubbing fastness (4–5) under both dry and wet conditions. In the case of myrobalan, a slight reduction in fastness (3–4) was observed at 4% shade, which may be due to the presence of unfixed dye, such as tannin complexes that may be removed during rubbing.

Table 9 showing the rubbing fastness that at 2% dye shade, all mordants exhibited excellent dry and wet rubbing fastness in range of (4–5), which is the representation of

strong dye fixation. At 3% and 4% dye shades, dry fastness remained persistently high, at (4–5), for all types of mordants, which represents good resistance to mechanical abrasion under dry conditions. On the other hand, a slight reduction in wet rubbing fastness (3–4) was observed mainly for orange peel, lemon peel, and myrobalan, likely due to the presence of unfixed dye on the fabric surface. However, gum acacia maintained wet rubbing fastness (4–5) at all dye concentrations, likely due to its film-forming nature that helps retain dye within the fiber structure.

Table 10 shows that dry rubbing fastness was persistently high (4–5) for all bio-mordants and dye concentrations. While wet rubbing fastness for orange peel, lemon peel, and myrobalan were slightly lower (3–4), gum acacia maintained excellent wet fastness (4–5). Overall, all mordants show good dry fastness, with wet fastness varying depending on mordant type and dye and fiber bonding.

The results presented in Table 11 indicate the light fastness of dyed samples using the blue wool scale (1–8) rating. Orange peel and gum acacia showed persistent results of light fastness 3–4 (BW scale) at 2% and 3% dye shades and 2–3 (BW scale) at 4% dye shade and all solvents. This poor to moderate light fastness value is due to only weak stabilization of hydrogen bonding with the dye. Meanwhile, Lemon peel and myrobalan show light fastness 4–5 (blue wool scale) consistently. This fair to good light fastness is likely due to tannins in myrobalan and citric acid in lemon peel, which form strong dye–fiber bonding that significantly stabilizes the dye against photodegradation.

3.3. Antioxidant and Ultraviolet Protection Assessment

Functional compounds present in the natural dyes and bio-mordants can mitigate skin-related issues by neutralizing free radicals as well as reducing oxidative stresses, which ultimately help induce the phenomenon of inflammation [40]. In addition, these compounds also help in the colorfastness and durability of the textile material [41]. The antioxidant activities of the developed samples using natural mordants recorded via the standard protocol are shown in Figure 5. Cotton fabrics (control) with no dyeing and mordanting were considered as control samples, and the observed antioxidant activity showed an $ABTS^{+\bullet}$ radical reduction of 10.05 ± 2.6% (Figure 5). After the application of beetroot dye and orange peel as mordants, a significant increase in antioxidant activity was observed in all dyeing and mordanting cases. The ethanol-extracted dyes and mordanting delivered the best results, with an antioxidant activity of 88.20% ± 8.5%. It is pertinent to mention that beetroot contains betacyanin and other phenolic compounds (tannins, phenolic acids and flavonoids), while the orange peel contains several tannins and other flavonoids, which possess strong antioxidant activities [42]. Orange peel bio-mordants delivered the best antioxidant activities, followed by lemon peels, gum acacia, and myrobalan. In the case of extraction methods, ethanolic extract dyed samples rendered the best antioxidant activities, followed by methanolic extracts, and finally aqueous extract. Natural dyes from madder applied to wool fabric revealed significant antioxidant activities due to the presence of potential functional compounds [43]. The maximum ABTS radical inhibition was 88.20 ± 8.5%, indicating substantial antioxidant activity in the treated cotton fabric. It is pertinent to mention that beetroot contains betacyanin and other phenolic compounds (tannins, phenolic acids and flavonoids), while the orange peel contains several tannins and other flavonoids, which possess strong antioxidant activities. Antioxidant results are reported as mean ± standard deviation from three technical replicates (n = 3).

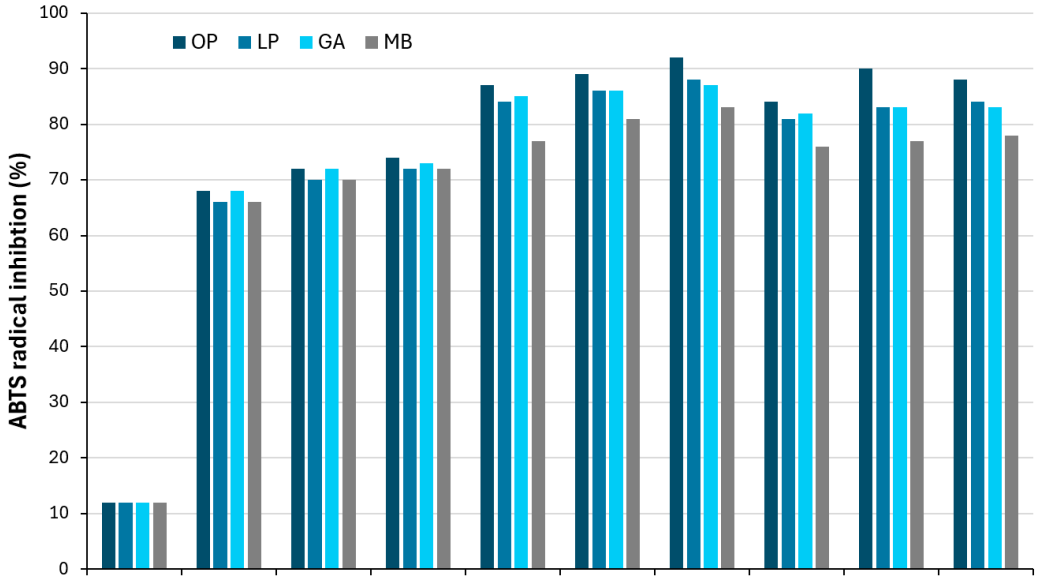

Figure 5. $ABTS^{+\bullet}$ radical inhibition (%) of the natural dyes and natural mordant-applied cotton fabrics. Control sample was undyed and unmordanted cotton fabric; OP, LP, GA, and MB correspond to the cotton fabrics dyed using orange peels, lemon peels, gum acacia, and myrobalan, respectively, as natural mordants. (AE, EE, and ME stand for aqueous extract, ethanolic extract, and methanolic extract, respectively).

The UPF results of the control, dyed, and mordanted samples are given in Table 12, which shows that the control cotton fabric does not meet the required UPF level. The observed UPF value for the control samples was 13, which corresponds to the low UVR category [44]. Interestingly, the natural extracts dyed and mordanted samples revealed more than 30 UPF ratings. The increase in beetroot concentration in the dyeing process resulted in significant enhancement of the UPF rating. It is pertinent to mention that darker samples revealed slightly higher UPF values as compared to lighter ones. This provides significant protection against UV radiation. The UPF values were recorded according to ASTM D6603.

Table 12. UPF ratings of beetroot-dyed cotton samples using different extraction media and four bio-mordants, recorded according to ASTM D6603.

Sample	Ultraviolet Protection Factor (UPF)			
	Orange Peel	**Lemon Peel**	**Gum Acacia**	**Myrobalan**
Control Fabric	13			
2% beetroot dye AE	41	38	34	30
3% beetroot dye AE	48	42	45	43
4% beetroot dye AE	66	57	51	48
2% beetroot dye EE	65	43	46	49
3% beetroot dye EE	71	63	52	49
4% beetroot dye EE	88	67	57	59

Table 12. *Cont.*

Sample	Ultraviolet Protection Factor (UPF)			
	Orange Peel	Lemon Peel	Gum Acacia	Myrobalan
2% beetroot dye ME	65	39	38	39
3% beetroot dye ME	68	43	47	41
4% beetroot dye ME	79	52	52	46

A mechanism is proposed for the dye–mordant–cotton fiber interaction using these natural materials (Figure 6). Hydrogen bonding is the dominant force that exists between the dye–mordant–cotton, along with other weak intermolecular forces such as Van der Waals interactions. The washing, rubbing and perspiration fastness of the textile materials mainly depends upon these bonds. Meanwhile, for better light fastness, a stable chromophore is the key to resisting the effect of UV radiation.

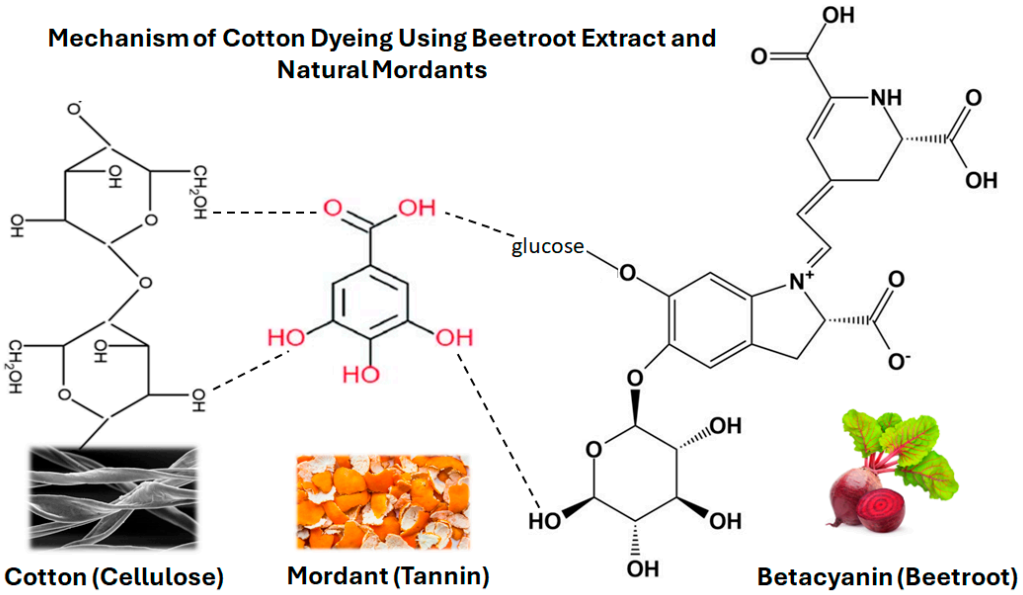

Figure 6. A generalized and proposed mechanism of bio-mordants (tannin), dye (betacyanin), and cotton fabric (cellulose) interaction using beetroot dyes and bio-mordants.

4. Conclusions

This study evaluated *Beta vulgaris* (beetroot) peel, an agri-food residue, as a colorant for cotton fabric in a factorial comparison of three extraction media, three dye concentrations, and four food-waste bio-mordants. The highest color strength was obtained with orange peel and the methanolic extract at 4% o.w.f. (K/S = 4.57) closely followed by the ethanolic extract under the same conditions (K/S = 4.44), while the aqueous extract did not exceed K/S 2.81 with any bio-mordant. The best light fastness (4–5 on the blue wool scale) was obtained with lemon peel and myrobalan at every concentration and in every medium, whereas orange peel fell to 2–3 at 4% o.w.f. The most uniform wet rubbing and perspiration ratings (4–5 throughout) were given by gum acacia, which at the same time gave the lowest color strength (K/S = 2.10–2.19). The highest ABTS radical inhibition (88.20 ± 8.5%) and the highest ultraviolet protection factor (UPF 88) were both recorded for orange peel with the ethanolic extract at 4% o.w.f. No single combination therefore maximizes all four responses simultaneously, and the bio-mordant should be selected according to the

property to be prioritized: orange peel for color strength, antioxidant activity and ultraviolet protection, lemon peel or myrobalan for light fastness, and gum acacia for wet rubbing and perspiration fastness.

5. Limitations and Future Recommendations

i. The study presented a novel approach to dye cotton fabric using natural dyes and bio-mordants, but there are several limitations in the commercial applications, which open the way for future research work.
ii. The mixture of natural dyes and bio-mordant must be characterized by HPLC, GC-MS, and UV-visible spectroscopy to check the presence of functional compounds and their concentrations. In addition, the mechanism of interaction between dye-mordant-fiber must be verified with the help of advanced analytical tools.
iii. The synergistic effect of the present functional compounds must be established, and their interaction with cotton fabric must be realized.
iv. Mechanical performance and antibacterial performance must be investigated because the explored natural dyes and bio-mordants are antimicrobial in nature.
v. The dyeing treatment used in this study (90 °C at pH 4.5) may also affect cotton cellulose and potentially cause some degradation, which could influence the mechanical properties of the fabric. Because tensile strength and other mechanical properties were not measured in the present work, the colorimetric and colorfastness results should be interpreted as measures of color performance only and not as evidence that the fabric's mechanical integrity was retained. Mechanical testing will therefore be included in future work to determine whether the applied dyeing conditions affect fabric strength and durability.
vi. The antioxidant assay was performed in triplicate (n = 3), and results are reported as mean ± standard deviation. Future studies should assess reproducibility across independently prepared dyeing batches and apply appropriate inferential statistical analyses when supported by experimental design.
vii. Post-washing color characterization, including K/S values, CIELab* coordinates, and total color difference (ΔE) after the first washing cycle relative to the unwashed samples, was not conducted in this study. These measurements will be addressed in future research to provide a more comprehensive evaluation of the color stability and washing durability of the dyed fabrics.

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