

Valorization of Sulfonated Kraft Lignin as a Natural Dye for the Sustainable Dyeing of Wool Fabrics: Effect of Peroxide Oxidation

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ABSTRACT: Lignin is an abundant and complex biopolymer with inherent color but has only a few applications. It has the potential to replace synthetic acid dyes for the coloration of wool fibers in brown shades, as traditional dyeing with acid dyes produces toxic effluent needing costly treatment. In this work, the valorization of sulfonated kraft lignin was explored by using it as a natural dye for the coloration of wool fabrics alone and with hydrogen peroxide (H₂O₂). The dyed fabrics were characterized by reflectance and diffuse reflectance spectroscopies to assess the color yield and UV protection performance respectively. The dyed fabrics exhibited reasonably good color yield and satisfactory colorfastness to washing (Grade 3/4), which improved to Grade 4 by the oxidation treatment with H₂O₂. The SEM images show that when fabrics are dyed alone with lignin, deposition of lignin particles is visible on the fiber surface, but with hydrogen peroxide, no deposition of lignin is visible on the fiber surface, and the color strength of the fabric became almost double. The UV light transmission through the fabric decreased from 6.73 and 13.23% at 311 and 365 nm to 2.19 and 5.23% respectively. The ATR-FTIR spectra of the dyed fabrics showed increased absorption of lignin sulfonate by the fabric, suggesting depolymerization of lignin by H₂O₂ to smaller macromolecules easing absorption into the wool fiber. Lignin sulfonate could be a cheap and sustainable alternative to harmful synthetic dyes for producing brown shades on wool fabric.

KEYWORDS: *lignin sulfonate, wool fiber, sustainable dyes, textile dyeing, colorfastness to washing, color yield*

1. INTRODUCTION

Wool fibers have many beneficial properties, such as high warmth, unique surface texture due to the presence of scales, good fire retardancy, and bacterial resistance.¹ Synthetic acid dyes are the most popular dye for the coloration of wool fabrics, and a small portion of wool fibers are dyed with reactive dyes. Synthetic dyes provide wool fabric with vibrant colors, but many of the dyes used for wool fiber coloration could be toxic or harmful to the environment.² The use of toxic dyes in textiles could be harmful to the wearer as they can migrate to the skin when a person sweats or the cloth becomes wet, causing skin sensitization or allergic effects.^{3,4} Most of these dyes are not biodegradable, and their persistent color affects photosynthesis reactions that are necessary for the survival of aquatic plants and organisms. When the effluent is discharged to watercourses, it affects the penetration of light into the water and does not allow photosynthesis reactions.⁵ Dyes derived from plants and fruits are generally considered biodegradable, and therefore they are envisaged as sustainable alternatives to toxic and fossil fuel-derived synthetic dyes.

Mostly tannin and other polyphenols extracted from barks, leaves, flowers, fruits, and fruit skins have been studied for the dyeing of wool. Tannins extracted from barks of various plants, including Ziziphus,⁶ cinnamon,⁷ walnuts,⁸ *Azolla pinnata* extract,⁹ and *Cassia reingera*,¹⁰ are studied for the dyeing of wool fiber. Natural dyes extracted from leaves of pomegranate,¹¹ *Berberis thunbergia*,¹² hibiscus flowers,¹³ *Hibiscus rosa-sinensis*,¹⁴ *Crocus sativus*,¹⁵ skins/peel of *Citrus sinensis* L,¹⁶ and various fruits including myrobalan,¹⁷ chamomiles,¹⁸ and peanut shell,¹⁹ are also studied as an alternative to synthetic dyes. Anthocyanin pigments extracted from peels/skins of various fruits, including *Brassica oleracea* L.,²⁰ purple sweet potatoes,²¹ red cabbages,²² and dragon berries,²³ have been studied for the dyeing of wool that produces a variety of colors. Tannins and other flavonoids are advantageous as some of them have antioxidant and antibacterial properties that make the treated fabric multifunctional.²⁴ Tannins mainly produce a dull brown color shade on wool fabrics. The anthocyanin extracted from various fruit skins produces a variety of colors. However, colorfastness to light and washing are the key bottlenecks for their industrial application as an alternative to synthetic dyes.

Lignin is a plant-derived biopolymer with a complex chemical structure and various molecular weights found in the cell walls of woody plants. It is the second-most abundantly available natural polymer after cellulose. The worldwide production of lignin has been estimated to be 100 million tons per year, which is expected to reach 225 million tons per year by 2030.²⁵ It is a byproduct of the pulp and paper industry, and lignosulfonate lignin accounts for 89% of the lignin production, and the rest is kraft lignin. It has very limited applications, and therefore its disposal is a nightmare for the pulp and paper industries, which warrants the development of its novel and high-value applications. A water-soluble lignin that contains a large number of sulfonate groups (lignosulfonates) is prepared from the by-products of the sulfite pulping process. Lignin is sulfonated, degraded, and solubilized for further modifications or applications. The weight-averaged molecular weight of the lignosulfonate polymers is from 5 to 400 kDa compared to 1 to 5 kDa for the kraft lignin.²⁶ Currently, lignin is mostly used to produce bioenergy,²⁷ but has also been studied to make fuel cell anode,²⁸ supercapacitor cathode,²⁹ flocculant,³⁰ thermoplastic adhesive,³¹ biodegradable composites,³² biodegradable packaging,³³ dye dispersant,³⁴ and adsorbents for the removal of heavy metals³⁵ and dyes³⁶ from textile dyeing effluent. Lignin is biodegradable under a compost environment by thermophilic micro-fungi and actinomycetes, and the optimum temperature for thermophilic fungi is 40–50°C which is also the optimum temperature for lignin degradation in compost.³⁷

Lignin is a colored material that has the potential to be used for the coloration of textiles and can be an alternative to anionic acid dyes for dyeing wool fibers, particularly in brown shades, as sulfonated lignin itself is anionic. Kraft lignin was also studied for the sustainable coloration of leather.³⁸ Even films made from lignin-derived composites were found biodegradable,³⁹ suggesting that lignin's addition to other polymers does not affect their biodegradability. However, its application as a green natural dye for the sustainable coloration of textiles was never realized. However, lignin is a high-molecular-weight polymer that limits its absorption into textile fibers, and normal lignin is insoluble in water. Therefore, depolymerization is necessary to decrease its molecular weight to ease the absorption of lignin into textile fibers. Lignin is weakly anionic because of the presence of many polyphenolic groups and has poor substantivity toward textile fiber. Sulfonated lignin has a higher degree of sulfonation than kraft lignin, and they are anionically charged due to the presence of sulfonate groups.⁴⁰ Sulfonation of lignin increases its water solubility as well as permits it to bind to cationic textile fibers like wool by forming ionic bonds. Therefore, the replacement of costly synthetic acid dyes with lignin sulfonate for specifically producing brown shades is not only a sustainable solution but also can result in the valorization of kraft lignin. However, lignin and sulfonated lignin were never studied for the coloration of textiles. In this article, for the first time, we are reporting color yields, shade produced, and colorfastness to washing of wool fabric dyed with sulfonated lignin alone and in the presence of

hydrogen peroxide. This article also reported the effect of peroxide oxidation on the color yield and colorfastness to washing of the treated wool fabrics.

2. RESULTS AND DISCUSSION

2.1. Shades produced. Figure 1 illustrates shades produced by wool fabrics treated with lignin sulfonate at 4, 6, 8, and 10% on the weight of fiber (owf) alone and also in the presence of 1.67 mL/L H₂O₂. The depth of shades progressively increased with an increase in the applied dosage of lignin sulfonate when the fabrics were dyed with lignin sulfonate alone. However, for the dyeing with lignin sulfonate in the presence of H₂O₂, the depth of shade considerably increased compared to the samples dyed with lignin alone. The shade produced by 10% (owf) lignin sulfonate alone was lighter than the shade produced by 4% lignin in the presence of 1.67 mL/L H₂O₂. For the dyeing of wool fabric with lignin sulfonate in the presence of H₂O₂, the absorption of lignin sulfonate reached saturation up to 8% (owf) lignin sulfonate and only a marginal increase in depth of shade was observed when the applied dosage of lignin sulfonate was increased from 8 to 10% (owf) in the case of dyeing with H₂O₂.



Figure 1. Photographic images of wool fabric treated with 4, 6, 8, and 10% (owf) sulfonated lignin without (top, left to right) and with (bottom, left to right) 1.67 mL/L H₂O₂.

2.2. CIE L*a*b* values and color yields. We studied the effect of treatment pH, lignin sulfonate concentration, and peroxide oxidation on the CIE L*a*b* values and color yields of wool fabrics dyed with lignin sulfonate.

2.2.1. Effect of Treatment pH on CIE L*a*b* Values and Color Yields. The CIE L*a*b* values of wool fabrics

dyed with 8% (owf) lignin sulfonate at various pHs are presented in Table 1. The L^* value represents whether the shade produced is lighter or darker. The lower the L^* value, the darker the shade produced. The fabric treated at pH 3 produced the lowest L^* value and the L^* of the fabric dyed with lignin increased with an increase in the treatment pH, i.e., the shade produced became lighter with an increase in the treatment pH. The fabric treated at pH 3 and 10 produced the darkest and the lightest shade. The value of a^* represents the yellowness/greenness of the shade produced. The value of a^* decreased with an increase in pH from 5.63 for pH 3 to 1.8 for pH 7, after which the a^* started increasing, indicating that the yellowness of the shade decreased with an increase to pH 7 and then again, the yellowness of the shade started increasing up to pH 10. The b^* value represents the redness or blueness of the fabric. The value of b^* also decreased from 23.37 for pH 3 to 14.4 for pH 7 and then again started increasing with an increase in pH, suggesting the redness of the shade decreased with an increase in pH until pH 7 and then again, the redness of the shade started increasing with an increase in pH.

Table 1. Effect of lignin dosage and treatment pH on the CIE $L^*a^*b^*$ values, color difference, and color yield.

Treatments	CIE $L^*a^*b^*$ values			Color difference (ΔE)	Color strength (K/S)
	L^*	a^*	b^*		
Effect of pH					
3	85.54±0.18	5.63±0.05	23.37±0.09	16.39	1.35
4	86.72±0.29	4.14±0.12	20.78±0.10	13.64	1.29
5	87.62±0.60	3.55±0.06	18.44±0.14	12.09	1.15
7	91.13±0.21	1.8±0.02	14.4±0.08	6.93	0.84
8	91.17±0.25	2.09±0.02	15.62±0.02	6.81	0.82
10	91.8±0.13	2.32±0.05	17.94±0.21	6.74	0.80
Effect of lignin dosage (% owf) without H ₂ O ₂					
2	91.46±0.03	4.02±0.04	20.28±0.20	10.19	0.92
4	90.02±0.09	4.38±0.10	21.13±0.04	12.09	1.08
6	88.19±0.15	4.96±0.03	22.47±0.04	14.4	1.21
8	86.58±0.19	5.51±0.04	23.03±0.02	15.68	1.35
10	85.58±0.26	5.62±0.15	23.5±0.20	16.87	1.48
12	83.72±0.29	6.11±0.05	24.54±0.12	18.62	1.53
Effect of lignin dosage (% owf) in the presence of H ₂ O ₂					
2	85.51±0.18	5.43±0.05	23.27±0.09	16.19	1.17
4	71.69±0.11	4.63±0.03	20.97±0.06	20.5	1.76
6	69.38±0.08	5.91±0.09	22.66±0.03	22.4	2.19
8	67.85±0.10	5.65±0.07	22.42±0.06	24.1	2.38
10	66.59±0.11	5.39±0.04	22.09±0.05	25.7	2.45
12	66.09±0.07	5.31±0.05	11.95±0.08	25.9	2.47

The effect of treatment pH on the color difference between the undyed and wool fabrics dyed with various concentrations of lignin is presented in Table 1. The color difference (ΔE) compared to the untreated wool fabric decreased with an increase in the pH suggesting the depth of color of the fabrics decreased with an increase in the treatment pH. Figure 2 shows the effect of pH on the color strength of wool fabrics treated with 8% owf lignin from pH 3 to 10. At all pHs, lignin showed the highest color strength at 400 nm, and the color strength increased with an increase in pH. The highest color strength (1.35) was exhibited at pH 3 and the lowest at pH 10. The isoelectric point of wool fiber surface in an aqueous solution is 4.5, i.e., below pH 4.5, the surface of wool fiber is cationic, and above 4.5, it is anionic. Below pH 4.5, the lower the pH is, the more the fiber surface is cationic. Similarly, above pH 4.5, the higher the pH is, the more the surface of wool fiber is anionic. Therefore, at pH 3, wool fibers absorbed the highest amount of lignin, and the lignin absorption decreased with an increase in pH. The results obtained suggest that pH 3 is the optimum pH for dyeing wool fabrics with lignin sulfonate, as in acidic conditions, wool fiber is quite safe from damage.¹

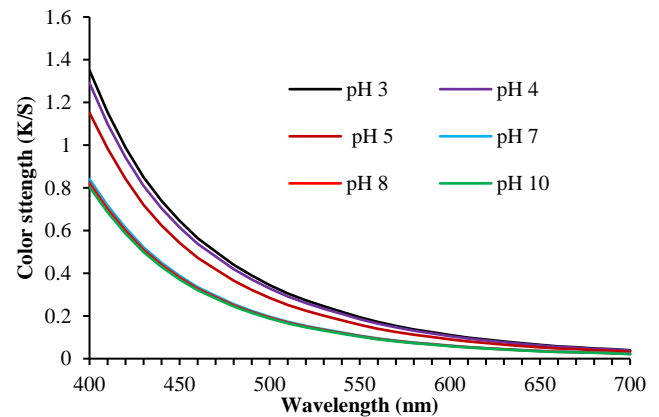


Figure 2. Effect of pH on color strength of wool fabric treated with 8% (owf) lignin sulfonate at 98 °C for 60 min.

2.2.2. Effect of Lignin Sulfonate Concentration on CIE $L^*a^*b^*$ Values and Color Yields. Table 1 shows the CIE $L^*a^*b^*$ values of wool fabric dyed with various concentrations of lignin alone. The lightness value (L^*) decreased with an increase in the concentration of lignin, indicating the depth of shade increased with an increase in the concentration of lignin. The values of a^* and b^* also increased with an increase in the concentration of lignin, suggesting the yellowness and redness of the shade also increased with an increase in the concentration of lignin.

Figure 3(a) shows the effect of lignin sulfonate concentration on the color strength of wool fabric dyed at pH 3 for 90 min. The color strength increased with an increase in lignin concentration. The lowest and the highest color strength was exhibited by the wool fabric samples treated

with 2 and 12% (owf) lignin sulfonate, respectively. The increase in the concentration of lignin increases the quantity of lignin absorbed by the fiber, and therefore the color strength increases. The overall color strength produced by the highest concentration of lignin applied is still low, suggesting producing lighter shades only, which could be sufficient for various apparel applications. Table 1 also shows that the depth of color increased with an increase in the concentration of lignin as the color difference between the undyed control wool fabric and the wool fabrics dyed with lignin increased with an increase in lignin sulfonate dosage.

2.2.3. Effect of Peroxide Oxidation on CIE $L^*a^*b^*$ Values and Color Yields. Table 1 shows the CIE $L^*a^*b^*$ values of wool fabric treated with various concentrations of lignin sulfonate in the presence of H_2O_2 . The value L^* decreased with an increase in the concentration of lignin, but a considerable decrease in the L^* values continued to a lignin sulfonate concentration of 8% owf, after which only a small decrease was observed, suggesting that the depth of the shade increased with an increase in lignin sulfonate concentration up to 8% owf and for which only a small increase in depth of shade was observed with an increase in the lignin concentration. It is also evident that the depth of color increased with an increase in the lignin concentration to 12% owf as the color difference (ΔE) increased with an increase in lignin dosage, but the color difference in the case dyeing in the presence of peroxide was considerably higher compared to the dyeing without it suggesting peroxide oxidation considerably increased the color intensity.

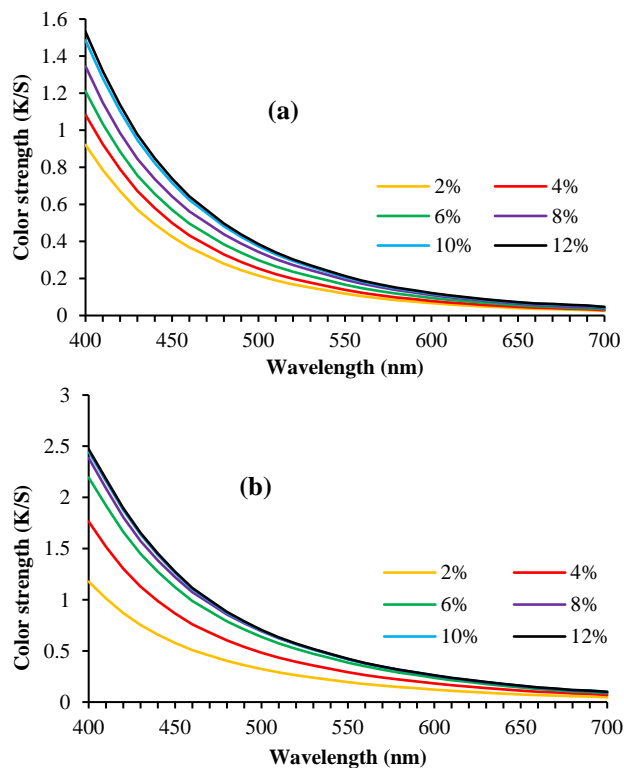


Figure 3. Color yield of wool fabrics dyed with various concentrations of lignin sulfonate without (a) and with (b) hydrogen peroxide.

Figure 3(b) shows the color strength vs. wavelength curves of wool fabric treated with various concentrations of lignin in the presence of 1.67 mL/L H_2O_2 at pH 3. The color strength quite dramatically increased when the dyeing of lignin was carried out in the presence of H_2O_2 . The color strength produced by 4% owf lignin in the presence of 1.67 mL/L H_2O_2 was 1.76, which is higher than the wool fabric treated with 12% owf lignin sulfonate alone (1.53). Figure S1 (Supporting Information) shows the effect of lignin sulfonate dosage on the color strength of the fabric treated with and without H_2O_2 . For the dyeing with lignin sulfonate without H_2O_2 , the color strength gradually increased with an increase in applied dosage up to 10% owf, and only a marginal increase was observed with a further increase in dosage but for dyeing with H_2O_2 , the color strength of the fabric increased until 8% owf and wool fibers were saturated with lignin sulfonate at 8% owf. Further increase in lignin sulfonate dosage only marginally increased the color strength of the treated fabric.

It is known that at acidic conditions, H_2O_2 can cause depolymerization of non-phenolic lignin structures.^{41,42} Lignin sulfonate used in this work has an average molecular weight of $\sim 18,000$, and peroxide oxidation probably degraded them forming lower molecular weight fragments. They are first absorbed by wool fiber and diffused into the interior of the fiber, and the comparatively higher molecular weight components are absorbed later onto the surface of wool fibers. The considerable increase in color intensity of dyed wool fabrics with lignin sulfonate with H_2O_2 compared to the wool dyed with lignin sulfonate alone suggests considerable depolymerization of lignin, which allowed its higher absorption into wool fibers. UV-visible spectra (Figure S2 in Supporting Information) of lignin sulfonate solution non-oxidized and oxidized with H_2O_2 show that the absorbance of oxidized lignin sulfonate solution increased compared to the solution of non-oxidized lignin sulfonate suggesting formation of new compounds by depolymerization of lignin and condensation of depolymerized lignin that absorbs visible lights, i.e., oxidized lignin sulfonate has higher color intensity compared to the non-oxidized lignin sulfonate. Therefore, the fabric dyed with lignin sulfonate in the presence of H_2O_2 exhibited higher color strength compared to the wool fabric treated with lignin sulfonate without the addition of H_2O_2 .

2.2.4. Colorfastness to Washing. Table S1 (Supporting Information) shows the colorfastness to washing of wool fabric dyed with lignin with and without hydrogen peroxide. It is evident that the colorfastness grade for the dyeing of wool fabrics with various concentrations of lignin was between Grade 2/3 and Grade 3/4, and the colorfastness to washing grade decreased with an increase in lignin dosage suggesting that more lignin was adsorbed rather than absorbed when the lignin dosage was increased, and the surface deposited lignin was removed by washing decreasing the colorfastness to washing. Overall, the colorfastness to washing achieved is reasonable compared to wool fabrics dyed with other natural dyes. Table S1 (Supporting Information) also shows the colorfastness to washing of

wool fabrics dyed with various concentrations of lignin in the presence of H_2O_2 . The colorfastness to washing slightly improved to Grade 4 after the oxidation treatment, which suggests that excellent absorption of lignin into wool fibers was achieved, increasing the colorfastness to washing. The colorfastness to washing achieved for the lignin sulfonate dyed wool fabrics is quite satisfactory as wool fabrics dyed with natural dyes also show similar colorfastness to washing grade.^{22,23}

2.3. Mechanisms of Dyeing. The mechanism of dyeing wool fiber with lignin sulfonate is presented in Figure 4. Wool fiber keratin protein contains amino, carboxyl, and hydroxyl functional groups. At acidic conditions below pH 4.5, anionic lignin sulfonate is attracted by cationic wool keratin and forms an ionic bond with the amino groups of keratin like the dyeing of wool fabric with anionic dyes.^{43,44} On the other hand, the hydroxyl groups of lignin sulfonate form hydrogen bonding with the amino, carboxyl, and hydroxyl groups of wool keratin, but the hydrogen bond is much weaker compared to the ionic bond. Therefore, lignin sulfonate is not only bonded to wool fiber by ionic bonding but also with hydrogen bonding providing good colorfastness to washing.

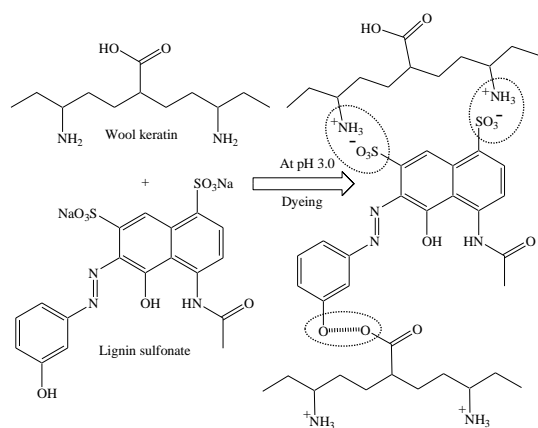


Figure 4. Mechanisms of dyeing of wool fiber with lignin sulfonate.

2.4. EDX Elemental Analysis. Figure 5 shows the EDX spectra and elemental analysis results of wool fabrics dyed with various concentrations of lignin sulfonate without hydrogen peroxide. Wool fiber has C, N, O, and S, but lignin sulfonate has only C, O, and S. Therefore, the addition of lignin to wool fiber should show a decrease in the N content but an increase in the O and S contents of wool fiber. Figure 5 shows that the C, O, N, and S content of untreated wool fabric is 51.63, 22.89, 23.29, and 2.70, which is consistent with published results.⁴⁵ As expected, the N content decreased, and the O and S content increased for the wool fabric treated with lignin sulfonate, suggesting increased absorption of lignin into the wool fiber. The N content decreased from 22.38 to 18.78, but the S and O contents increased to 4.38 and 25.08 for the wool fabric treated with 10% (owf) lignin sulfonate. The results indicate the presence of lignin in the treated wool fabrics.

Figure S3 (Supporting Information) shows the elemental distribution of C, N, O, and S on the surface of untreated and lignin sulfonate-treated wool fabrics measured by EDX. The elemental mapping of the control wool fabric surface shows that all elements are uniformly distributed on wool fiber surfaces. A similar phenomenon was observed for wool fabric treated with various concentrations of lignin sulfonate, indicating very uniform dyeing of wool fabric with lignin sulfonate.

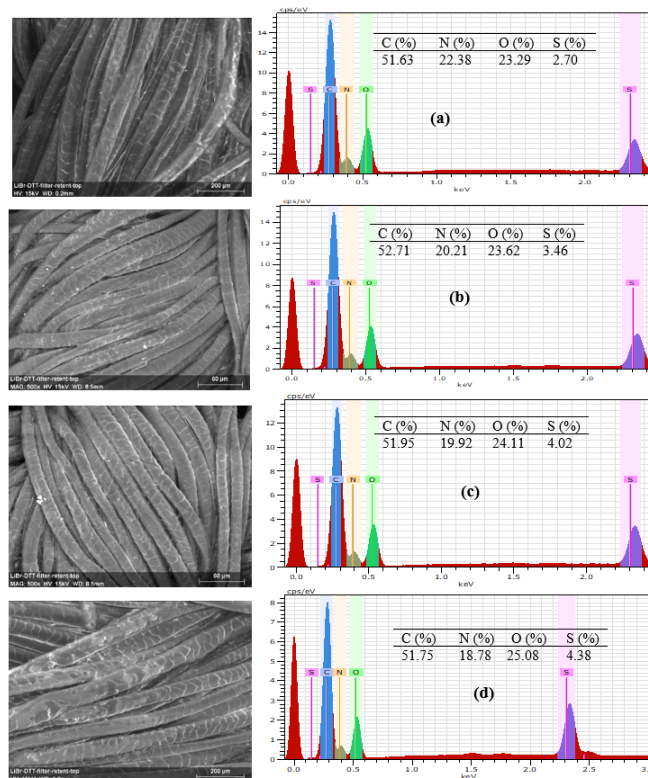


Figure 5. EDX spectra of wool fabrics treated with 0 (a), 4 (b), 8 (c), and 10% (d) (owf) lignin sulfonate.

2.5. UV transmission through the treated fabric.

Figure S4 (Supporting Information) shows the UV light transmission through the untreated control wool fabric and also wool fabric treated with 10% owf lignin sulfonate in the presence of 1.67 ml/L H_2O_2 . To measure the UV protection capability, UV transmission through the fabric is usually measured at 290 and 340 nm, which represent the UV transmission in UV-B and UV-A regions, respectively of the solar irradiation. The UV-C is the most dangerous UV region but most of the UV-C produced by solar irradiation is absorbed by the earth's atmosphere and does not come to the earth's surface. The UV radiation transmission through the untreated fabric is quite high as the UV transmission at 311 and 365 nm was 6.73 and 13.23% respectively. However, for the untreated fabric, it reduced to 2.19 and 5.23% respectively for the fabric treated with 10% owf lignin sulfonate. The UV protection capability achieved for the wool fabric treated with 10% lignin sulfonate is quite satisfactory and comparable to the fabric treated

with natural tannins but not as good as UV protection offered by wool fabrics treated with Ag nanoparticles.⁴⁵

2.6. ATR-FTIR Spectral Analysis. Figure 6 shows the ATR-FTIR spectra of wool fabrics treated with various concentrations of lignin sulfonate. The spectrum of control wool fabric shows typical characteristic IR bands at 1363, 1547, and 1657 cm^{-1} that are associated with the amide (III), amide (II), and amide (I), respectively. It also shows IR bands at 2842, 2910, and 2970 cm^{-1} and these IR bands are associated with the C-H stretching in methyl and methylene groups and the CH_2 asymmetric vibration of wool keratin protein, respectively. The large IR band at 3287 cm^{-1} represents hydroxyl groups present in wool keratin protein.⁴⁶ The wool fabric treated with lignin sulfonate also shows the same IR bands, but their intensity increased with an increase in the lignin content in wool, and also, the peak moved towards lower wavenumber, suggesting an interaction of lignin sulfonate with wool keratin protein. Moreover, the ATR-FTIR spectra of wool fabrics treated with lignin sulfonate show an extra IR band at 1040 cm^{-1} , which is associated with the sulfonate groups of the sulfonated lignin.⁴⁷ It indicates the presence of lignin in the treated wool fabric samples. The intensity of this band at 1040 cm^{-1} increased with an increase in the lignin sulfonate concentrations suggesting that with the increase in the lignin sulfonate concentrations, its absorption into wool fabric also increased.

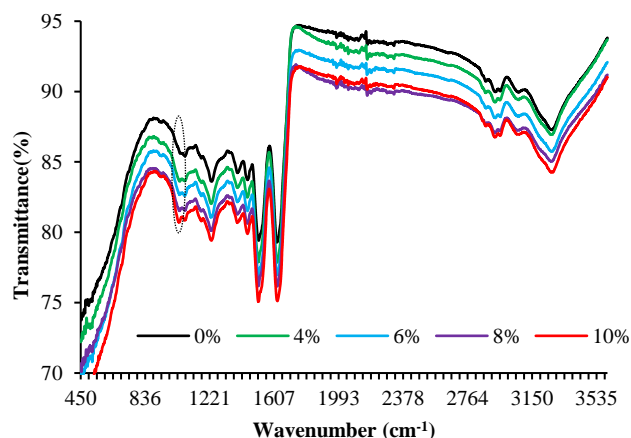


Figure 6. ATR-FTIR spectra of wool fabrics treated with various concentrations of lignin sulfonate.

It is reported that the oxidation of lignin with hydrogen peroxide at elevated temperatures depolymerizes lignin and introduces carboxyl groups in lignin.⁴⁸ Figure S5 (Supporting Information) shows the ATR-FTIR spectra of wool fabrics treated with 10% owf lignin sulfonate without and with the presence of 1.67 mL/L H_2O_2 . The intensity of the hydroxyl band increased for the fabric treated with lignin sulfonate compared to the control wool, as lignin has many hydroxyl groups. However, the intensity of the hydroxyl band for the wool fabric treated with lignin without and with hydrogen peroxide is the same. The intensity of

the C=O deformation band at 1230 cm^{-1} is also the same, indicating no formation of carboxyl groups in the peroxide oxidation of lignin.⁴⁹ The sulfonate group-related IR band at 1040 cm^{-1} is considerably higher for the wool fabric dyed with lignin with H_2O_2 compared to the fabric treated with the same concentration of lignin without H_2O_2 suggesting increased absorption of lignin sulfonate into wool fibers with an increase in the applied dosage of lignin sulfonate. The results suggest that the peroxide oxidation increased the depolymerization of lignin, increasing their absorption into wool fiber and therefore the intensity of the sulfonate-associated IR band at 1040 cm^{-1} was increased. In summary, the cause of the increase in the color intensity of wool fabric dyed with lignin sulfonate in the presence of H_2O_2 was due to the combined effect of increased lignin sulfonate absorption into wool fiber and also because of the increase in the color intensity of lignin sulfonate by peroxide-oxidation.

2.7. Surface morphologies. Figure 7 shows the surface morphologies of wool fibers dyed with various concentrations of lignin sulfonate. The undyed wool fabric shows a typical wool fiber surface with many scales on the surface of fibers and a few pieces of debris of damaged scales, which is obvious for non-shrink-resist treated wool fibers. The dyeing treatment of wool with lignin also did not cause any damage to wool fibers. It is evident that on the surface of wool fabric treated with 4% (owf) lignin sulfonate, no surface deposition of lignin is visible as the surface of fibers looks quite clean, but for increased lignin sulfonate dosages, 8 and 12% (owf) lignin sulfonate, a few depositions of lignin are visible, suggesting that the applied lignin sulfonate was only partially absorbed into fiber and the rest deposited on the wool fiber surface, thereby showed decreased colorfastness to washing for the wool fabrics treated with high concentrations of lignin sulfonate.

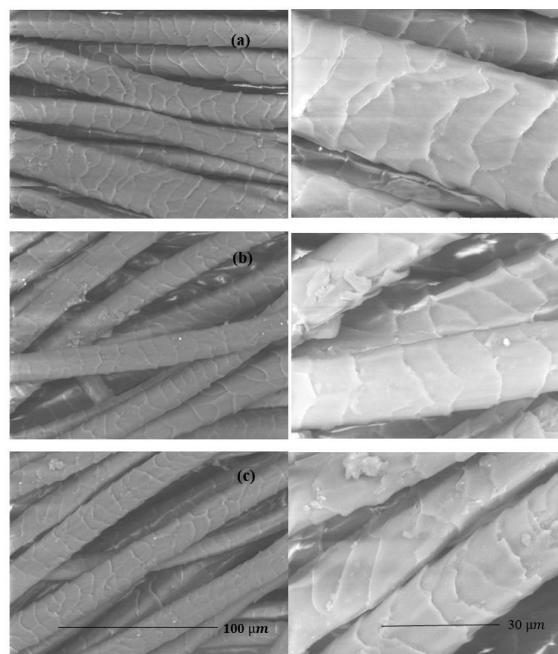
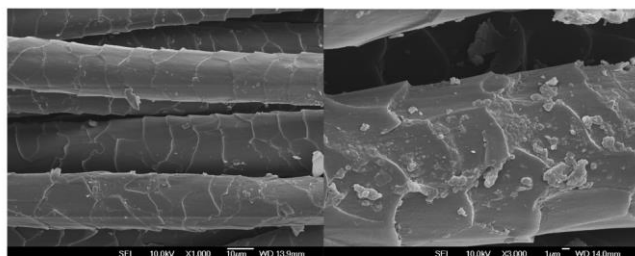
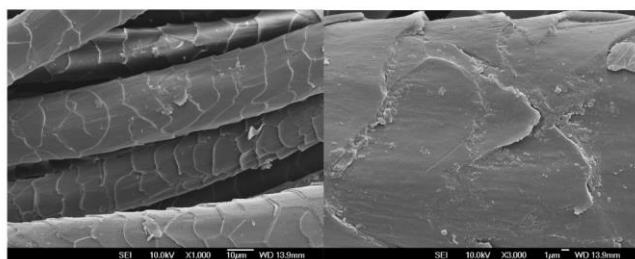


Figure 7. SEM optical micrographs of wool fabric surface dyed with 4(a), 8(b), and 12(c)% (owf) lignin sulfonate.

Figure 8 shows the SEM micrographs of wool fabric treated with 10% (owf) lignin sulfonate with and without hydrogen peroxide. In the case of dyeing without peroxide, some deposition of lignin is visible on the wool fiber surface (identified by EDX spot analysis), but for wool fabric dyed with lignin with H₂O₂, the surface of wool fabric is quite clean, suggesting depolymerization of lignin by H₂O₂, which caused increased absorption of lignin sulfonate into the wool fiber and thereby increasing the colorfastness to washing of the treated wool fabric samples. Therefore, it is evident that the addition of H₂O₂ to lignin sulfonate dye-bath is beneficial as the peroxide treatment not only increased color yield but also enhanced the colorfastness to washing of the lignin sulfonate-treated fabrics. The peroxide treatment also did not cause any damage to the wool fiber and therefore is safe for the fiber.



Wool fabric treated with 10% owf lignin sulfonate without H₂O₂



Wool fabric treated with 10% owf lignin sulfonate in combination with H₂O₂

Figure 8. SEM optical micrographs of wool fabric treated with 10% (owf) lignin sulfonate without and with 1.67 mL/L H₂O₂.

2.8. Sustainability Aspects. Lignin is a waste material produced by the pulp and paper industry as a byproduct of the pulping process, which is very cheap compared to synthetic dyes. Its application as an alternative to synthetic dyes not only solves its disposal issue but repurposing it as a dye improves its sustainability and solves the end-of-life issues. On the other hand, synthetic dyes are highly expensive, they can have harmful effects on human skin, and effluent produced by them needs expensive treatments before discharging into the environment, and their degraded version sometimes could be more toxic than the parent dyes.⁵⁰ If the effluent is discharged into the environment, it can affect our aquatic and soil environments, ultimately affecting our food webs as they are designed to be recalcitrant.⁵¹ The toxic synthetic dyes present in textiles affect their biodegradability,⁵² which is unlikely for wool fabrics dyed with lignin sulfonate. Although in terms of energy and water usage, no significant savings can be made, the environmental savings achieved are quite

huge, which considerably improves the environmental sustainability of the treatment.

3. EXPERIMENTAL METHODS

3.1. Materials. A plain-woven wool fabric of 150.0 g/m² having 26 ends/cm and 25 picks/cm made from non-shrink-resist treated merino wool of an average diameter of 19 µm was used in this work, which was purchased from a local cloth merchant. Sodium lignosulfonate (average M_w = 18,000, C content = 33.7%, and S content = 8.8%), H₂O₂ (35%), and acetic acid were purchased from Sigma-Aldrich Chemicals (USA). Sandozin MRN and Sandoclean PC were procured from Arkema Chemicals (Switzerland). Figure 9 shows the typical chemical structure of Sodium lignosulfonate. The phosphate-free standard detergent used for the assessment of colorfastness to washing was supplied by the Society of Dyers and Colorists, UK.

3.2. Dyeing of Wool Fabrics with Sulfonated Lignin. The dirt, lubricating oil, and other contaminants present in the wool fabric were removed by scouring the fabric with 2 g/L Sandoclean PC and 0.2 g/L Sandozin MRN in a 9-L Vald Henriksen package dyeing machine at 50 °C for 20 min. After which, the fabric was washed with hot water and then with cold water until all the detergents were removed from the fabric and then dried.

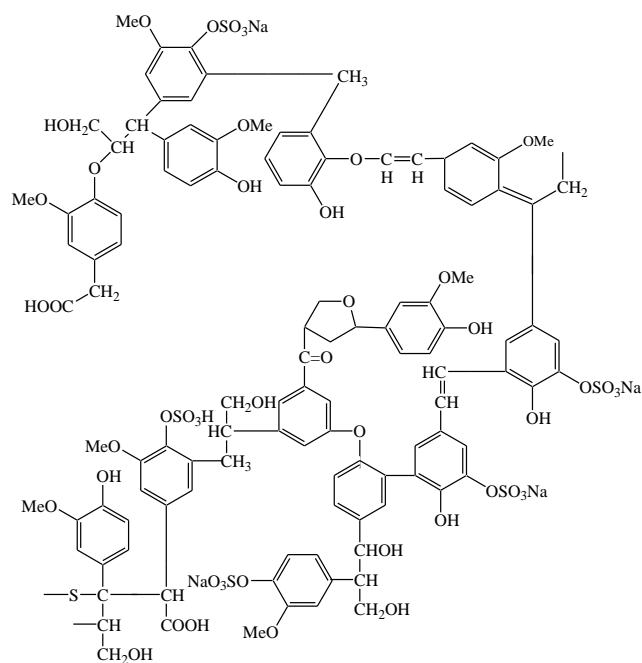


Figure 9. Chemical structure of sulfonated lignin.

The dyeing treatment of wool fabric was carried out in a Roaches Infrared sample dyeing machine (Model: Roaches Pyrotec, Roaches International, UK) using materials to liquor ratio of 1:20. The required quantity of pre-dissolved lignin was taken in a dyeing pot, and 0.2 g/l Sandozin MRN was added to it. After which, the fabric

samples were introduced, and dyeing continued for 10 minutes. Then the dyebath was set at pHs 3, 4, and 5 with sodium acetate/acetic acid and 7 and 9 with sodium carbonate. The temperature was raised to 98 °C at 2 °C/min and held for 60 min. For dyeing with lignin sulfonate in the presence of H₂O₂, 1.67 mL/L oxidant was added when the dyeing temperature reached 98 °C, and dyeing continued for another 60 min at that temperature. After the completion of dyeing, the bath was cooled to 45 °C, and the fabrics were washed with 0.5 g/l Sandoclean PC at 45 °C for 10 min. The fabric samples were then washed with cold water and then dried. The dyeing with 10% owf lignin sulfonate was carried out with and without H₂O₂ but without the addition of fabric to the dyeing pot. The UV-vis spectra of the lignin solution, non-oxidized and peroxide-oxidized, were recorded by using a Shimadzu UV-vis spectrophotometer (Model: UV2600i, Shimadzu Corporation, Japan) to study the effect of oxidation on the color intensity of lignin sulfonate.

3.3. Measurements of Color Yield, UV Radiation Absorption, and Colorfastness to Washing.

The color strength (K/S) values of fabrics dyed with various lignin at various pHs and concentrations were measured at the wavelength of maximum absorption using a Datacolor reflectance spectrophotometer (Model: DC 550, Datacolor International, Switzerland). All the measurements were carried out under illuminant D65, using a 10° standard observer with the UV component excluded and specular included. Each sample was folded four times, and one measurement was made at five different positions. The CIE L*, a*, b* values of dyed wool fabrics were measured under illuminant D65, using a 10° standard observer by an X-rite hand-held spectrophotometer (Model 45/0, X-Rite, Incorporated, USA). A Shimadzu UV-vis spectrophotometer (Model: UV2600i, Shimadzu Corporation, Japan) fitted with diffuse reflectance spectroscopy (DRS) measurement apparatus was used for the measurement of the UV radiation absorption capability of the lignin sulfonate dyed wool fabrics. The percent transmission of UV light through the control and treated wool fabrics was measured at wavelength intervals of 5 nm in the 290–380 nm spectral span. Sobolewski et al. reported that the UV intensity was the highest at 311 and 365 nm when the UV protection of different fabrics was measured using a standard photo-therapy wall with TL-01 UV phototherapeutic tube lamps as an irradiation source and a mannequin.⁵³ Therefore, we measured the UV transmittance (%) at 311 and 365 nm.

The colorfastness to washing of wool fabrics dyed with lignin was measured according to the ISO Test Method 105-C03 1987: Textiles – Tests for colorfastness – Part C03: Colorfastness to washing: Test 3 by washing them in a Gyrowash (Model 415/8, James Heal, Halifax, England) using the phosphate-free standard detergent. The color of the fabrics before and after dyeing was compared to the 3 M Grey Scale to determine the colorfastness grades.

3.4. Surface Characterizations. To identify the reasons for better colorfastness to washing exhibited by wool fabrics dyed with lignin in the presence of hydroperoxide compared to the fabrics dyed with lignin alone, the

surface of fabric samples was scanned on a Thermo Scientific scanning electron microscope (Model: Phenom Pure G6, Thermo Fisher Scientific, Inc., USA) at an accelerated voltage of 15 kV without any conductive coating. The wool fabrics dyed with various concentrations of lignin with and without hydrogen peroxide were characterized by a Fourier transform infrared (FTIR) spectroscope equipped with an attenuated total reflectance (ATR) attachment (Model: IRSpirit, Shimadzu Corporation, Japan) at a resolution of 4 cm⁻¹ in the range from 450 to 3600 cm⁻¹ by using a diamond crystal and 64 scans were signal-averaged.

4. CONCLUSIONS

This work demonstrates that sulfonated lignin can be used for the dyeing of wool fabrics as a sustainable alternative to synthetic acid dyes with reasonable colorfastness to washing. Lignin produced various depths of brown shades, and the dyed fabric showed reasonable colorfastness to light. The shade became darker as the color difference with undyed fabric increased with a decrease in pH and an increase in lignin sulfonate concentration. The addition of H₂O₂ to the lignin sulfonate dyebath was found beneficial for increasing the color intensity of the dyed wool fabrics and their colorfastness to washing. FTIR and SEM analyses suggest that peroxide oxidation caused the depolymerization of lignin but did not introduce new carboxyl functional groups, which eased the absorption of lignin sulfonate into the wool fiber. Lignin sulfonate can be used for the coloration of wool and other polyamide fibers as an alternative to synthetic acid dyes for producing brown color shades.

ASSOCIATED CONTENT

Supporting Information: Effect of lignin dosage on color strength, UV-vis spectra of lignin with and without H₂O₂, elemental distribution of C, N, O, and S, UV transmittance, ATR-FTIR spectra, and colorfastness to washing data. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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Notes

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Supporting Information Content:

Effect of lignin dosage on color strength of treated fabrics, UV-vis spectra of lignin sulfonate with and without H₂O₂, elemental distribution of C, N, O, and S on the treated fabrics' surface, UV transmittance (%) through the treated fabrics, ATR-FTIR spectra of various treated fabrics, and colorfastness to washing data of various treated fabrics.

SYNOPSIS TOC

Wool fabrics were dyed with lignin sulfonate with/without H₂O₂ and peroxide addition improved color yield and colorfastness to washing.

