

Eco-friendly dyeing of leather and wood samples using *Parthenocissus* quinquefolia L. fruits aqueous extract

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Abstract – The present study is relevant to extracting natural colorant from *Parthenocissus* **Keywords:** quinquefolia L. fresh fruits and applying the extracted dye on leather and wood samples in Parthenocissus the presence and absence of various mordants. The effect of mordant type (copper sulfate Quinquefolia L, (CuSO₄.5H₂O) ferrous sulfate (FeSO₄.7H₂O) and alum (KAI(SO₄)₂.12H₂O) with different Leather, mordanting methods (pre-mordanting, meta-mordanting, post-mordanting) on dyeing quality was also examined. The effects of mordants on the color of dyed samples were investigated in terms of CIELab (L*, a*, b*) and K/S values. The dyed samples' light, washing, and Fastness rubbing fastness were evaluated according to ISO standards. Although generally good fastness values were obtained for all three mordants, light fastness was low. These results suggest that *P. quinquefolia* fruits have sufficient potential in dyeing leather and wood.

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1. Introduction

Wood.

Dyeing,

Until the second half of the 19th century, natural dyes were used extensively in dyeing fabrics, threads, ornaments, leather, hair, and wood. However, natural dyes were also used in dyeing foodstuffs [1]. Although their negative effects on humans were known, after the discovery of synthetic dyes in 1856, interest in natural dyes gradually decreased [2-3]. Environmentally friendly natural dyes exhibit many bioactive properties, such as antioxidants [4] and antimicrobials [5]. Despite their various advantages, some drawbacks are associated with natural dyes, such as low availability, high cost, and poor fastness of dyed fabrics. To overcome these problems, attempts have been made, which have mainly focused on using mordants [6].

Önem et al. [7] studied the five-year-old *Rubia tinctorum* plant roots for dye extraction. The application of dye was tested on chrome and vegetable-tanned Iranian sheep leather. Different mordants (potassium aluminum sulfate, copper sulfate, and ferrous sulfate) were used for dyeing analysis. The study concluded that chrometanned leather had better color fastness results than vegetable-tanned leather. The study analysis also described that extracted dye was antioxidant, sustainable, environment-friendly, and helpful in reducing aquatic pollution.

Özen et al. [8] examined the washing performance of wooden materials colored with madder (*Rubia tinctorum*) and determined that aluminum alum provided better adhesion for all samples. Okca [9] conducted studies on dyeing wooden toys with madder, buckthorn, and indigo and reported that walnut, oak, and beech trees were

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dyed better than hornbeam, mulberry, and boxwood trees and that the dyestuff was bonded better with aluminum alum. Additionally, he determined that the light fastness of the samples he painted increased by polishing them with coconut and soybean oil. Attlgan et al. examined the proliferation of natural dyes that are harmless to the environment and human health and their color change properties under rapid aging conditions and used 3% iron sulfate, 5% alum, 10% grape vinegar, and mordants in the dyeing. According to the experiment, while the least total color change value (ΔE) can be obtained in pinar paint with Scots pine wood and iron sulfate mordant, the highest color change can be achieved with aluminum sulfate mordant in Scots pine wood [10].

Parthenocissus quinquefolia L. is a deciduous climber plant that belongs to the Vitaceae family and is native to North America and can be found in Southern Africa, and Australia [11]. This plant is a very widely known ivy and ornamental plant, and its fruits are highly toxic to humans. It can be said that there are many flavonoids, such as anthocyanin groups in the chemical structure of the plant and many other substances. *P. quinquefolia* L. is used for many different purposes today. While the plant itself is used to prevent soil erosion, it is reported that some parts (fruit, root, bark) are used as an expectorant in traditional medicine, its fruits are used to relieve rheumatism pain, its roots are used as a diuretic, and its bark and branches are used as cough syrup. It is also an ornamental plant due to its pleasant appearance and perfect covering of fences and garden walls [12].

This study investigated the dyeing properties of chromed lambskin and pine wood samples with dye extract obtained after heat treatment from the ripe fruits of the *P. quinquefolia* L. plant. Copper sulfate, ferrous sulfate, and alum were used as a mordant to fix the dyestuff to the samples and change the color. Dyeings were achieved using the methods without mordanting and mordanting (pre-, meta-, post-mordanting). Colorimetric parameters (L*, a*, and b*) relative color strength (K/S) and Pantone color codes were carried out, and all results were interpreted. The dyestuff in *Parthenocissus quinquefolia* L. is shown in (Figure 1a) cyanidin-3-sophoroside (Figure 1) [13].



Figure 1. The chemical structure of cyanidin

2. Materials and Methods

2.1. Materials and Chemicals

2.1.1. Plant Material

P. quinquefolia fruits were harvested in Tokat (Türkiye) in November 2023. The ripe purple fruits of the plant were refluxed in the Soxhlet device 2 hours after they were collected, and the purple solution obtained (w/V, 50 g/500mL) was used in the dyeing process.

2.1.2. Preparation of Leather

Oat tanned leather sample was purchased from the Tokat Maturation Institute, Tokat, Türkiye. Chrome-tanned wet blue goat leather was cut into 8-cm diameter circular pieces per SLTC 1996 official sampling method and neutralized to pH 6.0–6.5 using sodium format (1%) and sodium bicarbonate (1%) solution.

2.1.3. Preparation of Wood

The wood of the yellow pine (*Pinus sylvestris*) was chosen for the experiments as a Türkiye native and commercially available species, frequently used in applications from furniture to constructions. Planned wood samples with $(50 \times 25 \times 25)$ mm dimensions and radial or semiradial faces were employed.

2.1.4. Chemicals and Devices

Mordants, such as alum hydrate (KAl(SO₄)₂.12H₂O, Merck), ferrous sulfate hydrate (FeSO₄.7H₂O, Merck), copper sulfate hydrate (CuSO₄. 5H₂O) Merck), were of analytical grade. Water was also used after distillation. Extraction was performed using the Soxhlet apparatus. Premier Colorscan SS 6200A Spectrophotometer assessed the color properties of the dyed samples in terms of CIELab values (L*, a*, b*, C*) and color strength (K/S) values. Color codes were determined by visual perception by the Pantone Color Guide. The light fastness (Atlas weather-ometer), wash fastness (Launder–ometer), and crock fastness (255 model crock–meter) of all dyed samples determined according to ISO 105–C06 and CIS were tested.

2.2. Extraction of Dyestuff Solution

50 g of fresh fruit of *P. quinquefolia* was refluxed to colorlessness using the Soxhlet device's distilled water (500 mL). This procedure continued until a total of 4 liters of colored extracts was obtained, and finally, all extracts were collected to get a stable color tone.

2.3. Leather Dyeing Without Mordant

The conventional chromed wet blue goat-tanned leather sample was purchased from the Tokat Maturation Institute, Tokat, Türkiye. Chrome-tanned wet blue goat leather was cut into (60x60) mm. Dyeing procedures were carried out for 2 hours at 45 °C. The dye bath was mixed with milk during the dyeing. At the end of the period, it was cooled, filtered, rinsed with deionized water, and dried on a flat surface at room temperature.

2.3.1. Pre-Mordanting

Leather samples (60 x 60) mm were mordanted by mixing in 0.1 M mordant solution at 40 °C for 20 minutes, and at the end of the period, they were filtered and rinsed with pure water. 100 mL of dye solution was added to the dye bath and was dyed at 40 °C for 120 min. After filtering, it was rinsed with water and dried.

2.3.2. Meta-Mordanting

Leather samples to be dyed, dyestuff and solid mordant (equivalent to 0.1 M concentration) were added to the dyeing container. It was dyed at 40 °C for 120 min. After being filtered, it is rinsed with water and dried.

2.3.3. Post-Mordanting

Leather samples were first dyed in 100 mL dye solution at 40 °C for 120 minutes. After, they were rinsed and mordanted in 0.1M 100 mL of mordant solution at 40 °C for 20 min. Finally, they were filtered, rinsed with water and dried.

2.4. Wood Dyeing Without Mordant

The wood of the yellow pine (*Pinus sylvestris*) was chosen for the experiments as a Türkiye native and commercially available species, frequently used in applications from furniture to constructions. Planned wood samples with (50 x 25 x 25) mm dimensions and radial or semiradial faces were employed. Wooden samples were mixed in a 200 mL dye bath at 60 °C for 120 min. After cooling, it was rinsed with deionized water and dried on a flat surface.

2.4.1. Wood Dyeing with Mordanting Techniques

2.4.2. Pre-Mordanting

Wood samples (50 x 25 x 25) mm were mordanted by mixing in 0.1M mordant solution at 60 °C for 20 minutes, and at the end of the period, they were filtered and rinsed with deionized water. Then, it was added to the 200 mL of dye bath, and dyed at 60 °C for 120 min. After filtering, it was washed with distilled water and dried [15-19].

2.4.3. Meta-Mordanting

Wood samples to be dyed, dyestuff and solid mordant (equivalent to 0.1 M concentration) were added to the dyeing container. It was dyed at 60 °C for 120 min. After being filtered, it was washed with distilled water and dried [15-19].

2.4.4. Post- Mordanting

Wood samples were first dyed in 100 mL dye solution at 60 °C for 120 minutes. After, they were rinsed and mordanted in 0.1M 100 mL of mordant solution at 60 °C for 20 min. Finally, they were washed with distilled water and dried [15-19].

2.5. Phytodyeing Quality Evaluation

The dyeing quality was evaluated using the color strength parameter (K/S), and the CIELab coordinates L*, a*, and b* of the dyed samples were achieved using Premier Colorscan SS 6200A Spectrophotometer). K/S value is relative color strength and was determined at 410 nm using the Kubelka Munk equation.

3. Results and Discussion

We dyed wood and leather samples using *Parthenocissus quinquefolia* L. fruit extract in this study. According to the fastness evaluations, washing and rubbing fastnesses of the dyed samples gave good results except for light fastness. Light fastness is moderate and below average. The fact that the fastness values obtained in dyeings with mordant are higher than those in dyeings without mordant shows that using mordant increases the fastness. The reason for this is the formation of strong internal complexes between the mordant cation and the auxochrome groups of the dyestuff molecule. Fastness values and color codes for dyed leather and wood samples have been presented in Tables 1 and 2, respectively.

Method	Mordant	Wash fastnes ^a	Rubbing fastness ^b	(wet-dry)	Light fastness ^c	Color code (Pantone)
Pre-	FeSO ₄	4	4	4/5	3-4	425CS
Meta-	FeSO ₄	4-5	4-5	4/5	4	411 CS
Post-	FeSO ₄	3	3	5	4	426CS
Pre-	CuSO ₄	4	4-5	4-5	4	409 CS
Meta-	CuSO ₄	4	4-5	4-5	4	410CS
Post-	CuSO ₄	4	4-5	4-5	3-4	WmGy8CS
Pre-	AlK(SO ₄) ₂	4	4	4	3-4	5015CS
Meta-	AlK(SO ₄) ₂	5	5	5	4	519 CS
Post-	AlK(SO ₄) ₂	4	4	4/5	3-4	425CS
	unmordant	4-5	3	4	4	5035 CS

Table 1. Fastness values and color codes of dyed leather

^aWash and ^brub fastness 1 = poor, 2 = moderate, 3 = fairly good, 4 = good, 5 = very good, ^cLight fastness 1 = very poor, 2 = poor, 3 = moderate, 4 = fairly good, 5 = good, 6 = very good, 7 = excellent, 8 = outstanding

When Table 1 is examined, washing, dry, and wet fastnesses in all dyed leather samples were found to be low (3) only for ferrous sulfate in meta-mordanting, while for other mordants, it was between 4 and 5. If we were to rank them in terms of fastness values for mordants, we can write it as:

$$Alum > CuSO_4 > FeSO_4$$

If we were to rank the mordanting methods in terms of their effectiveness, we can write it as:

 $Meta-mordanting > Pre-mordanting > Post\ mordanting > no\ Mordanting.$

Light fastnesses are four or below four for all three mordant and mordanting methods and are close to each other. As a result, according to Tables 1-3, it can be deduced also that mordanting improved wash and rubbing (wet-dry) fastness. Leather dyeing with *P. quinquefolia* extract shades of brown, bluish green with copper sulfate, shades of dark gray with ferrous sulfate, and lilac tones with alum were obtained.

Method	Mordant	Wash fastnes ^a	Rubbing fastness ^b	(wet-dry)	Light fastness ^c	Color code (Pantone)
Pre-	FeSO ₄	4-5	4-5	5	2-3	499 CS
Meta-	FeSO ₄	3-4	3-4	5	3	426 CS
Post-	FeSO ₄	4-5	4-5	5	3	411 CS
Pre-	CuSO ₄	4	3-4	5	3	5205CS
Meta-	CuSO ₄	3-4	3	5	2	424 CS
Post-	CuSO ₄	3	4-5	5	2	4495 CS
Pre-	AlK(SO ₄) ₂	4	4-5	5	3	5005CS
Meta-	AlK(SO ₄) ₂	3-4	4-5	5	3	5125 CS
Post-	AlK(SO ₄) ₂	4-5	4-5	5	2-3	5015CS
	unmordant	3	4-5	1/2	WmGy 2CS	3

Table 2. Fastness values and color codes of dyed wood.

^aWash and ^brub fastness 1 = poor, 2 = moderate, 3 = fairly good, 4 = good, 5 = very good, ^cLight fastness; 1 = very poor, 2 = poor, 3 = moderate, 4 = fairly good, 5 = good, 6 = very good, 7 = excellent, 8 = outstanding

As seen in Table 2, the washing fastnesses for all three mordants are slightly lower in the combined mordanting technique (3-4), while they are very good (4-5) in the pre- and post-mordanting techniques.

The wet rubbing fastness is good (3-4) when mordanted with iron sulfate and copper sulfate and has a medium value (3) when mordanted with copper sulfate. For dyeing done with alum, wet rubbing fastness was found to be very good (4-5) for all three dyeing methods. Dry rubbing fastness is good for all mordants and mordanting techniques (5). The fact that wet rubbing fastness is lower than dry rubbing fastness can be explained by the partial dissolution of the dyestuff in water. Light fastness is moderate (3) and below 3 for all three mordant and mordanting techniques. If we were to rank them in terms of fastness values, we can write it as:

$$FeSO_4 > CuSO_4 > Alum$$

If we were to rank the mordanting method in terms of its effectiveness, we could write it as:

Pre-mordanting > Post mordanting > Meta-mordanting>no Mordant

As a result, according to Tables 1 and 2, it can be deduced also that mordanting improved wash and rubbing (wet-dry) fastness. In wood dyeing with *P. quinquefolia*, shades of pale green with copper sulfate, shades of dark gray with ferrous sulfate, and lilac tones with alum were generated.

3.1. Determination of color performance

The pre–, meta– and post–mordanting dyed samples were investigated for fastness properties. The spectral reflectance measurements of the naturally dyed samples were determined using a spectrophotometer (Konica Minolta 3600d). Color strength was determined as K/S values of the dyed samples using the Kubelka-Munk equation [14]. K/S and L*, a*, and b* values of leather and wood samples are given in Tables 3 and 4, respectively.

Sample	Mordant/Method	L^*	<i>a</i> *	b^*	K/S
	FeSO ₄ (pre-)	47.83	0.047	-2.38	2.10
	FeSO ₄ (meta)	46.60	0.07	1.44	2.71
	FeSO4(Post)	47.33	-0.01	-2.14	2.20
	CuSO ₄ (pre-)	66.24	3.85	8.07	1.00
Ŧ .1	CuSO ₄ (meta-)	61.03	6.51	7.40	1.40
Leather	CuSO ₄ (post	63.38	5.93	12.42	1.01
	KAI(SO ₄) ₂ (pre-)	67.42	8.77	7.41	0.85
	KAI(SO ₄) ₂ (meta)	65.93	8.81	7.56	1.01
	KAI(SO ₄) ₂ (post)	71.43	2.74	7.91	0.73
	no Mordant	71.30	12.04	10.07	0.74

Table 3. K/S and L*, a*, and b* values of leather

	Table 4	. K/S and L* a* a	and b* values of	wood	
Sample	Mordant/Method	L*	a*	b*	K/S
	FeSO ₄ (pre-)	37.17	-0.85	1.39	5.40
	FeSO4(meta-)	31.63	-0.22	0.07	7.33
	FeSO4(post)	63.38	5.93	12.42	5.66
	CuSO ₄ (pre-)	33.16	0.77	-0.98	8.66
Wood	CuSO ₄ (meta-)	28.78	2.28	7.54	6.95
	CuSO ₄ (post-)	47.57	-0.14	13.77	5.00
	KAI(SO ₄) ₂ (pre-)	67.42	8.77	7.41	4.35
	KAI(SO ₄) ₂ (meta-)	4.30	7.28	2.37	4.66
	KAI(SO ₄) ₂ (post)	58.01	1.55	10.40	2.3
	no Mordant	44.12	8.21	7.25	3.80

Table 3 reveals that, for leather samples, mordanting increased relatively the color strength, especially in the case of leather mordanted with $FeSO_4$ (meta-mordanting (K/S =2.71) compared to no mordanted leather K/S =0.74). The color strength (K/S) of the leather samples increased according to the following order:

 $FeSO_4 > CuSO_4 > Alum > no Mordant$

Table 4 reveals that mordanting increased wood's color strength, especially in wood mordanted with $CuSO_4$ (pre-mordanting (K/S = 8.66) compared to no mordanted wood K/S = 3.80). The dyeing performances of the wood fabrics, based especially on the color strength (K/S) values, increase as follows:

$$CuSO_4 > FeSO_4 > alum > no Mordant$$

When the K/S values of dyed leather and wood samples are compared, we can write the following order:

Wood > leather

If we were to rank the mordanting method in terms of its effectiveness, we could write it as:

 $\label{eq:pre-mordanting} Pre-mordanting > Meta \ mordanting > Nordant$

Dyed examples of leather and wood samples with P. quequefolia fruit extract are shown in Figure 2.



Figure 2. Examples of leather and wood dyed with P. quequefolia fruit extract.

Compared with some studies in the literature, our wood dyeing results are in harmony with the findings obtained by Boahin et al. [20] in their wood dyeing studies with alum mordant. The analysis results obtained by Önem et al. [7] with *Rubia tinctorum* roots and the leather dyeing analysis results of Sundari [21] with *Mucuna pruriens* extract are compatible with our leather dyeing results.

Özen et al. [8] obtained high-fastness color tones in their wood dyeing work using *Rubia tinctorum* and alum mordant. This study is consistent with our findings. Ocak [9] achieved the best bonding with alum at pH = 9 in his wood dyeing study with indigo, buckthorn, and *Rubia tinctorum*. In our research, the best K/S value (8.66) was obtained in the pre-mordanting with copper sulfate, while the lowest K/S value (2.3) was obtained in the final mordanting with alum. Washing and rubbing fastnesses are compatible with our study.

The most important problem encountered in vegetable dyeings is their low light fastness. A study in the literature reported that lubricating the painted wood samples with coconut or soybean oil increased light fastness [9]. Moreover, an attempt will be made to increase the lightfastness by using the natural mordant mixture developed in the previous study carried out in our group [15-19]. All of these will be implemented in future projects, and analyses will be carried out to increase light fastness.

4. Conclusion

This study is interested in the dyeing properties of *P. quequefolia* fruit extract. It was found that this fruit is essentially composed of anthocyanins which are the main colouring components of the dye extract. Then, it was established that using mordant improves the phytodyeing quality resulting from the aqueous extract of *P. quinquefolia* fruits and also, leads to good fastness properties. The fastness properties of the leather and wood are improved by the use of mordants. Taking into account the fact that natural dyes have generally weak fastnesses. Studies that protect the environment and all living things are followed with interest. These eco-friendly dye extracts could be of scientific or industrial interest for wood and leather coloring in the future. Considering that in this study, especially the wood and leather samples with ferrous sulfate mordant were dyed in exotic and attractive colors, it can be said that these color tones will attract consumers' attention. When all the results were evaluated, it was understood that *P. quinquefolia* L. fresh fruits could be used in dyeing leather and wood.

Author Contributions

All the authors equally contributed to this work. They all read and approved the final version of the paper.

Conflict of Interest

All the authors declare no conflict of interest.

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