Natural Dyeing and Antimicrobial Functionalization of Wool Fabrics Dyed with Chinese Dragon Fruit Extract to Enhance Sustainable Textiles

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Abstract: Recently, the natural dyeing process has achieved great importance in the textile wet processing industry due to its clean dyeing, eco-friendliness, and nontoxicity in nature. In the above research project, a unique natural dye extracted from dragon fruit was applied to wool fabric using various mordanting agents to encourage the use of natural dyes and lessen the negative environmental effects caused by synthetic dyeing. The color characteristics ($K/S$), fastness properties, Fourier transform infrared spectroscopy (FTIR), absorption spectra, and thermal and ultraviolet (UV) resistance of the extracted dye and dyed wool samples were tested and characterized. The $K/S$ values of the dyed wool fabrics were between 5.75 and 13.29. The color fastness ratings obtained from the dyed wool fabric were found to be between good and excellent. Hence, the overall results proved that the novel natural dye obtained from dragon fruit can be utilized for dyeing wool material for the production of eco-friendly and sustainable antimicrobial textiles.

Keywords: dragon fruit; natural dyeing; wool; mordanting; antibacterial; textiles

1. Introduction

Natural products in the textile processing industry are gaining significant importance due to their eco-friendliness, low toxicity, non-carcinogenic nature, and natural biodegradability. The dyeing process in textile manufacturing plays a vital role in the acceptability of textiles. Natural dyes are coloring compounds that are derived from several sources (such as animals, fungi, insects, minerals) and various plant parts (such as fruits, roots, flowers, leaves, woods, and seeds), and they have the ability to impart color to substances that have a wide range of shades [1]. Researchers are turning towards natural dyes as a potential alternative to synthetic dyes because bulk production and the extensive use of synthetic dyes may result in environmental damage and may be hazardous to human health due to the toxicity of synthetic dyes [2]. Several natural dyes not only diffuse color to the textile material but also possess several functional characteristics, such as antimicrobial activity, fragrance, insect repellence, and UV protection. Numerous studies on the effects of natural dyes on textiles have produced useful findings for researchers. Several other advanced and modified methods for improving the natural dyeing of wool have also been studied. Shabbir et al. investigated and contrasted the antibacterial capabilities and color features of several naturally occurring dyes obtained from plants such as T. erecta, T. chebula, and A. tinctoria on wool yarn. The findings demonstrated that woolen yarns dyed with carefully chosen natural dyes produced vibrant hues with acceptable color and fastness attributes.
The effects of the natural dyes *T. chebula* and *A. tinctoria*, of which *T. chebula* demonstrated great results in terms of both antibacterial capabilities and color features, were particularly effective in resisting *S. aureus* and *B. subtilis* bacteria [3].

Using distilled water and a Soxhlet device, Eser and Onal extracted natural dyes from nettle leaves. The dyeing of the nettle leaves produced a rich color owing to the absorption of light beams of various wavelengths. Thus, they concluded that nettle leaves are likely a crucial raw material, particularly for dyeing wool fibers [4]. Motaghi, in his research, dyed wool fabric with an aqueous extract of natural dyes from the skin of red onion and barberry roots. The results demonstrated that wool material dyed with barberry roots and red onion skin obtained significant ultraviolet protection factor (UPF) ratings and antibacterial activity. Wool fabric can be effectively protected against ultraviolet radiation by dyeing it with an aqueous extract of barberry roots and red onion skin, both of which have strong UV-blocking properties. Both the red onion peel and the barberry roots used to dye wool fabric had significant antibacterial effects on *S. aureus* [5].

Overall, the ultrasonic dyeing of wool with coconut coir extract as a natural dye was carried out by using natural bio-mordants such as acacia, henna, pomegranate, and turmeric extracts to increase the sustainability of the process. It was discovered that bio-mordanting produced good color features, significantly increased the value of coloring, and improved the fastness ratings of wool materials that had been dyed [6].

The natural dyes obtained from fruits include several different classes of compounds that are responsible for dye ability and biological and other functional properties. Dragon fruit is a vine cactus species belonging to the cactus family that has wide adaptability and is grown in slightly heavy-textured soil. The fruit is highly attractive, with dark red skin that is covered with green scales and red or white flesh that contains numerous small black seeds, much like kiwi fruit. The red or dark pink color of the inner flesh and peel of the dragon fruit is due to the presence of betacyanins, which become pigmented during ripening. Dragon fruit has strong potential for use as a natural dye because it consists of betacyanin, which is a violet–red substance that can be obtained from pigments known as betalains; moreover, betacyanin includes nitrogen molecules and is soluble in water [7,8].

Keeping the above information in mind, in the above research experiment, natural dye was extracted from dragon fruit by an ultrasonic method, and it was applied to wool fibers. The development of different shades and the interactions between natural dyes produced from dragon fruit and wool fibers were also investigated using a variety of metallic mordants. Several functional properties, such as UV protection, antibacterial activity, UPF, and thermal and fastness qualities, of dye wool fabric were tested and characterized. A novel natural dye extracted from Chinese dragon fruit was reported for the dyeing of wool fabric.

### 2. Materials and Methods

#### 2.1. Materials

Pure 100% woolen fabric at 120 g/m² (warp-27, weft-13) was obtained from Xiang Shan Henda Co., Ltd., Shenzhen, China. Dragon fruits were obtained from a local fruit supermarket in China. Absolute ethanol, potassium dichromate (K₂Cr₂O₇), stannous chloride (SnCl₂·2H₂O), and copper sulfate (CuSO₄·5H₂O), which were supplied by Sinopharm Chemical Reagent Co., Ltd., Shanghai, China, were used as mordanting agents. A non-ionic detergent obtained from Hangzhou Xinsheng Dyeing & Printing Pvt. Ltd., Hangzhou, China was applied in the fastness to washing procedure.

#### 2.2. Methods

##### 2.2.1. Extraction of Dye

An ultrasound-assisted extraction technique was used to extract natural dye from dragon fruit. Initially, 60 g of fresh dragon fruit flesh was mixed with 400 mL of solvent (240 mL of ethanol and 160 mL of distilled water) by maintaining a material to liquor ratio (MLR) of 1:20. The mixture was blended for 10 min until the mixture was homogeneous.
in color, after which the mixture was transferred to a conical flask. After that, the conical flask was placed in an ultrasonic bath and sonicated for an hour at 60 °C at a frequency of 28–30 MHz and 160 volts of provided power [9]. Following sonication, the solutions were filtered to remove any remaining dye particles using Grade 1 Whatman filter paper, pore size—11 µm, diameter—90 mm, and parameter—0.25 psi wet burst. The unwanted ethanol was evaporated by a rotary evaporator (RE52CS-1, Shanghai Yarong Biochemical Instrument Factory, Shanghai, China) for reuse.

2.2.2. Pre-Mordanting

The wool fabrics were pre-mordanted in a shaker dyeing machine using 5% of the weight of fabric (OWF) of potassium dichromate, stannous chloride, and copper sulfate as mordanting chemicals for 60 min at 80 °C with a material-to-liquor ratio of 1:20. After the pre-mordanting procedure, the samples were dried for 30 min at room temperature. After drying, the wool samples were further used for dyeing.

2.2.3. Dyeing

Wool samples were dyed in a shaker dyeing machine using a 1:40 material to liquor ratio. Dyeing was performed for both the mordanted and un-mordanted samples to examine the dye ability of the wool fabrics. Initially, dyeing was initiated at 40 °C for 15 min before heating to 90 °C for 50 min at a temperature increase rate of 1.5 °C/min. To remove the unfixed dye on the fiber surface, the dyed wool fabrics were passed through a soaping process. Each soaping procedure lasted for 10 min. Following a first cold wash at room temperature, a second hot wash at 70 °C, a third hot wash at 70 °C using 3 g/L of a soaping detergent, and a fourth hot wash at 70 °C were used to remove the soaping detergent from the fabric. All of the previously washed samples were dried for 24 h at room temperature.

2.2.4. Color Measurement and Analysis

The colorimetric characteristics of the dyed wool fabrics were measured using a Spectra Flash-Data Color (SF-600) device. The Kubelka–Munk equation was used to compute the spectrophotometer’s color yields \( \frac{K}{S} \) and CIE Lab values (L*, a*, b*, c*, and h°). Each sample was measured 3 times, and the average value was recorded.

\[
\frac{K}{S} = \frac{(I - R)^2}{2R}
\]

where \( K \) is the absorbance factor, \( S \) is the scattering factor, and \( R \) is the reflectance of the dyed wool at the wavelength of greatest absorption (McDonald 1997).

2.2.5. Scanning Electron Microscopy

The surface morphology of the undyed and dyed samples was obtained via SEM (HI-TACHI TM-1000, Chiyoda, Japan) at a magnification of 800× using an acceleration voltage of 5 kV. All the samples were sputtered under vacuum with gold before the assessment.

2.2.6. FTIR

The FTIR spectra of dye extracted from dragon fruit and wool samples were measured using an AVATAR 380 instrument (Thermo Electron, Waltham, MA, USA) with a wavenumber range of 400–4000 cm\(^{-1}\) and a resolution of 1 cm\(^{-1}\).

2.2.7. Color Fastness Assessment

The International Organization for Standardization (ISO) standard test methods were used to evaluate the color fastness of the dyed wool fabrics for their wash, rubbing, light, and perspiration fastness properties. These methods included ISO 105-C10, ISO 105-X12, ISO 105-B02, and ISO 105-E04 [10].
2.2.8. Evaluation of UV Protection Properties

The UPF is a measurement of a fabric’s ability to resist UV rays. According to the EU standard 13758-2001, the UPF values of undyed and dyed samples were determined using a YG912E textile anti-ultraviolet tester \[11\]. The transmittance of both UV-A and UV-B radiation was measured across the wavelength range of 290 to 400 nm at 10 nm intervals. Every sample was tested five times at different locations, and the average value was reported.

2.2.9. Thermogravimetric Analysis

TGA was used to determine the degradation temperature of the samples because it provides information on the decomposition of samples. TGA was performed on undyed and dyed samples using a TQ 209 FI Libra® Netzch, GMBH, SELB, Germany analyzer by heating the dyed and undyed wool samples to 900 °C at an increasing rate of 20 °C/min under continuous nitrogen gas flow.

2.2.10. UV-Visible Spectral Analysis

Spectroscopy is a versatile technique used to detect the presence of molecules in a compound. A TU-1901 UV-Vis spectrophotometer (Taiyuan, China) was used in the current study. The prepared solution of dye extracted from dragon fruit was analyzed at wavelengths ranging from 200 to 800 nm.

2.2.11. Liquid Chromatography Mass Spectroscopy

To identify the degradation products from the dragon fruit extracted natural dye, the sample was analyzed by triple quadruple mass spectrometry (Xevo TQ-S cronos, SCIEX corporation, Milford, MA, USA). To separate the degraded products, a separate Terra C-18 capillary column was connected to the LC–MS instrument. The mixture of acetonitrile and water (70/30, v/v) was filtered through a 0.22 m Millipore syringe filter to create a mobile phase. The flow rate of the elute was maintained at 0.08 mL min\(^{-1}\), and the injection volume was 20 µL. The eluent from the chromatographic column was inserted into the UV–Vis diode array detector, the ESI interface, and the quadruple ion trap mass analyzer. In the mode of positive ions, MS analysis was carried out using a mass spectrometer attached to an ESI ion source. The ESI probe tip and capillary potentials were set at 2.5 kV and 25 V, respectively, by maintaining the mass range at 50–400 m/z. The temperature of the heated capillary was adjusted to 200 °C. The chemical structures of the obtained intermediate products were determined according to fragment analysis from peak-by-peak software.

2.2.12. Antibacterial Test

The American Association of Textile Chemists and Colorists (AATCC) 100-1999 was used to quantitatively assess the antibacterial activity of samples of both undyed and dyed wool against both \textit{E. coli} and \textit{S. aureus} bacteria. By evaluating the difference in colony counts between dyed and undyed samples during incubation, the antibacterial activity of the wool samples was quantified. The results are reported as a percentage of the inhibition ratio according to

\[
\text{Inhibition Ratio(\%)} = \left( \frac{A_{\text{control}} - A_{\text{sample}}}{A_{\text{control}}} \right) \times 100
\]

where \(A_{\text{control}}\) is the absorbance (or another relevant measure) of the control sample (without the inhibitor). \(A_{\text{sample}}\) is the absorbance (or another relevant measure) of the sample containing the inhibitor.
3. Results

3.1. Color Efficiency and Color Values of the Dyed Samples

Wool samples were pre-mordanted using copper sulfate, potassium dichromate, and stannous chloride. Table 1 shows the impact of the dyed wool samples with and without a mordant on the color coordinates L*, a*, b*, c, and h and color strength. In terms of mordanting, stannous chloride (SnCl2·2H2O) outperformed all other dyed samples, with a K/S value of 13.29. The lowest color efficiency (5.75) of all the mordants was displayed by the potassium dichromate (K2Cr2O7) mordant. Both the samples dyed without a mordant and copper sulfate (CuSO4·5H2O) showed almost identical K/S values of (9.93) and (8.17), respectively.

Table 1. Color characteristics of dyed wool fabrics with and without mordants.

<table>
<thead>
<tr>
<th>Mordant</th>
<th>K/S</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C*</th>
<th>h</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without mordant</td>
<td>8.17</td>
<td>43.66</td>
<td>45.62</td>
<td>5.26</td>
<td>45.93</td>
<td>6.57</td>
</tr>
<tr>
<td>K2Cr2O7</td>
<td>5.75</td>
<td>46.88</td>
<td>27.68</td>
<td>6.24</td>
<td>28.38</td>
<td>12.71</td>
</tr>
<tr>
<td>CuSO4·5H2O</td>
<td>9.93</td>
<td>38.12</td>
<td>28.98</td>
<td>17.30</td>
<td>33.75</td>
<td>30.84</td>
</tr>
<tr>
<td>SnCl2·2H2O</td>
<td>13.29</td>
<td>39.11</td>
<td>31.36</td>
<td>5.12</td>
<td>31.77</td>
<td>9.27</td>
</tr>
</tbody>
</table>

The color characteristics of dyed wool material depend on the affinity of the dye particles for the wool and the dyeing mechanism. As a result, wool materials dyed with dragon fruit extract showed great color efficiency when mordanting chemicals were used, particularly when stannous chloride was used as a mordant [12]. The color characteristics of the wool samples dyed with dragon fruit extract demonstrated that various color shades were produced using several different mordanting chemicals, as shown in Figure 1.

![Color shades obtained by wool samples after mordanting, dyeing, and control sample.](image)

According to the L*, a*, b*, C and h° values, when individually assessed, there was a slight increase in lightness when potassium dichromate was used as a mordant, whereas there was a decrease in lightness for the other mordant. Stannous chloride and the sample dyed without a mordant were slightly more red, showing 31.36 and 45.62 red (a*) values, whereas potassium dichromate and copper sulfate showed similar red (a*) values of 27.68 and 28.98, respectively. On the other hand, copper sulfate displayed the highest yellow (b*) value of 17.30. The color saturation values (C*) were found to be the highest in samples dyed without a mordant (45.93), while they were found to be the lowest in samples dyed with potassium dichromate (28.38). The samples dyed with copper sulfate had the highest hue angle of 30.84, whereas the lowest hue value was obtained for the samples dyed without a mordant [13].
3.2. Surface Morphology of Wool Fabric

SEM was used to examine the surface morphology of both dyed and undyed samples. Wool fibers are clearly composed of scaled and fibrillar components. Figure 2a shows the SEM image of undyed fabric, where the surface was found to be smooth and clean with normal fibrils, which are usually found on the fabric surface. Figure 2b–e shows the surface morphology of the dyed wool fabric with and without mordants. The dyed fabrics showed undamaged wool and the deposition of some dye particles on the wool surface. Hence, compared with undyed fabrics, natural dyes have more desirable interactions with the hair cuticle surfaces of wool. The energy-dispersive X-ray analysis (EDX) graph proved the presence of elements like carbon, nitrogen, and oxygen.

![SEM images](image-url)

**Figure 2.** SEM, EDX, and mapping images of (a) undyed wool, (b) wool dyed without mordant, and (c–e) wool dyed with different mordant, (f) carbon mapping, (g) nitrogen mapping, (h) oxygen mapping, and (i) mixed mapping.

3.3. FTIR

The FTIR spectra of the raw wool fabric, dye extracted from dragon fruit, wool fabric dyed without a mordant, and mordanted dyed wool fabric are shown in Figure 3, with the complexation reaction mechanism shown in Figures 3–6. The O-H and N-H stretching of the keratin extract’s ordered and folded structure was detected at 3100–3400 cm⁻¹. The presence of a triple band in the wavenumber range between 2800 and 3000 cm⁻¹ is attributed to C-H vibrations. The absorption bands of the raw wool at wavenumber 1623 are attributed to amide I, N-H bending.
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For the FTIR spectra of dye extracted from dragon fruit, the large peak at 3234 cm$^{-1}$ is also attributed to the O-H stretch. The peaks in the wavenumber range between 2800 cm$^{-1}$ and 3000 cm$^{-1}$ are attributed to CH and CH$_2$ vibrations. The peak at 1644 cm$^{-1}$ is attributed to C=O stretching vibrations, which appear as carbonyl groups for ketone structures. In dye extracted from dragon fruit, the active carbonyl and hydroxyl groups are normally carboxylic acids [14–17].

In all dyed samples, the absorption band of wool at wavenumber 1623 shifted to 1631, which clearly indicated successful dyeing [15]. The peak shift is attributed to the hydrogen bonding interaction between the amide I group and the carboxylic group of the betalain pigment of wool. The intensity of the peaks was greater for all dyed samples because of the presence of C=O groups and O-H groups in the betalains within dragon fruit.

**Figure 3.** FTIR spectra of raw wool, natural dye extracted from dragon fruit, wool dyed without mordant, wool dyed with copper sulfate mordant, wool dyed with potassium dichromate mordant, and wool dyed with tin chloride mordant.

**Figure 4.** Complex reaction mechanism of wool–potassium dichromate mordant for extracted natural dye from dragon fruit.
Figure 4. Complex reaction mechanism of wool–potassium dichromate mordant for extracted natural dye from dragon fruit.

Figure 5. Complex reaction mechanism of wool–copper sulfate mordant for natural dye extracted from dragon fruit.

Figure 6. Complex reaction mechanism of wool–stannous chloride mordant for natural dye extracted from dragon fruit.

3.4. Color Fastness Properties of Wool Samples Dyed with Dragon Fruit Extract

Table 2 shows the light, wash, perspiration, and rubbing properties of all wool samples dyed with and without a mordant. Compared to those of the un-mordant samples, the mordanting improved the overall fastness qualities of the mordanted samples [18–20].

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Table 2. Color fastness properties of dyed wool fabrics with and without mordants.

<table>
<thead>
<tr>
<th>Mordants</th>
<th>Light Fastness ISO 105-B02</th>
<th>Washing Fastness ISO 105-C10</th>
<th>Perspiration Fastness Acidic ISO 105-E04</th>
<th>Alkali ISO 105-E04</th>
<th>Dry ISO 105-X12</th>
<th>Wet ISO 105-X12</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without mordant</td>
<td>4</td>
<td>5–6</td>
<td>4–5</td>
<td>4–5</td>
<td>4–5</td>
<td>4</td>
</tr>
<tr>
<td>K(_2)Cr(_2)O(_7)</td>
<td>4–5</td>
<td>4–5</td>
<td>4–5</td>
<td>4–5</td>
<td>4–5</td>
<td>4</td>
</tr>
<tr>
<td>CuSO(_4)·5H(_2)O</td>
<td>4</td>
<td>4–5</td>
<td>4–5</td>
<td>4–5</td>
<td>4–5</td>
<td>4</td>
</tr>
<tr>
<td>SnCl(_2)·2H(_2)O</td>
<td>5–6</td>
<td>5–6</td>
<td>4–5</td>
<td>5</td>
<td>4–5</td>
<td>4</td>
</tr>
</tbody>
</table>

3.4.1. Light Fastness

All of the wool samples dyed with dragon fruit extract had excellent light fastness characteristics. The copper-sulfate-mordanted wool samples were comparatively less light-tolerant than the control, potassium dichromate, or stannous-chloride-mordanted dyed wool samples. This is probably due to the photolytic degradation and weak coordination bonding of copper sulfate with dye molecules. On the other hand, wool fabrics dyed with dragon fruit extract and stannous chloride as the mordant displayed an exceptional rating of light fastness. Overall, the findings show that the mordanting process increased the light fastness of the dyed wool fabrics in comparison to that of the samples dyed without a mordant.

3.4.2. Wash Fastness

From the wash fastness results in Table 2, all the dyed wool samples (un-mordanted and mordanted) showed good to excellent wash fastness ratings ranging from 4 to 6. The wool fabric dyed with stannous chloride as the mordanting agent showed the highest wash fastness rating compared to that of another mordant of copper sulfate and potassium dichromate. The wool sample dyed without a mordant showed an excellent wash fastness rating due to the affinity of wool fibers for coloring compounds in the form of strong hydrogen bonding and ionic bonding, respectively.

3.4.3. Perspiration Fastness

The results for the perspiration fastness of both the acidic and alkaline media of wool fabric dyed with dragon fruit extract are displayed in Table 2. The samples treated with and without a mordant displayed the same rating of 4–5 in an acidic medium. However, stannous chloride had the greatest effect on alkaline perspiration, followed by copper sulfate and potassium dichromate.

3.4.4. Rubbing Fastness

Rubbing fastness performance is based on the presence of unfixed dyes on the fiber surface of dyed fabrics. The dry rubbing fastness of potassium-dichromate-mordanted samples was relatively lower than that of un-mordanted and mordanted samples with copper
sulfate and stannous chloride, which was due to the weak co-ordination complexation of potassium ions. However, there was no difference between the results for the mordanted and un-mordanted wool fabrics in the wet rubbing test.

3.5. UV Protection

Because of their natural light resistance, textile fabrics are often utilized for UV protection. The UV-A band encompasses wavelengths between 315 and 400 nm, the UV-B band encompasses wavelengths between 280 and 315 nm, and the UV-C band encompasses wavelengths below 280 nm [21,22].

Table 3 provides the UPF (mean) rating and percentage values of UV-A and UV-B radiation transmission through wool fabric. All dyed samples with and without a mordant showed greater UPF values than the undyed samples. The wool samples dyed with stannous chloride mordant had the highest UPF values [16]. The enhanced UPF values with various metallic mordants may be caused by the metallic salts bridging the dye molecules and the variations in the phytochemical constituents of the mordanted samples. Hence, from the above UPF values, it can be concluded that wool material dyed with dragon fruit extract can block UV radiation [23,24].

Table 3. UV protection readings of undyed, dyed without mordant, and mordanted dyed wool fabrics.

<table>
<thead>
<tr>
<th>Sample</th>
<th>UPF (Mean)</th>
<th>UV (A %)</th>
<th>UV (B %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Undyed wool</td>
<td>298</td>
<td>2.87</td>
<td>0.08</td>
</tr>
<tr>
<td>Dyed without mordant</td>
<td>363</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>K₂Cr₂O₇</td>
<td>517</td>
<td>0.10</td>
<td>0.05</td>
</tr>
<tr>
<td>CuSO₄·5H₂O</td>
<td>680</td>
<td>0.05</td>
<td>0.08</td>
</tr>
<tr>
<td>SnCl₂·2H₂O</td>
<td>1058</td>
<td>0.58</td>
<td>0.05</td>
</tr>
</tbody>
</table>

3.6. Thermal Properties

The TG curves of dyed and undyed wool fabrics under a nitrogen atmosphere are shown in Figure 7 for three different stages of the weight loss pattern. Due to the hydrophilicity of the wool fibers, the initial weight loss was attributed to the loss of water from the wool between 50 °C and 170 °C. The second stage of degradation occurred between 200 °C and 400 °C. The breakdown of the helical structure of wool fibers and the thermal degradation of keratin, which occurred at 230 °C and where the maximal decomposition rate occurred, may be referred to as the degradation process. The third stage of degradation occurred above 800 °C and was attributed to the loss of several side chains from the wool peptides.

Figure 7. TGA curves of undyed and dyed wool fabric with dragon fruit extract.
3.7. Absorption Spectra

Figure 8 shows the absorption spectra of dye extracted from dragon fruit. The dye extracted from dragon fruit using ethanol resulted in a deep-colored solution. Certain plants (such as beets) and cacti (such as dragon fruit) have pigments called betalains, which are soluble in water and contain nitrogen. Both the flesh and skin of dragon fruit are red because of the presence of betalains. The use of dragon fruit extract as a natural food coloring agent may also be influenced by the amount of betalains in it. However, the kind and level of maturity of the fruit may affect the amount of betalains in dragon fruit. In general, betalains are found in larger amounts in redder breeds.

![Dye absorption spectrum of dragon fruit dye in the range of 400 nm to 700 nm.](image)

The UV-Vis spectrum showed that the dragon fruit extract had a limited band covering the 500–560 nm region, which is consistent with its dark pink color. This absorption range of 500–560 nm confirmed the presence of betalain pigments in the dragon fruit dye. A group of red and yellow tyrosine-derived pigments called betalains are present in plants, which include reddish to dark violet betalain pigments with antioxidant properties that exert various shades of violet color in wool fabric samples dyed with different mordants [25–28].

3.8. Identification of Compounds

The degraded compounds formed during the photo degradation process were identified from the LC-MS test. LC-MS may act as a valuable tool for the analysis of compounds present in the natural dyes. Figures 9 and 10 describes the chromatogram and mass spectra obtained after the degradation of compounds. The presence of the betacyanin compound was found at an m/z value of 360.0827, forming the biggest peak among all the compounds. The structure of betacyanin was identified by fragment analysis using peak view software. As the compounds are highly sensitive to photo degradation, it was observed that all the compounds disappeared after 9 h of photolytic treatment due to the shift in the retention time of the parent compound owing to the changes in the ionic strength of the sample solutions.
Figure 9. Chromatogram of dragon fruit dye.

Figure 10. Mass spectra of degraded compounds.
3.9. Antimicrobial Properties

As shown in Figure 11, the antibacterial test was carried out according to AATCC 100-1999 test standards. The wool samples were placed inside the Petri dishes during the test. After completing the test, the wool samples were taken out to for colony counting and to ensure the visibility of the number of colony counts [29,30]. The antimicrobial activities of undyed, dyed without a mordant, and mordanted dyed wool samples were examined against both E. coli and S. aureus bacteria. The calculated results are shown in Figure 11 and Table 4. The test results prove that approximately a 90–95% bacterial reduction was achieved after dyeing wool fabric with dragon fruit extract. The dyed wool samples exhibited greater bactericidal effects against E. coli than against S. aureus due to structural differences in the bacteria. There was no bacterial reduction in the undyed samples, whereas the mordanted dyed wool samples showed the greatest bacterial reduction percentage among the samples dyed without a mordant. As mordanting improves the stability of the dye in fabric, many metallic mordants inhibit the growth of bacteria and kill them at very low concentrations [31].

![Figure 11. Antibacterial activity of undyed and dyed wool fabrics with and without mordant against E. coli and S. aureus.](image)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Microbial Growth</th>
<th>Bacterial Reduction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>E. coli</td>
</tr>
<tr>
<td>Undyed wool</td>
<td>Completely Present</td>
<td>-</td>
</tr>
<tr>
<td>Dyed without mordant</td>
<td>Partially present</td>
<td>91</td>
</tr>
<tr>
<td>Potassium Dichromate</td>
<td>Partially present</td>
<td>94</td>
</tr>
<tr>
<td>Copper Sulfate</td>
<td>Partially present</td>
<td>95</td>
</tr>
<tr>
<td>Stannous Chloride</td>
<td>Partially present</td>
<td>94</td>
</tr>
</tbody>
</table>

The main reason for the antimicrobial activity of dragon fruit was the presence of antimicrobial groups, such as phenolic and non-phenolic compounds. Phenolic compounds, such as quercetin, affect S. aureus through protein denaturation, which leads to the inactivation of enzymes and the inhibition of bacterial metabolism. However, non-phenolic compounds such as saponins and steroids interact with the phospholipid membrane of bacteria and reduce cell wall combinations, which causes cell death [32].

4. Conclusions

The natural dye was successfully extracted from dragon fruit by ultrasonication and applied to wool fabrics. Using several mordanting chemicals, the dyed wool samples
produced a variety of colors, ranging from light pink to dark pink and light brown with great color strength. The morphology and chemical structure of the undyed and dyed wool samples were determined by SEM and FTIR, and the accuracy of the results of these processes was proven. The MS technique coupled with chromatographic separation was used for the identification and accurate determination of betacyanin compounds in complex matrices, eliminating the need for cleanup procedures. Using ISO test methods, all the dyed samples demonstrated good to exceptional color fastness properties in light, wash, perspiration, and rubbing fastness tests. However, the wool fabric dyed with stannous chloride as the mordanting agent showed excellent fastness properties. The antimicrobial properties of dragon-fruit-dyed wool samples were compared with those of undyed fabric samples against *E. coli* and *S. aureus* bacteria. The undyed wool fabric showed no bacterial reduction, while the fabrics dyed with or without a mordant exhibited the greatest bacterial reduction against both *E. coli* and *S. aureus*. Despite this, the addition of mordants to the wool samples resulted in a greater bacterial reduction than that in the non-mordanted wool samples. The antimicrobial activity of these dyed wool fabrics will be a valuable addition to products such as antimicrobial textiles for medical applications and other end uses.

As a result, this research developed a brand new source of dye extracted from dragon fruit and demonstrated vibrant shades of color along with superior functional properties when applied to wool fibers. This study paves the way for the use of natural dyes in the textile dyeing industry for a cleaner production of sustainable textiles. In the future, several other derivatives of plants can be utilized to extract natural dyes for the sustainable dyeing of wool material to replace synthetic dyeing, resolving environmental issues in the textile wet processing industry.

**Author Contributions:** T.Z. supervised the research; M.K.S. performed the experiments and wrote this research paper; X.D. developed the FTIR and TGA graphs using the data; A.P. and F.S. reviewed this research paper. S.R.I. helped to solve the comments in major revision. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** The data underlying this article will be shared upon reasonable request to the corresponding author.

**Acknowledgments:** This research was carried out in the Key Laboratory of Science & Technology of the Eco-Textile of Donghua University. This work is dedicated to Zhao Tao.

**Conflicts of Interest:** The authors declare no conflicts of interest.

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