

Assessment of Dyeing Properties of Hibiscus sabdariffa on Ostrich Leather

Mehmet Mete M[u](#page-0-0)tlu^l [®], Deniz Kılıçaslan² ®, Nilay Ork Efendioglu³

1,2,3Department of Leather Engineering, Faculty of Engineering, Ege University, İzmir, Türkiye

Abstract − Natural dyestuffs derived from various plants can be used for dyeing processes in the textile and leather industries. *Hibiscus sabdariffa*, one of these natural dyestuffs, is known to be used in textiles and produces a red color with good dyeing properties. This study aimed to investigate the dyeing effect of *Hibiscus sabdariffa* flower on leather materials and to determine its dyeing properties. To achieve this, a dye was extracted from *Hibiscus sabdariffa*, and all wet processes of ostrich skins—an exotic type of leather—were carried out, incorporating the dye extract by adding a mordant during the dyeing process. Both chrome and vegetable tannins were utilized to examine the effects of the tanning process. Extraction yield, pH measurement, color measurement, to-and-fro rubbing fastness tests, and dyestuff exhaustion measurements were conducted. The results indicated that, particularly when the post-mordant process was applied, the extracted dyestuff yielded superior dyeing properties and color results compared to the non-mordant process. The extract from *Hibiscus sabdariffa* was determined to effectively dye the skins, producing beautiful and satisfying colors, especially when using iron sulfate mordant in vegetable-tanned leathers.

Keywords *− Hibiscus sabdariffa, dyeing, leather, natural dyestuff, ostrich skin*

1. Introduction

With its distinctive and special skin appearance, Ostrich leather is considered one of the most exotic leathers available. The unique skin texture is characterized by the rounded formations of hair follicles on the skin surface. This type of leather is widely utilized in the leather industry's production of bags, wallets, shoes, and clothing [1]. Due to its softness, flexibility, and durability, ostrich leather is regarded as a luxury material. Further, interest in this product, which can be easily shaped, has grown and rapidly expanded its market [2].

Ostrich skins possess distinct differences from other leathers in shape and grain pattern, which are crucial for marketing purposes and necessitate specialized removal, conservation, and processing technologies [3]. Dyeing is paramount among the various processes that impact leather's visual appeal. A wide range of dyestuffs is employed in the fashion industry to accommodate the ever-evolving fashion preferences and technological advancements, reflecting aesthetic values, luxury, and customer demands. Consequently, the color of finished leather plays a significant role in decision-making and customer satisfaction. In contemporary times, growing environmental awareness and legal regulations have heightened the demand for natural products within the leather industry, mirroring trends in other sectors. Research into natural dyes as alternatives to the chemical products that were prevalent in the past has gained importance. Natural dyestuffs do not contribute to environmental pollution and allow for good fastness properties and various color combinations when combined with mordants [4].

¹mete.mutlu@ege.edu.tr; ²denizkilicaslan29@gmail.com; ³nilay.ork@ege.edu.tr (Corresponding Author)

As implied by their name, natural dyes are derived from renewable resources such as plants, animals, and minerals [5]. It is well established that vegetable dyes can impart color to materials derived from nature through specific processes. Some plants have their entire parts utilized for dyeing, while others employ particular organs (e.g., flowers, leaves, seeds, bark, and roots). For instance, flowers are harvested at their peak maturity, seeds post-ripening, and leaves during the plant's blooming phase. The shells are utilized after the plant has shed its leaves. Vegetable dyes are integral to natural dyeing due to their abundance and diverse color palette. The dyeing process using natural vegetable dyes involves several steps, including harvesting, drying, and preparation, leading to a ready-to-use dyestuff [6].

One notable natural dye is the extract from *Hibiscus sabdariffa*, which has numerous industrial and medicinal applications [7]. It is widely used in various fields, particularly textile dyeing [8-10]. *Hibiscus sabdariffa* (common name: Roselle) belongs to the Malvaceae family and contains anthocyanin, a water-soluble pigment whose color is influenced by pH levels. After undergoing specific natural processes, the pigment from *Hibiscus sabdariffa* (Figure 1) can be utilized as an alternative to synthetic dyes, potentially mitigating environmental damage. The color is generated by grinding and soaking the plant in water, where pH variations impact color intensity [11-12].

Figure 1. Main colorant in *Hibiscus sabdariffa* flower—cyanidin-3-sophoroside [13]

Several methods exist to extract dye from *Hibiscus sabdariffa*, particularly from the calyces, which can be ground or fully soaked in water. Due to their water-soluble pigment nature, they can yield dye without additives, although color intensity may vary [12]. The dyeing material and the plant parts used for dye extraction can be combined directly or through the addition of a mordant, a process referred to as "mordanting." Mordants can be either chemical or natural [14]. Typically, mordants consist of metallic salts (e.g., aluminum, chromium, iron, copper, and tin) and tanning agents [15]. These substances are essential for fixing dyes to fibers, improving dye uptake, and enhancing color and light fastness [15]. Various dyeing techniques also allow for a broader spectrum of colors [16]. The primary purpose of using a mordant is to ensure dye fixation and to achieve diverse color tones [17-18].

To the best of the authors' knowledge, no studies in literature address the dyeing of ostrich skins with natural dyes derived from the *Hibiscus sabdariffa* flower. This study aims to investigate the dyeing of ostrich skins using the natural dye extracted from dried *Hibiscus sabdariffa* flowers, examining its dyeing properties and considerations during the process. For this purpose, two ostrich skins were tanned using chrome and vegetable tannins and then divided into four parts, resulting in eight samples. The leathers tanned with chrome and vegetable tannins were split into four pieces, with one piece of each remaining undyed as a control. This approach allowed for the evaluation of the dyeing processes using the extract from dried *Hibiscus sabdariffa* flowers, with and without the assistance of mordants (copper sulfate and iron sulfate). The dyeing efficacy of *Hibiscus sabdariffa* and the fastness properties of the ostrich leathers were investigated, yielding satisfactory dyeing results.

2. Experimental

2.1. Materials

This study obtained two raw ostrich skins from Hasmera Ostrich Farm, Çanakkale (Figure 2). Dried flowers of *Hibiscus sabdariffa* (Figure 3), a natural dyestuff sourced from Koç Kardeşler Baharat (Bornova, İzmir), were used for dyeing the ostrich skins. Copper sulfate $[CuSO_4·5H_2O]$ and iron sulfate $[FeSO_4·7H_2O]$, obtained in technical grade from BOR-KİM Chemicals, were utilized as mordants. The configuration of the ostrich skins prior to processing is detailed in Table 1. A calibrated Metrohm 848 Titrino plus potentiometer was used to adjust the pH levels. The CIE L^{*}, a^{*}, b^{*} color measurements were performed using a Konica Minolta CM-3600d spectrophotometer, ensuring precise and reproducible results. The fastness tests were conducted using a Bally Finish Tester 9029.

Figure 2. Raw ostrich skins

Figure 3. Dried *Hibiscus sabdariffa* flower

Table 1. The layout of the ostrich skins

2.2. Materials

2.2.1. Extraction Process, Extraction Yield, and pH Measurement

Dried *Hibiscus sabdariffa* flowers (40 g) were weighed and steeped in 1 liter of water at room temperature for 24 hours to prepare an infusion (Figure 4). The infused solution was then subjected to extraction at 75°C for 3 hours, and the resulting extracts were combined to form a stock solution (Figure 5). This stock solution was concentrated to a constant weight by transferring it into a glass dish and drying it in a hot-air oven at $100 \pm$ 2°C until complete evaporation of water. The total solids were determined by weighing the residue left in the dish. The extraction yield was calculated using the following formula:

Evaporation Residue (mg/L) = $(A-B)$ x 1000/V

A= Total weight of the evaporation dish and solid material (mg)

B= Weight of the empty evaporation dish (mg)

V= Volume of sample (mL)

Figure 4. The state of *Hibiscus sabdariffa* flowers after infusing in hot water

Figure 5. Extracted *Hibiscus sabdariffa* flower

The pH measurement of the dye extract was conducted using a Metrohm 848 Titrino plus potentiometer. After adjusting the extract temperature to $20 \pm 1^{\circ}\text{C}$, pH values were measured with a precision of 0.05 pH units. The pH readings were taken at 30 and 60 seconds after immersing the electrodes in the extract.

2.2.2. Leather Processing

The weights of the two salted dry ostrich skins were recorded before processing. The main recipe applied to the raw ostrich skins is presented in Table 2. After the pickle process, two different tanning methods—chrome and vegetable tanning—were employed, following the recipes outlined in Tables 3 and 4.

In studies on mordant use, both pre-mordanting and post-mordanting have been highlighted in the literature [19-22]. While pre-mordanting is commonly applied in textile dyeing processes, some research also indicates its importance in leather processing [23-24]. However, other studies suggest that post-mordanting is more suitable for leather materials [25]. In pre-mordanting, the textile material is treated with the mordant prior to dyeing, facilitating better dye binding. Nevertheless, in some cases, pre-mordanting may cause the mordant to adhere primarily to the surface of the leather, thereby hindering sufficient dye absorption.

Post-mordanting tends to yield better results because the mordant helps bind loosely bound dyes that have penetrated the leather during the dyeing process. After applying the mordant, these dyes more effectively bond to the leather, resulting in a more uniform and stable coloration. The mordant acts as a chemical agent that strengthens the bond between the dye and the leather fibers. Initially, the dyes may form a temporary bond with the surface, but the mordant application allows these bonds to become more permanent. This interaction enables the dye to penetrate deeper into the material, ensuring more consistent and intense coloration.

Based on these considerations, this study employed post-mordanting. In this method, the mordanting process is performed after the material has been dyed. Post-mordanting typically involves treating the dyed material with a solution of the selected mordant. The duration of treatment and the concentration of the mordant solution can significantly influence the final color and properties of the material. The final phase of the process focuses on developing the desired color shade. The effectiveness of post-mordanting depends on various factors, such as the type of material, the dye used, and the desired color outcome [26-29].

The dyestuff was first applied to the dye bath (drum), which penetrated and partially bonded with the leather. Afterward, the mordant was introduced to ensure the residual dyestuff fully bonded to the leather, facilitating a more stable and uniform coloration. This sequence ensures that the mordant works effectively in conjunction with the dye, enhancing the overall depth and permanence of the color.

Table 2. Main recipe up to pickle process for ostrich leathers

2.2.3. Determination of Color Measurement

A Konica Minolta CM-3600d Spectrophotometer was used to numerically determine the color differences of leathers tanned with different tanning agents and dyed using various mordants. The color measurements were performed according to the CIE L*, a^* , b^* color system. In this system, the L* value represents the lightness of the color on a scale from 0 to 100 (with 100 indicating white and 0 indicating black). The a* value represents the color's position on the red-green axis, where negative values indicate green and positive values indicate red. Similarly, the b* value represents the blue-yellow axis, where negative values indicate blue and positive values indicate yellow. Color measurements were taken from five distinct points on the leather surface using the device's standard reading area, and the average of these measurements was calculated. The color difference $(ΔE)$ was determined using the following formula:

$$
\Delta E = \sqrt{(L_{sample} - L_{target})^2 + (a_{sample} - a_{target})^2 + (b_{sample} - b_{target})^2}
$$
 (2.1)

The color strength values (K/S) were calculated (at λ max = 400 nm) according to the Kubelka–Munk formula (2.2) , which is written as. K is the scattering coefficient, S is the absorption coefficient, and R is the reflectance. R is the decimal fraction of the reflectance of dyed leather, $R = 1.0$ at 100% reflectance.

$$
K/S = (1 - R)^2 / 2R
$$
 (2.2)

2.2.4. Determination of Color Fastness to Cycles of-to-and-fro Rubbing

To assess the color fastness of the leathers, a rubbing test was performed in accordance with ISO 11640, using both dry and wet felts. Samples were cut from the leather using a 15 x 8 cm, with the assistance of a pressing machine. The samples were then subjected to 50 to-and-fro rubbing cycles on a Bally Finish Tester 9029 for the dry rubbing test. For the wet rubbing test, felts soaked in pure water were wrung to remove excess water, and the leathers were rubbed 25 times back and forth. The color transfer and intensity of coloring were evaluated after the tests.

2.2.5. Calculation of Dyestuff Exhaustion

To determine the dyestuff exhaustion from the *Hibiscus sabdariffa* extract used for dyeing, bath samples were collected and analyzed with a UV-VIS spectrophotometer at the end of the dyeing process. Initially, the dyestuff was scanned within the 350-750 nm wavelength range to determine the wavelength with maximum absorbance.

Next, solutions of known concentrations were prepared by diluting the 40 g/2410 mL stock solution to 2, 4, 6.25, 10, and 20 times its concentration. Calibration curves were drawn based on the absorbance values at the determined wavelength. After the dyeing process, samples from the dye baths were analyzed using the UV-VIS spectrophotometer to calculate the dyestuff exhaustion.

3. Results and Discussion

3.1. Extraction Yield and pH Measurement Findings

A total of 2410 mL of *Hibiscus sabdariffa* extract was obtained through the extraction process, and the extraction yield was calculated as 10.6% based on solid matter determination. The pH of the extract was measured at 2.8, indicating an acidic nature suitable for dyeing applications.

3.2. Color Measurement Findings

Images of the finished and dyed ostrich leathers processed according to the designed recipes are shown in Figure 6. The images show that the *Hibiscus sabdariffa* extract produced greenish-blue tones on chrome-tanned leathers and reddish-brown tones on vegetable-tanned leathers. This variation is primarily attributed to the intrinsic color characteristics of the tanning agents. Additionally, ostrich leathers dyed with iron sulfate mordant exhibited darker shades.

Figure 6. Dyed ostrich leathers (Top row: Chrome-tanned; bottom row: vegetable-tanned, in order from left to right according to the codes)

The color measurement results for the leathers are provided in Table 5, and the color difference (∆E) values are presented in Table 6. According to the CIE Lab color system, it was observed that leathers dyed using iron sulfate mordant had lower L* values, indicating darker colors. This finding is consistent with previous studies in leather and textile dyeing research [30-32]. Moreover, the a* values for chrome-tanned leathers were negative, indicating a shift towards green, while for vegetable-tanned leathers, the a* values were positive, indicating a shift towards red. This observation further supports the color trends mentioned earlier.

The K/S values, representing color strength, highlight the impact of mordant treatments on dye uptake. Nondyed samples (C0 and V0) exhibit the lowest K/S values (2.241 and 1.717), reflecting minimal color strength. Mordant-treated samples show significantly higher values, with iron sulfate (C2 and V2) producing the highest K/S (4.126 and 3.983), indicating its superior ability to enhance dye absorption by forming stronger dye-fiber complexes. Copper sulfate (C1 and V1) also improves color strength (2.641 and 3.373), though less effectively than iron sulfate. Non-mordant dyed samples (C3 and V3) show moderate K/S values (2.616 and 2.209), suggesting limited dye uptake without mordants. These results demonstrate the pivotal role of mordants, particularly iron sulfate, in intensifying color strength.

When evaluating the color difference values (ΔE), a significant difference of 13.8 was observed between C0 and C2, calculated using the ∆E formula. In contrast, a minimal color difference of 2.2 was found between C1 and C3. A substantial color difference of 18.6 was noted between V0 and V2 for vegetable-tanned leathers.

These findings indicate a considerable color difference between leathers dyed with iron sulfate mordant and those not dyed, particularly for vegetable-tanned leathers. It can also be concluded that copper sulfate as a mordant does not significantly affect the dyeing process, particularly for chrome-tanned leathers. Similar results were reported by Bouagga et al. (2020) [33], who also found that iron sulfate was a more effective mordant than copper sulfate in their studies on wool.

Table 9. The color measurement results							
Data Name					$L^{*}(D65)$ a [*] (D65) b [*] (D65) K/S Pseudo Color		
CO (non-dyed)	60.866	-1.036	11.536 2.241				
$C1$ (Copper Sulfate) $54.090 -3.416$			5.418	2.641			
C ₂ (Iron Sulfate)	48.250	-3.072	6.254	4.126			
C3 (non-mordant)	54.094	-5.162	4.090	2.616			
$V0$ (non-dyed)	66.320	9.988	17.106 1.717				
V1 (Copper Sulfate) 53.654		10.770	15.864 3.373				
V2 (Iron Sulfate)	50.036	7.478	8.528	3.983			
V3 (non-mordant)	60.372	13.336	13.472 2.209				

Table 5. The color measurement results

3.3. Color Fastness to Cycles of To-And-Fro Rubbing Findings

In the to-and-fro rubbing fastness test results of leathers tanned with both chromium and vegetable tannins, it was observed that the dry felt did not leave any significant marks on the leather surface, and no color transfer occurred from the leathers to the felts (Figure 7).

When Table 7 is examined, it can be seen that the rubbing fastness of chrome-tanned leathers (5-4/5) is higher than that of vegetable-tanned leathers (4/5-4). This observation is consistent with findings from a review by Mandal and Venkatramani (2023) [34]. However, the wet rubbing fastness test results indicate a slight color transfer from the leather surfaces to the felts. Specifically, the wet rubbing fastness values for chrome-tanned leathers decreased to 3 or 4, while for vegetable-tanned leathers, both undyed and dyed samples showed values as low as 2. This decrease in wet rubbing fastness may be attributed to the hydrophilic properties imparted to the leathers by vegetable tannins. Previous studies have also reported that leathers dyed with other natural dyes exhibit lower wet rubbing fastness than dry rubbing fastness [31, 34-36].

Figure 7. Images of leathers after rubbing fastness test (Top row: chrome-tanned; Bottom row: vegetabletanned, in order from left to right according to the codes)

	Wet		Dry		
	Leather	Felt	Leather	Felt	
CO (non-dyed)	5	4	5	5	
C1 (Copper Sulfate)	4	4	4/5	5	
C ₂ (Iron Sulfate)	3	4/5	4/5	5	
C ₃ (non-mordant)	3	4/5	4/5	5	
V0 (non-dyed)	2	4/5	4	5	
V1 (Copper Sulfate)	2	4	4/5	4/5	
V2 (Iron Sulfate)	2	4	4/5	4/5	
V ₃ (non-mordant)	2	4	4	4/5	

Table 7. Rubbing fastness test results

3.4. Dyestuff Exhaustion Findings

For *Hibiscus sabdariffa* extraction, the maximum absorption wavelength was measured at 519.0 nm. The calibration curve and equation for the stock solution of *Hibiscus sabdariffa* extract are shown in Figure 8, and the absorbance values corresponding to different concentrations are presented in Table 8.

Figure 8. Calibration curve and curve equation of *Hibiscus sabdariffa* extract stock solution

Concentration (g/L)	Absorbance		
16.600	1.711		
8.300	0.763		
4.150	0.430		
2.080	0.266		
1.040	0.098		

Table 8. Absorbance values of the Hibiscus sabdariffa extract stock solution

5% dye solution (50 g of dye in 1000 mL of water) was used during the dyeing process. Based on the calibration curve results, it was determined that approximately 23 g of the 50 g of dye was absorbed by the chrome-tanned ostrich leather, leaving about 27 g of dye remaining in the bath. In contrast, for the ostrich leather tanned with vegetable tannins, around 43 g of dye was absorbed, with approximately 7 g remaining in the bath. The dyestuff exhaustion analysis was based on the stock solution and dye baths used for leather samples dyed without mordants. Samples dyed with mordants were excluded from this analysis due to the impact of mordants on the bath's color, which could affect the accuracy of the dye exhaustion measurements.

4. Conclusion

This study evaluated the dyeing properties of ostrich leathers using *Hibiscus sabdariffa* extract, a natural dye. Based on the analyses and tests conducted, several key findings were identified:

The *Hibiscus sabdariffa* extract produced distinct color variations on chrome-tanned and vegetable-tanned leathers.

The dye exhaustion analysis revealed that the chrome-tanned ostrich leather exhibited lower dye uptake than the vegetable-tanned leather (Mimosa), demonstrating a higher affinity for the dye. This suggests that the type of tanning process significantly influences the effectiveness of dye absorption, highlighting the potential of vegetable tanning in enhancing the coloration of ostrich leather.

Although the dye produced visible color variations on chrome-tanned ostrich leathers, it did not yield the expected red tones. Color measurement tests revealed that leathers mordanted with iron sulfate exhibited superior color intensity than undyed leathers. In contrast, those mordanted with copper sulfate displayed colors nearly identical to those of leathers without mordants. The findings suggest that post-mordanting yielded the best color results, improving the dye's effectiveness on ostrich leathers.

In conclusion, *Hibiscus sabdariffa* extract can successfully dye ostrich leathers, producing appealing shades, especially when iron sulfate is used as a mordant. These results pave the way for further investigations into using natural dyes in the leather industry, emphasizing the need for eco-friendly practices. Future studies could focus on the long-term durability of colors produced by *Hibiscus sabdariffa* extract and the exploration of additional natural dye sources.

Author Contributions

The first author identified the topic, directed the project, and supervised this study's findings. The first and third authors developed the theoretical and practical framework. The second author performed the experiments and obtained/ interpreted the findings. The second and third authors wrote the manuscript with support from the first author. The first authors reviewed and edited the paper. All authors read and approved the final version of the paper. This paper is derived from the second author's bachelor thesis supervised by the first author.

Conflicts of Interest

All the authors declare no conflict of interest.

Ethical Review and Approval

No approval from the Board of Ethics is required.

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