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Research Article

Ficus sycomorus as a Sustainable Source of Leather Dyeing Material: An Ecofriendly Approach

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Natural colorants play a vital role in the production of eco-leather by substituting environmentally hazardous and toxic synthetic dyestuffs. In this study, a natural dye is extracted and optimized from the bark of *Ficus sycomorus*, a plant species locally known as shola, by designing different combinations of concentration, temperature, and time parameters. A dye extracted from 120 g/l concentration at a temperature of 1000°C for 60 minutes showed the highest absorbance value, and it has been used for further dyeing processes with and without mordant. Properties such as color strength (k/s), L*a*b* values, and color fastness of a dyed leather specimen were measured and analyzed. Oxalic acid mordant-dyed leather samples mordanted with the postmordanting method have revealed the highest k/s value among other experimental samples. Moreover, better color fastness properties were obtained from a leather sample mordanted with oxalic acid. Consequently, a dye extracted from the bark of *Ficus sycomorus* can be used as a leather dyeing material.

1. Introduction

Being a flexible, hygienic, and durable biomass material, leather is obtained from the by-products of animals through a series of processes, in which various chemicals are employed [1]. Leather processing involves pretanning, tanning, wet finishing, and finishing processes [2]. Imparting and improving the mechanical and sensory properties of tanned leathers are some of the central roles of post-tanning operations [3]. Leather dyeing is one of the crucial processes after tanning, where the color of the leather matrix is built [4]. Dyes can be either natural or synthetic based on their source. While synthetic dyes are derived from petroleum and coal-tar sources [5], natural colorants are obtained from natural sources like plants and invertebrates [6]. Besides, leather dyes are among the critical chemical pollutants of industrial origin. It is estimated that during the dyeing process, around 10–35% of the dye is lost in the effluent [7]. According to Saravanan and Chandramohan, currently around ten million tons of synthetic dyestuffs are consumed annually [8].

This is a serious cause of health and environmental hazards during its application by releasing significant amounts of waste and unfixed dyestuffs [9, 10]. On the other hand, due to the high level of biodegradability of natural dyes, wastewater can degrade organic substances without the need for physical and chemical processes. Hence, the demand for eco-friendly natural colorants has increased in the past few decades [11]. In an attempt to dye a leather matrix with sustainably sourced dying materials, a number of natural dyes have been reported. According to research [12], the leather matrix can be efficiently dyed using walnut bark, tea leaves, turmeric rhizomes, eucalyptus bark, and tea leaves. In another study, it was discovered that four distinct flower species, including *Tagetes erecta*, *Lantana camara*, Celosia cristata, and *Rosa damascena*, could color wet-blue goat leather [10]. Generally, beetroot [13], fungal pigment [14], *Mucuna pruriens* [15], and *Garcinia mangostana* Linn peels [16] were some of the sources from which natural colorants were sourced in the leather dyeing process so far. The fastness properties of leather effects in the dyeing

process can be enhanced by using mordants such as chitosan [17].

Ficus sycomorus is a fairly hardy, medium to a large spreading fig tree with a distinctive yellow stem that is often seen along riverbanks. Despite having its origins in Africa, it is now found in practically all tropical nations [1]. According to [18], the phytochemical analysis of *Ficus sycomorus* revealed that its bark extract contains phenols, tannins, flavonoids, coumarins, quninol, alkaloids, steroids, and saponins. Also, traditionally, its bark was used as a source of dyeing material for coloring fabrics in Ethiopian Orthodox Tewahedo Church monasteries. Hence, in this study, an attempt was made to optimize conditions for the extraction of dye from the bark of *Ficus sycomorus* plant species and the application of the dye extract onto leather samples. Afterward, the fastness properties of experimentally dyed leather samples were analyzed along with their color strength and color differences.

2. Materials and Methods

Sheep crust leather (0.3 kg based on shaved weight) was chosen as experimental leather and sourced from Bahir Dar tannery, Bahir Dar, Ethiopia. The barks of *Ficus sycomorus* extract were collected from Bahir Dar district of the Amhara regional state, Ethiopia. Moreover, a concentration of 1 M oxalic acid and aluminum sulfate ($KAl(SO)_2.12H_2O$) were used for the mordanting method.

2.1. Dye Extraction Process. The bark of *Ficus sycomorus* was collected and dried at room temperature. The dried bark extract was then grinded and sieved using a 0.9 mm sieve. The dye was extracted from the sieved powder by varying concentration, temperature, and time. A concentration of 100 g/l, 80 g/l, and 120 g/l at a temperature of 60°C, 80°C, and 100°C for about 45 minutes, 60 minutes, and 75 minutes was used to optimize the best condition for the extraction process.

2.2. Determination of Total Phenolic Content. The total phenolic content (TPC) of *F. sycomorus* extract was measured with the Folin–Ciocalteu reagent by the method employed by [19] using a UV-Visible spectrometer at 760 nm, and the result was expressed as mg of gallic acid equivalent per gram of sample (GAE/g) plus standard deviations using gallic acid as the reference.

2.3. Determination of Flavonoid Content. The total flavonoid content (TFC) of *F. sycomorus* extract was estimated according to the procedure outlined by [20] using a UV-Visible spectrometer at a wavelength of 425 nm. Quercetin was used as standard and the result was presented as mg quercetin equivalent per gram of sample (QE/g) plus standard deviations.

2.4. Characterization of Ficus sycomorus Colorant

2.4.1. UV-Visible Spectroscopy and Fourier-Transform Infrared Spectroscopy (FTIR). A UV-Vis spectrophotometer was used to examine the optimum absorption of the dye extract. On the other hand, the functional groups present in *F. sycomorus* colorant were investigated using Fourier-transform infrared spectroscopy (FTIR) with a resolution of 4 cm^{-1} over the range of 4000–500 cm^{-1}.

2.5. Leather Dyeing Process with and without Mordant. Ten pieces of sheep crust leather were taken from Bahir Dar Tannery Ltd. for dyeing with and without mordant. The dyeing condition was optimized by varying the dyeing temperature and time to 40°C, 50°C, 60°C, and 45 minutes, 55 minutes, and 65 minutes, respectively. Oxalic acid (2.5%) and aluminum sulphate ($KAl(SO)_2.12H_2O$) (2.5%) based on the shaved weight were applied both in premordanting and postmordanting methods onto *Ficus sycomorus* extract dyed leather specimen.

2.6. Color Fastness Properties. Dyed leather samples were cut from experimentally dyed leather samples after conditioning them at a temperature of 20 ± 2°C and $65 \pm 2\%$ relative humidity for about 48 hours.

2.7. Dry and Wet Rub Color Fastness. Dyed leather samples were cut and tested for dry and wet rub fastness to assess their resistance to color fading using a grey scale according to EN ISO 105-A02 by following the standard procedure. The rating scales were from 1 to 5, with 1 being the worst rating and 5 being the best rating.

2.8. Color Fastness to Light. The resistance to change in color of dyed leather samples to the action of a standard artificial light source (a Xenon Lamp) was also assessed using EN ISO 105-B02 under controlled conditions by following the standard guidelines. The fastness of dyed sample leather was then assessed by comparing the color fading of dyed samples with standards of blue scale from 1 to 8. Blue scale 1 indicates that the light fastness property of the dyed leather sample is low, and 8 implied that the light fastness property of the dyed leather sample is best.

2.9. Color Fastness to Washing. The resistance to change in color of a dyed leather specimen and the staining of a dye substrate onto cotton and wool were assessed by following IUF 453. Dyed leather samples in contact with cotton and wool were agitated in a standard wash detergent solution for 30 minutes at 400 C. Then, the change in color of a leather sample was compared with the original leather specimen. Similarly, the staining on cotton and wool was assessed with a standard grey scale.

2.10. Color Fastness to Perspiration. The resistance of a dyed leather sample to the prolonged action of an artificial perspiration solution was assessed using the IUF 426 standard procedure. The change in color of a dyed leather sample was then compared with the original leather sample. The staining of cotton and wool textiles was also assessed by using standard grey scales.

2.11. K/S Values and Color Difference. To measure the color depth of a sample at the surface, the surface reflectance of dyed leather samples was measured using a Macbeth color eye 3100 reflectance spectrophotometer using the Kubelka–Munk equation.

$$\frac{K}{S} = \frac{(1 - R\lambda \max)}{2R\lambda \max}, \quad (1)$$

where K is the light absorption coefficient, S is the light scattering coefficient, and Rλmax is the light reflectance value of a dyed surface of leather samples at a wavelength of maximum absorption. Moreover, the color difference of dyed leather samples was measured to evaluate their lightness-darkness (L^*), greenness-redness (a^*), and blueness-yellowness (b^*).

2.12. Condition Optimization for Extraction and Dyeing Process. Temperature, time, and concentration of the dye extract were variables considered for extracting dye from *Ficus sycomorus* plant species. The experimental designs were done by using the Taguchi orthogonal array method with three-level experimental designs by Minitab 17 software [21]. Nine experimental conditions were organized by the software for optimizing the best conditions for extraction. Finally, the best combinations of variable conditions that yield the highest concentration of dye (highest absorption indicates the highest dye concentration) were used to color a leather sample for further process.

The duration of dyeing a leather sample and temperature were optimized to obtain the best condition of variable combination for the dyeing process. A temperature of 40°C, 50°C, and 60°C, for a dyeing duration of 45 minutes, 55 minutes, and 65 minutes were used to optimize the best match of conditions as presented in Table 1 above. The effects of dyed leather samples were also analyzed by dyeing the sample with and without the application of mordant. Oxalic acid and aluminum sulfate ($KAl(SO)_2.12H_2O$), both 2.5% based on the shaved weight of a leather sample were used for premordant and postmordant dyeing at a temperature of 60°C and a dyeing duration of 1 hour. A comparison for K/S values, color difference, and fastness properties were then made between leather samples dyed with and without the application of mordant.

3. Results and Discussion

3.1. Total Phenolic and Total Flavonoid Content of F. sycomorus Dye Extract. The results of the quantitative determination of the total phenolic content of *Ficus sycomorus* stem bark dye extract revealed 171.849 ± 0.251 mg GAE/g polyphenols, as shown in Table 2 underneath. On the other hand, the total flavonoid content of *F. sycomorus* stem bark dye extract was found to be (19.727 ± 0.638 mg QE/g) which is in agreement with the study conducted by [19]. Here, according to the TPC and TFC results of *F. sycomorus*, it can be noted that polyphenols are the major constituents of the dye extract.

3.1.1. UV-Visible Spectrometer and Fourier Transform Spectroscopy. The absorption spectra of aqueous *F. sycomorus* bark extract revealed a maximum absorption wavelength at 492 nm (Figure 1(a)). On the other hand, the FTIR spectrum of the dye showed influential absorption peaks at 3383.6370, 2942.7473, 1706.9902, and 1632.8439 cm^{-1} neglecting the fingerprint region (Figure 1(b)). Due to the presence of chemicals like flavonoids and other phytochemicals within the crude extract, the bands signify stretched vibration bands [22]. The broad, medium absorbance at 3383.6370 is similar to an O-H stretching vibration. Weak C-H stretching of an aldehyde or an alkane's hydroxyl group was noted at 2942.7473, and FTIR spectrogram observations made around 1632.8439 indicates C=C stretching of an alkene, or C=O stretch of the amides identified in alkaloids lying within the given range [23]. The peak at 1706.9902 cm^{-1} corresponds to the color intensity present in the extract [24].

3.2. Conditions Optimized for Extraction. The process of dye extraction from *Ficus sycomorus* was carried out with an experimental design for parameters illustrated in Table 3 to identify the best match of conditions from those parameters, the maximum absorbance of a dye extracted from *Ficus sycomorus* was measured using an instrument called a UV-Visible spectrophotometer. The maximum absorbances measured were diluted with a ratio of 1 : 5 [21], and the outcome is presented in Table 3 underneath. A dye that has a maximum value of absorbency has a maximum dye concentration, as stated by Abera and Negassi [21].

A dyed leather sample with a concentration of 120 g/l at a temperature of 100°C for 60 minutes duration has revealed the highest absorbance among other experimental test conditions performed with a different arrangement of parameters. Consequently, further dyeing processes were undertaken with a dye extracted with this set/arrangement of variables.

4. Conditions Optimized for the Dyeing Process

4.1. Mordant-Free Dyeing. Mordant-free dyeing of a leather sample was undertaken with a dye extract optimized based on conditions of parameters designed and presented in Table 4, and both *K*/*S* and CIE Lab values of *Ficus sycomorus* extract dyed leather samples were measured with color eye 3100 spectrophotometer by following a standard procedure. The results obtained from *K*/*S* of a dyed leather sample were used to judge the best-optimized matches of parameters for dyeing with mordants. Accordingly, test number eight dyed at a temperature of 60°C for 55 minutes duration was found to give the highest *K*/*S* value and was considered to be the best condition optimized for dyeing with mordant using oxalic acid and aluminum sulfate.

4.2. Dyeing with Mordant. Oxalic acid and aluminum sulfate (both 2.5% based on sample shaved weight) were used for mordant dyeing with a condition of variables optimized in Table 5, dyed samples were then measured for color strength to identify a mordant that yield the highest *K*/*S* value. The

Table 1: Condition optimization for extraction and dyeing process.

Test#	Dye concentration (g/L)	Extraction process		Dyeing process	
		Temperature (°C)	Time (minute)	Temperature (°C)	Time (minute)
1	80	60	45	40	45
2	80	80	60	40	55
3	80	100	70	40	65
4	100	60	60	50	45
5	100	80	70	50	55
6	100	100	45	50	65
7	120	60	70	60	45
8	120	80	45	60	55
9	120	100	60	60	65

Table 2: Extraction yield, total phenolic, and flavonoid contents of *F. sycomorus* bark.

No.	Description of contents	Approximated value
1	Dye extraction yield (%)	21.539
2	Total phenolic content (mg GAE/g)	171.849 ± 0.251
3	Total flavonoid content (mg QE/g)	19.727 ± 0.638

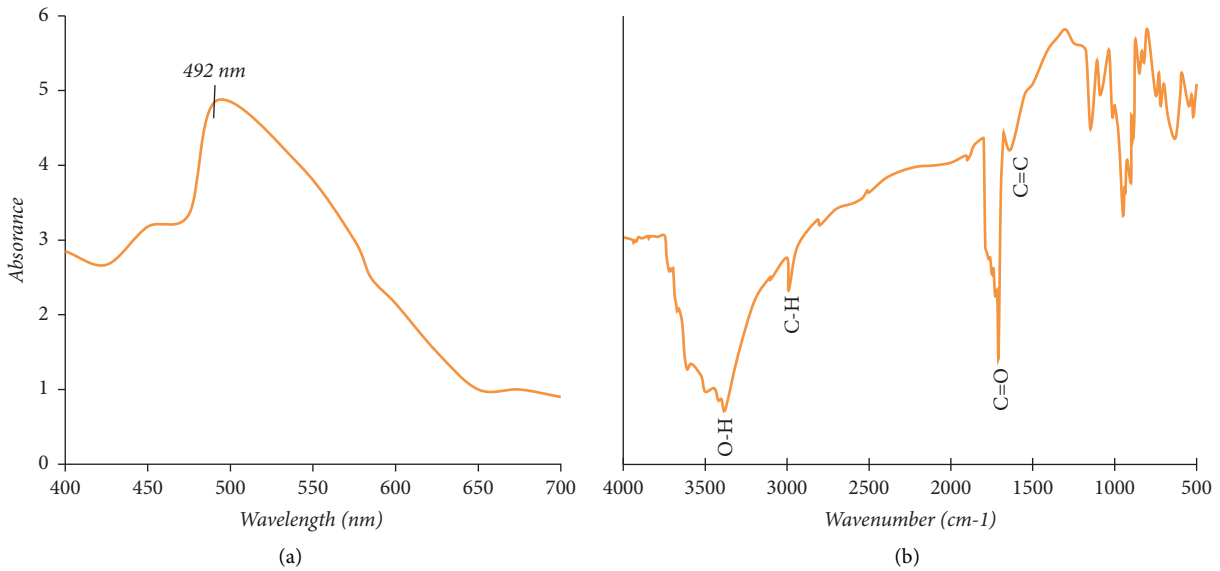

Figure 1: Qualitative analysis of the *F. sycomorus* dye extract: (a) UV-Visible spectroscopy and (b) FTIR spectra of *Ficus sycomorus* colorant.

Table 3: Optimized conditions for the extraction method.

Test#	Parameters/variables			Absorbance at $\lambda = 492$ nm
	Dye concentration (g/L)	Temperature (°C)	Time (minute)	
1	80	60	45	1.971
2	80	80	60	3.081
3	80	100	70	2.539
4	100	60	60	3.271
5	100	80	70	2.947
6	100	100	45	4.224
7	120	60	70	2.415
8	120	80	45	4.039
9	120	100	60	4.983

Table 4: Conditions optimized for mordant-free dyeing method.

Test#	Parameters/variables		K/S values at $\lambda = 492$ nm
	Temperature (°C)	Time (minute)	
1	40	45	2.229
2	40	55	2.873
3	40	65	3.492
4	50	45	3.297
5	50	55	3.902
6	50	65	4.117
7	60	45	4.317
8	60	55	4.585
9	60	65	4.009

Table 5: Conditions optimized for mordant dyeing method.

Trial#	Mordant type and technique	Mordanting parameters		K/S $\lambda = 492$ nm	CIE values		
		Temperature	Time		L^*	a^*	b^*
1	Without	60	55	4.585	45.3	12.3	15.5
2	Oxalic acid (pre)	60	55	6.626	36.9	14.3	16.2
3	Oxalic acid (post)	60	55	7.982	30.5	20.6	18.5
4	Alum (pre)	60	55	5.403	40.2	17.5	11.7
5	Alum (post)	60	55	5.927	38.6	14	9.4

color differences of those mordant dyed samples were also measured to assess the lightness-darkness, redness-greenness, and blueness-yellowness of the samples. The K/S values of dyed leather samples become optimum at a wavelength of around 490 nm, as stated in Figure 2 below.

As stated by Negassi and Abera, dyeability is directly proportional to the color strength of a dyed leather sample [21]. In this study, an oxalic acid mordant dyed leather sample has revealed a K/S value of 6.626 and 7.982 in premordanting and postmordanting techniques signifying its increased dyeability. This was a bit higher when compared with a K/S value of a leather sample dyed with alum mordant. Similarly, the K/S value of alum mordant dyed leather samples were 5.403 and 5.927 in premordanting and postmordanting techniques which were greater than the controlled (a sample dyed without mordant) leather sample having a K/S value of 4.585. All the dyed leather samples have been presented in Figure 3 above.

On the other hand, color difference values of leather samples dyed without, oxalic acid and aluminum sulfate mordant were also measured to assess values of L^*, a^*, and b^*. The lightness value of a leather sample dyed without mordant, and alum mordant dyed leather sample with the premordanting technique were relatively higher than the oxalic acid mordant dyed leather sample as illustrated in Table 5 characterizing a lighter shade. The value of a^* for a sample dyed with oxalic acid and alum mordant in the postmordanting and premordanting techniques, respectively, were found to give 20.6 and 17.5, signifying more pinker in color than the leather sample dyed without mordant.

Similarly, 16.2 and 18.5 were b^* results for oxalic acid mordant dyed leather samples in premordanting and postmordanting methods respectively indicating that the samples were more yellow in color than other experimental samples. To sum up, controlled and aluminum mordant dyed leather samples were found to give a lighter hue, and an oxalic acid mordant dyed leather sample was found to give redder and yellower color.

5. Color Fastness Properties

The FTIR analysis of the *F. sycomorus* colorant in Figure 1 revealed that the spectrum of the dye extract showed a peak at 1706.9902 cm^{-1} indicates the presence of a carbonyl group, and peaks at 1203.7739 and 1219.4626 cm^{-1} denote the presence of a C-O stretch which signify the formation of covalent bond between polyphenols of the dye extract and the leather matrix. The development of covalent bond between the colorant and that of the leather substrate indicates strong fixation of dye with fiber which leads to better fastness properties. Moreover, due to the presence of a O-H functional group in the FTIR spectrum of the dye extract at around 3383.6370 cm^{-1}, hydrogen bonds would be formed between the colorant and the collagen matrix.

5.1. Fastness to Dry and Wet Rubbing. The fastness values of a leather sample dyed with optimized dyeing conditions were assessed for dry and wet rubbing. The results for dry and wet rub fastness values are presented in Table 6 underneath. As it can be inferred from the table, the dry rub fastness value of the postoxalic acid mordant dyed leather sample was found to be grade 5 being a good indicator of its resistance to dry rubbing.

On the other hand, a color grade of 4/5 was found for the wet rub fastness value of the postoxalic acid mordant dyed leather sample. This implied that oxalic acid mordant dyed leather samples have resulted in a good resistance to rubbing, as indicated in Table 6 above. As far as staining values

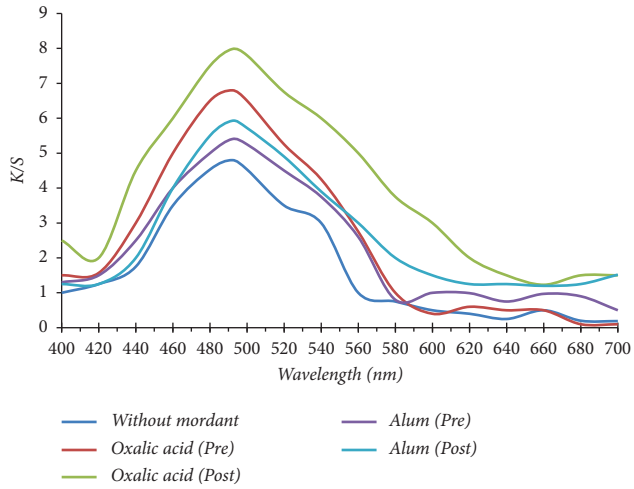

Figure 2: *K*/*S* vs. wavelength curves for dyed samples.

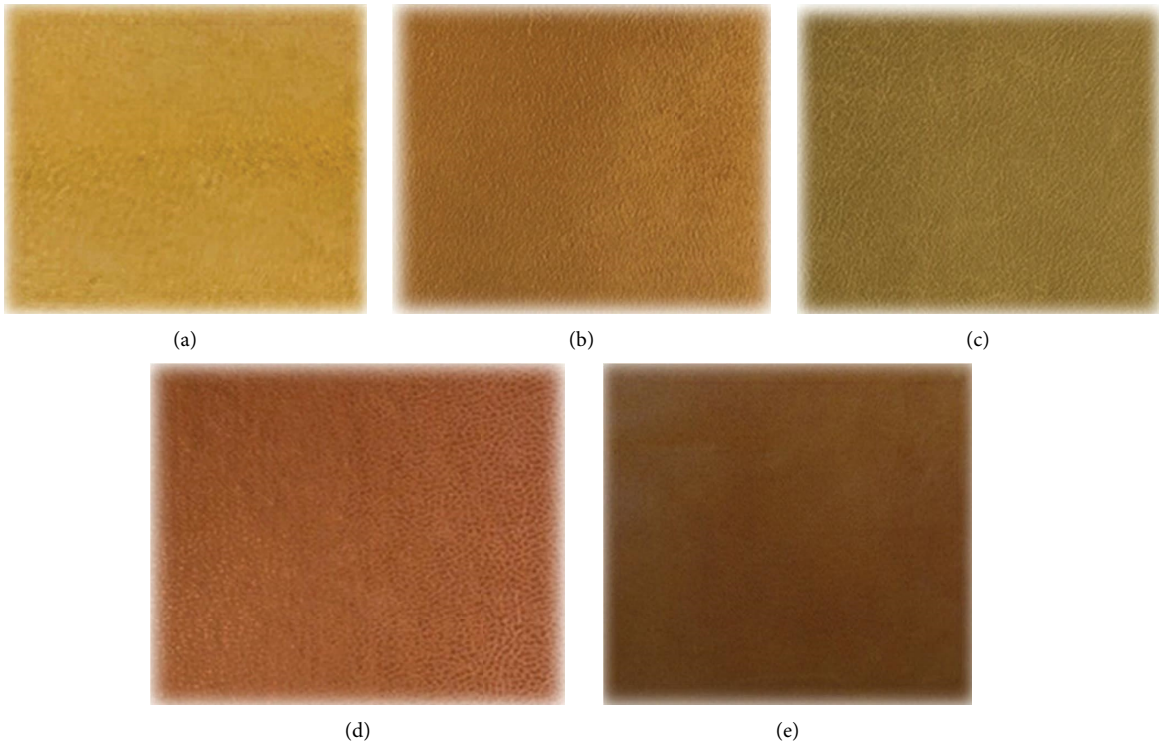

Figure 3: Images of dyed leather samples: (a) dyed sample without mordant, (b) dyed sample with alum premordanting method, (c) dyed sample with alum postmordanting method, (d) dyed sample with oxalic acid premordanting technique, and (e) dyed sample with oxalic acid postmordanting technique.

are concerned, oxalic acid mordant dyed leather samples mordanted with the postmordanting method were comparatively better than that of controlled and alum mordant dyed leather samples.

6. Color Fastness to Washing

The colorfastness to washing of a leather sample dyed with *Ficus sycomorus* extract was assessed by following the EN ISO 15702 standard procedure. The change in color of a dyed leather sample and the staining onto wool and cotton textile materials were compared with the initial sample of these textile materials with the help of grey scale reading, and the value of the output is depicted in Table 7. The staining of the controlled dyed sample has resulted in satisfactory values of 3/4 and 3, respectively.

On the other hand, oxalic acid mordant dyed leather sample in postmordanting technique were found to have 4/5 and 5 staining values by cotton and wool, respectively. This value proved that a sample dyed with postmordanting technique of oxalic acid has very good to excellent resistance to washing.

Table 6: Values for dry and wet fastness properties.

Trial#	Mordanting technique	Fastness values			
		Fastness to dry rub		Fastness to wet rub	
		Change in color	Staining	Change in color	Staining
1	Pre (oxalic acid)	4/5	4	3	2/3
2	Post (oxalic acid)	5	4/5	4/5	4
3	Pre (alum)	4	3/4	3/4	2/3
4	Post (alum)	4/5	4/5	4	3/4
5	Controlled	4	4	3	2/3

Table 7: Values for washing fastness properties.

Test#	Mordant and mordanting technique	Color fastness to washing		
		Staining		Color change
		Wool	Cotton	
1	Controlled sample	3	3/4	3/4
2	Oxalic acid (pre)	4	4	4
3	Oxalic acid (post)	4/5	5	4/5
4	Alum (pre)	3/4	4	3/4
5	Alum (post)	4	4/5	4

Table 8: Values for light and perspiration fastness properties.

Test#	Mordant and mordanting method	Color fastness to light		Color fastness to perspiration		
		24 hrs.	48 hrs.	Staining		Color change
				Cotton	Wool	
1	Controlled	5	4	3	3/4	3/4
2	Alum (pre)	5/5	4/5	3	2/3	3
3	Alum (post)	7/5	7	4	3	3/4
4	Oxalic acid (pre)	6	6	3/4	3	4
5	Oxalic acid (post)	7/5	7	5	4/5	4/5

6.1. Color Fastness to Light and Perspiration. A dyed leather sample was exposed to a light source for about 24 and 48 hours to assess its light fastness properties and the result is recorded in Table 8 underneath. As it can be inferred from Table 8, a sample mordanted with oxalic acid and alum in a postmordanting technique have resulted a higher color fastness to light of 7/5 and 7 when exposed to a light source for 24 and 48 hours, respectively. This indicates that a dyed sample mordanted with oxalic acid and alum has higher resistance to color fading. Similarly, a dyed sample mordanted with oxalic acid with premordanting technique has showed satisfactory results of 6 when exposed to 24 and 48 hours light source exposure. On the other hand, inferior resistance to color fading by light source were recorded from a controlled and prealum mordant dyed leather samples with 48 hours light source exposure.

The perspiration fastness of a dyed leather sample was assessed by following the IUF 426 method in such a way that the staining on cotton and wool were compared with the original dyed samples using grey scale measurement. The value obtained from the staining of cotton and wool for color fastness to perspiration was presented in Table 8 below. The staining of a postoxalic acid mordanted leather sample has revealed very good to excellent rating of 4/5 and 5 on wool and cotton, respectively.

This implied that the resistance to staining of a dyed leather sample mordanted with oxalic acid using the post-mordanting method was higher than the controlled and other mordant dyed leather samples. Preoxalic acid and postalum mordant dyed leather samples have revealed a staining value of 3-4 and 4 with wool and cotton, respectively. On the contrary, prealum mordant dyed leather samples showed inferior color fastness properties to perspiration.

7. Conclusion

Natural dyestuffs can be extracted from different parts of plants among other sources revealing eco-friendly colorants. This study revealed that a dyeing material obtained from *Ficus sycomorus* bark extract with a 120 g/l concentration at a temperature of 100°C for 60 minutes can be used as a dye for dyeing a leather matrix. Oxalic acid mordant dyed leather sample in the postmordanting method has resulted in better color strength indicating its increased dyeability. Best ratings were also recorded for the fastness properties of a dyed leather sample mordanted with oxalic acid making it suited for a feasible commercial application. The accessibility of the plant is abundant in the Horn of Africa, making the extraction process to be economical.

Data Availability

The data collected and analyzed during this study are included in the paper and more can be accessed from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Authors' Contributions

All authors contributed to the study conception and design.

Acknowledgments

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