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Progress in Color Colorants Coating 18 (2025), 363-373

Ministry of Science, Research and Technology
Institute for Color Science & Technology

Sustainable Wool Dyeing: Utilizing Syzygium Aromaticum (Clove Bud) Extract and Plant-Derived Mordants

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ARTICLE INFO

Article history:
Received: 30 Nov 2024
Final Revised: 07 Mar 2025
Accepted: 17 Mar 2025
Available online: 07 May 2025

Keywords:
Eco-friendly dyeing
Syzygium aromaticum (Clove)
Bio-mordant
Color strength
Fastness properties

ABSTRACT

Pollution from synthetic dyes and metallic mordants used in the dyeing industry is a significant environmental problem that poses risks to human, animal, and ecosystem health. This study investigates the dyeing of wool yarn using clove bud extract as a natural colorant and various bio-mordants (pomegranate rind, myrobalan, date kernel, oak bark, and pinecone) as eco-friendly alternatives to metal mordants. Colorimetric properties, including L, a*, b*, C*, h°, color strength, wash, and light fastness, were evaluated for samples dyed using the pre-mordanting, simultaneous, and post-mordanting methods. The results showed that bio-mordants, especially myrobalan, gave comparable color strength and better light fastness than metal mordants (alum). While copper sulfate led to darker shades, it resulted in lower brightness and a greenish-brown hue. Bio-mordants, on the other hand, gave the dyed yarns a more reddish-yellow hue. The post-mordanting method generally produced the highest color strength, while the simultaneous method produced the lowest. The findings suggest that bio-mordants offer a promising alternative to metal mordants for dyeing wool yarn with clove bud extract, providing a more sustainable and environmentally friendly approach to textile dyeing. Prog. Color Colorants Coat. 18 (2025), 363-373© Institute for Color Science and Technology.*

1. Introduction

Today, more than 10,000 synthetic dyes are produced in the textile, food, paper, and cosmetics industries. The textile industry consumes an incredible 1.07 million tons of synthetic dyes annually, more than any other industry [1]. The extensive use of synthetic dyes and other auxiliaries in dyeing has resulted in harmful chemical compounds being released into the environment via textile industry wastewater. Previous studies have shown that about 12 % of the synthetic dyes used in the textile industry enter wastewater, and about 20 % of this amount is released back into the environment along with the treated wastewater [2].

As mentioned, despite the textile wastewater treatment,many chemical substances used in the dyeing process are still released into the environment via wastewater from industrial factories. They have led to the pollution of ecosystems, including soil, water, and the human body. Pollutants emitted by the textile industry include organic and inorganic compounds such as formaldehyde, heavy metals, sulfur, nitrate, naphthol, benzidine, soaps, phenols, chromium, and chlorine [3]. The presence of these pollutants in water and soil can have harmful effects, including blocking sunlight and inhibiting photosynthesis in aquatic plants. They can also inhibit the germination of plant seeds and cause them to fail to grow [4, 5]. These pollutants can have long-lasting effects on the environment by persisting in water and soil, entering the food chain through plant uptake, and endangering the health of humans and other organisms. The toxic nature of these pollutants has mutagenic, carcinogenic, and genotoxic

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https://doi.org/10.30509/pccc.2025.167426.1343

effects. They can lead to malfunctions in various organs of the human body [6]. In recent years, some efforts have been made to develop the use of natural substances in the dyeing process to reduce the harmful effects of chemical substances. Natural colorants and bio-mordants are environmentally friendly alternatives that decompose naturally and thus reduce the accumulation of toxic waste [7]. These colorants have calming properties and are used in certain cases to promote the human body's well-being. Natural colorants can be used for special purposes on textiles, e.g., for coating with antimicrobial, anti-allergic, and UV-protective agents [8-10].

Syzygium aromaticum (clove bud) is one of the plants that not only has coloring properties but also has a positive effect on the health of the human body. Clove bud is a flower from a tree in the Myrtaceae family. This tree is native to Indonesia, India, and the Madagascar islands and usually grows to a height between 7 and 13 meters. The buds of this plant are initially pale red and then turn green and brown when harvested. The harvested clove buds are usually 1.5 to 2 cm long and are characterized by an elongated calyx surrounded by four sepals. Figure 1a shows the appearance of the harvested clove bud. The clove bud is considered one of the most important medicinal plants and has been used since ancient times for its antifungal, antibacterial, and soothing properties [11-13].

Clove buds contain a total of 23 constituents, with eugenol being the most abundant at 76.8 %, followed by β-caryophyllene, alpha-humulene, ferulic acid, eugenol acetate, caffeic acid, and ellagic acid [14-16]. The researcher previously reported eugenol in clove, supporting its potential as an active molecule responsible for the color of clove buds (Figure 1c).

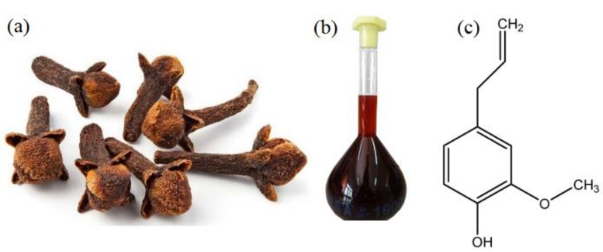

Figure 1: Clove buds: (a) physical appearance, (b) clove bud extract, and (c) chemical structure of eugenol [17].

In its pure form, eugenol is not a colorant; however, due to the presence of phenolic and allyl groups, it reacts with other substances, subsequently generating chromatic compounds. The phenolic groups within eugenol can form chromatic complexes with metal ions, such as iron or copper. These complexes can exhibit various hues depending on the metal and reaction conditions [17, 18].

The colored compounds in clove buds have led to research into their possible use as a natural dye. In a study by Yameen et al. [17], the researchers investigated the feasibility of using clove buds as a natural dye for wool yarn. They investigated the use of microwave treatment and optimized its application using a central composite design. The researchers found that using microwave radiation in extraction is more economical and environmentally friendly than traditional extraction methods. Ahmadi et al. [18], Lee et al. [19], and Hwang et al. [20] conducted studies in which they investigated the colorimetric parameters as well as the deodorizing, antibacterial, and antimicrobial properties of wool, cotton, and silk fibers dyed with clove extract in the presence of metal mordants. The results of these studies indicate that clove buds have beneficial antibacterial and deodorizing properties and can also produce high-quality yellow or brown colors, especially on wool fibers. Rajabi et al. [8] and Tehrani et al. [13] investigated the influence of various dyeing parameters, including the type and concentration of mordant, temperature, and dye concentration, on the color obtained by dyeing with clove buds on wool fibers. The researchers found that by changing the dyeing parameters and using different metal mordants, a range of colors, such as yellow, brown, and dark gray, can be achieved on wool fibers. Furthermore, the dyed fabrics exhibited suitable washing fastness properties.

Despite the advantages of natural colorants, their use is associated with certain limitations. The color strength of textiles dyed with natural colorants usually decreases significantly when exposed to sunlight and heat or when washed and rubbed [21]. To overcome these limitations, researchers have suggested using metal salts for mordanting [22]. As mentioned above, using heavy metals as mordants can lead to various environmental problems [5]. In recent years, the use of natural substances as mordants in dyeing has been explored. These natural mordants, or bio-mordants, are obtained from natural sources, especially agricultural waste products. These materials have environmentally friendly properties, and their performance in terms of color fastness and color strength is comparable to or even better than metal mordants [23]. Bio-mordants utilized in previous studies include myrobalan [23, 24], oak bark [23], sumac [17], pomegranate peel [17, 25], walnut shells [26], pine cones [27], and lemon peel [27].

Previous studies have not comprehensively investigated the colorimetric and fastness properties of textiles dyed with clove buds and plant-based mordants. This study presents a novel approach to dyeing wool yarns, using clove bud as a natural colorant and bio-based mordants as a sustainable and environmentally friendly alternative to synthetic dyes and metal mordants. In addition, this study conducts a comprehensive investigation into the type of bio-mordant and provides valuable insights for selecting the most suitable dyeing method. An important innovation of this study is the demonstration that bio-based mordants can produce dyed yarns with comparable or even better strength and color fastness than conventional metal mordants. This finding is important, especially for hand-woven carpets and traditional clothing industries.

2. Experimental

2.1. Materials and dyeing process

A solvent mixture of 70 % distilled water and 30 % ethanol was used to extract the colorants from the clove bud powder. The mixture was heated to 70 °C and maintained at this temperature for 90 minutes. The extracted solution was passed through a Whatman No. 42 filter to obtain a filtrate with a concentration of 0.2 g/L (Figure 1b). It was heated to 80 °C for 5 minutes before being used to remove the ethanol from the dyeing solution.

Before dyeing or mordanting, the 100 % pure semi-worsted wool yarn (200 tex/2 fold and 100 TPM) from an Iranian company was cleaned by soaking it for 30 minutes at 60 °C in a non-ionic detergent solution (2 mL/L of Lissapol NC) and then washed it thoroughly with tap water. For mordanting, both metal mordants (potassium aluminum sulfate (Al) and copper II sulfate (Cu) from Merck, Germany) and bio-mordants (pomegranate rind, myrobalan, date kernel, oak bark, and pinecone) were utilized.

The clove buds and natural mordants, obtained from local markets in Iran, were washed with distilled water to remove the dust, dried, and ground using an industrial milling machine. The dyeing process used pre-mordanting, simultaneous, and post-mordanting techniques.

In the mordanting process, the wool yarns were treated with bio-mordants at a concentration of 15 % owf, copper II sulfate at 5 % owf, and potassium aluminum sulfate at 10 % owf. The dyeing process used an exhaust method with 80 % owf colorants. The mordanting or dyeing process began with the wool yarns immersed in a mordanting/dyeing bath at 40 °C. The temperature was then raised to 80 °C and maintained at this temperature for 60 minutes [8, 13]. The dyeing and mordanting processes were carried out at a pH of 4.5 and a liquor ratio 50:1 [28]. Acetic acid from Merck, Germany, was utilized to adjust the pH value.

2.2. Colorimetric and fastness characterization

The color properties of the dyed sample, including reflectance, L*, a*, b*, C*, h°, λ max, and relative color strength ($(K/S)_R$), were measured using a YS6010 benchtop reflectance spectrophotometer under D65 lighting conditions and a 10° standard observer. Eq. 1 was used to quantify the color change between the un-mordanted (control) and mordanted yarns.

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (1)$$

Where ΔL is the difference in lightness between the mordanted and control samples, Δa is the difference in a* between the mordanted and control samples, and Δb is the difference in b* between the mordanted and control samples. The Kubelka-Munk equation (Eq. 2) and Eq. 3 were used to calculate the color strength and the relative color strength values.

$$K/S = \frac{(1-R)^2}{2R} \quad (2)$$

$$(\frac{K}{S})_R(\%) = \frac{(\frac{K}{S})_m}{(\frac{K}{S})_{um}} \times 100 \quad (3)$$

Where $(\frac{K}{S})_m$ is the color strength of the mordanted sample, $(\frac{K}{S})_{um}$ is the color strength of the control sample, and R is the reflectance value of the sample at the wavelength of maximum absorption.

The wash fastness of the dyed samples was determined using ISO 105-C06 (A1S):2010. The samples were washed in a laboratory machine with 4 g/L non-ionic detergent at 40 °C for 30 minutes. After washing, the samples were rinsed with running tap water, squeezed, and dried. The color change of the samples was assessed using the ISO 105-A02 grayscale with a rating scale of 1 to 5. The light fastness of the dyed samples was assessed using ISO105-B02 and a blue scale (grades 1 to 8).

3. Results and Discussion

Digital images of wool yarns dyed with clove bud extract as well as metal and bio-mordants are presented in Figure 2. By combining metal mordants and polyphenolic organic mordants with clove colorants, different shades of brown, from light brown to dark brown, can be achieved. The color of the Cu-mordanted sample differs slightly from that of the other samples. The color of this sample is greenish-brown. Metal ions (in metal mordants) or hydroxyl and carbonyl groups in bio-mordants (Figure 3) [29-35] can form coordinate or hydrogen bonds with amino and carbonyl groups in wool fibers as well as with hydroxyl groups in clove colorant (Figure 4) [17]. The structure of eugenol obtained from clove buds contains a hydroxyl group. Adding acid to the dye bath leads to the protonation of the carboxyl groups (COO^-) in the wool fiber by H^+ ions from the acid, thus giving the fiber a positive net charge [36]. The acidic medium thus facilitates the transfer of electrons from the carboxyl and amino groups (positive ligands) within the wool structure to the metal mordants, promoting the formation of coordinate bonds between these groups [37]. Metallic or phenolic bio-mordants and their bonds have caused a color change in wool fibers dyed with clove bud extract.

The color properties of the samples dyed with metal and bio-mordants and various dyeing methods are presented in Tables 1 to 3. The results reveal that the sample mordanted with Cu has a lower brightness than the other samples for all dyeing methods. The brightness of this sample is 25-38 % lower than that of the control sample and 21-35 % lower than that of the other samples. In all dyeing methods, the sample mordanted with Al exhibited an equally or higher brightness than the bio-mordant samples. In the simultaneous method, the Al sample showed a significantly higher brightness (10 to 15 %) than the bio-mordant samples. Among the bio-mordanted samples, the sample with oak bark is slightly darker (5-6 % in different dyeing methods) than the others.

Table 1: Color parameters of the dyed yarns (pre-mordanting).

sample	L*	a*	b*	ΔE	c*	h*	K/S	$(K/S)_R$	Washing fastness	Light fastness
Control	58.34	7.59	33.93	--	34.77	77.39	14.40	--	3-4	4-5
Al	61.18	6.47	38.08	5.14	38.62	80.36	13.47	93.54	4-5	5
Cu	42.90	3.26	27.79	17.17	27.98	83.30	19.26	133.75	4-5	6
Pomegranate rind	58.70	7.18	34.11	0.57	34.86	78.11	13.64	94.72	5	5-6
Myrobalan	56.83	7.22	34.59	1.69	35.34	78.21	14.08	97.77	5	6-7
Date kernel	56.57	8.03	33.13	1.99	34.09	76.38	14.03	97.43	4-5	5-6
Oak bark	54.90	8.48	30.75	4.77	31.90	74.59	13.85	96.18	4-5	5-6
Pinecone	59.04	7.72	33.47	0.85	34.35	77.00	13.10	90.97	5	5-6

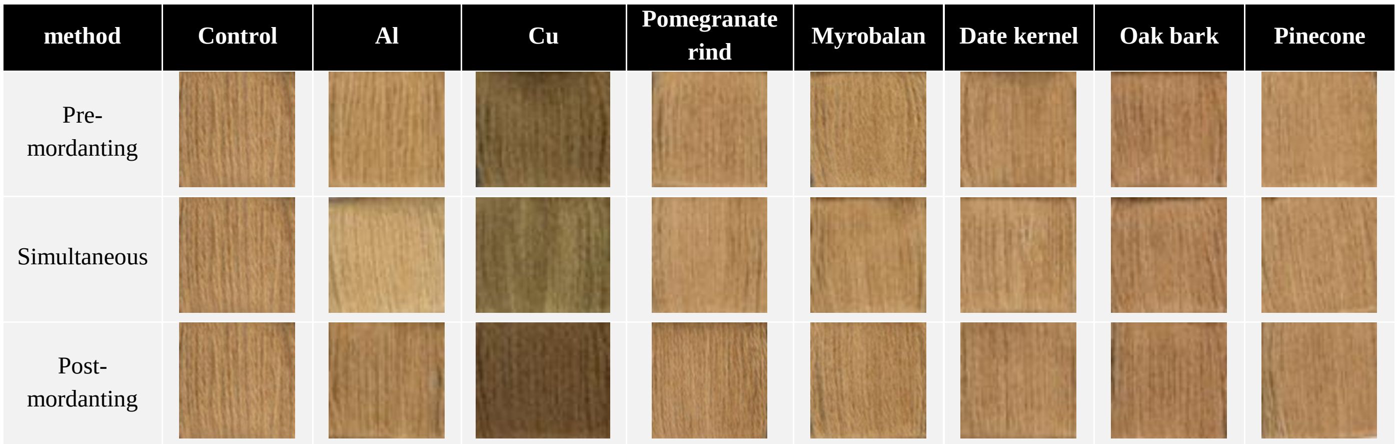

Figure 2: Digital images of the dyed wool yarns with metal and bio-mordants.

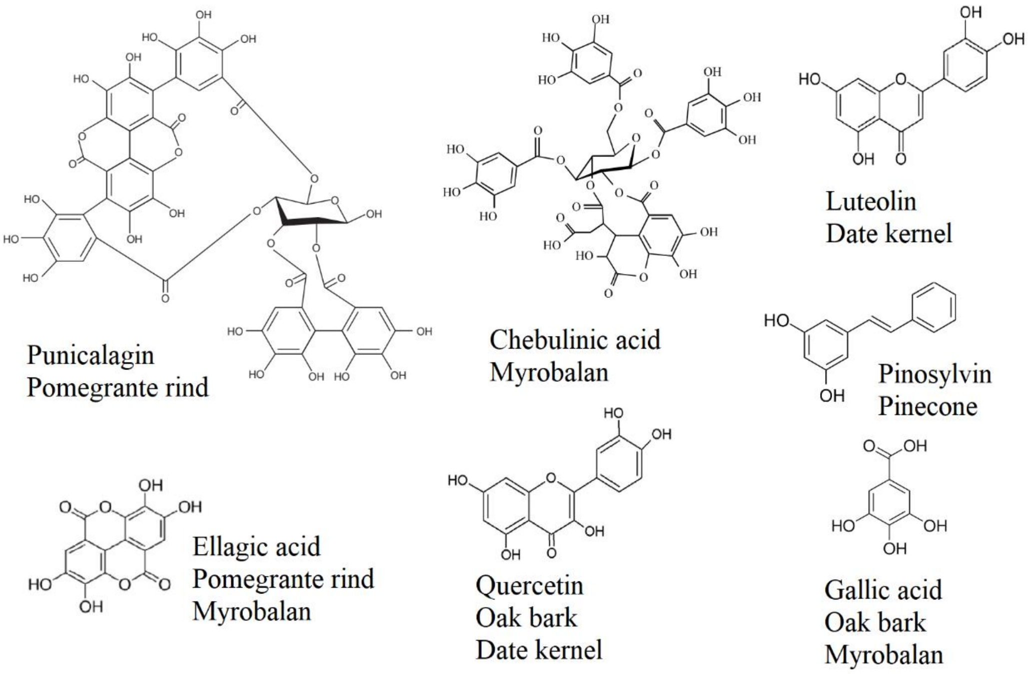

Figure 3: The major components of natural mordants [27, 35].

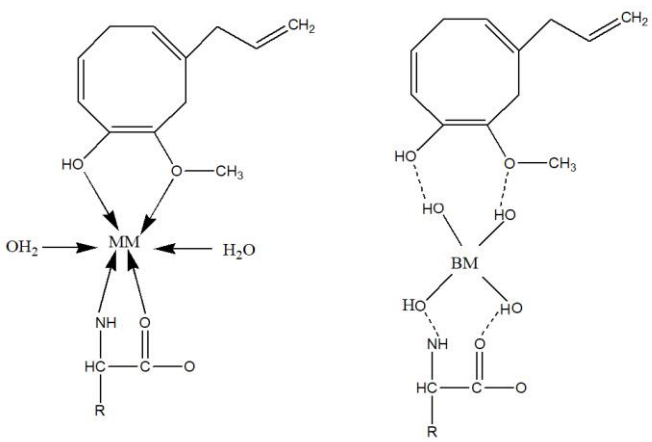

Figure 4: Proposed mechanism of colorant-fiber bonding with metallic (MM) and bio-mordant (BM) [17].

Table 2: Color parameters of the dyed yarns (simultaneous).

sample	L*	a*	b*	ΔE	c*	h*	K/S	$(K/S)_R$	Washing fastness	Light fastness
Control	58.34	7.59	33.93	--	34.77	77.39	14.40	--	3-4	4-5
Al	67.03	4.49	37.75	10.31	38.02	83.22	9.80	71.42	4-5	5
Cu	43.56	1.61	27.61	16.76	27.66	86.67	18.47	134.62	5	6
Pomegranate rind	59.68	7.31	34.26	1.74	35.03	77.96	13.06	95.18	4-5	5-6
Myrobalan	58.65	6.99	34.50	1.23	35.20	78.55	13.51	98.46	4-5	6-7
Date kernel	60.00	7.21	33.17	1.92	33.94	77.74	12.83	93.51	4-5	5-6
Oak bark	57.00	7.82	30.63	3.09	31.62	75.68	13.14	95.77	5	5-6
Pinecone	59.21	7.61	33.09	1.17	33.95	77.05	13.72	100	5	5-6

Table 3: Color parameters of the dyed yarns (post-mordanting).

sample	L*	a*	b*	ΔE	C*	h°	K/S	$(K/S)_R$	Washing fastness	Light fastness
Control	58.34	7.59	33.93	--	34.77	77.39	14.40	--	3-4	4-5
Al	55.51	6.72	34.18	2.69	34.84	78.87	14.35	98.02	5	5
Cu	36.13	4.43	23.92	24.13	24.33	79.50	23.05	157.44	4-5	6
Pomegranate rind	56.65	7.34	32.54	1.74	33.35	77.29	14.99	102.39	5	6
Myrobalan	56.22	7.61	34.10	1.89	34.94	77.42	14.08	96.17	5	6-7
Date kernel	56.71	7.38	31.52	2.45	32.38	76.82	14.79	101.02	4-5	5-6
Oak bark	53.29	8.11	29.68	6.17	30.77	74.73	14.50	99.04	5	6
Pinecone	55.66	7.75	31.31	3.29	32.25	76.10	13.43	91.73	5	5-6

The results presented in Tables 1 to 3 and Figure 5 indicate that although the type of mordant and the dyeing method influence the values of a* and b*, the a*-b* quadrant of the CIELab color space remains unchanged. The control and mordanted samples' a* and b* parameters are in the red-yellow quadrant of the CIELab color space for all dyeing methods. The results of a* and b* show that in all dyeing methods, the sample with bio-mordants have more redness and yellowness than the Cu sample. Using Cu ions as a mordant led to a decrease in the redness of the dyed sample. The digital images in Figure 2 confirm these results. The sample with bio-mordants show a higher redness than the Al sample, especially in the simultaneous method. However, their yellowness is 20 % lower than that of the sample mordanted with Al in both the pre-mordanting and simultaneous methods.

The results indicate that the saturation (C*) of the samples mordanted with bio-mordant is 11 to 20 % higher than that of the Cu samples and lower than that of the Al sample in the pre-mordanting and simultaneous methods (Tables 1-3). The saturation of these samples is 11 to 20 % higher than the Cu sample in the pre-mordanting and simultaneous methods. In the post-mordanting method, the saturation of the bio-mordant samples is 22 to 32 % higher than that of the Cu samples. In this method, the saturation of the bio-mordanted samples does not differ significantly from that of the Al samples. The results of the color change between the control and the mordanted samples (ΔE) show that using metal mordants, especially Cu and oak bark, causes a color change. However, using other bio-mordants had no significant effect on the color change (Figure 2 and Tables 1-3).

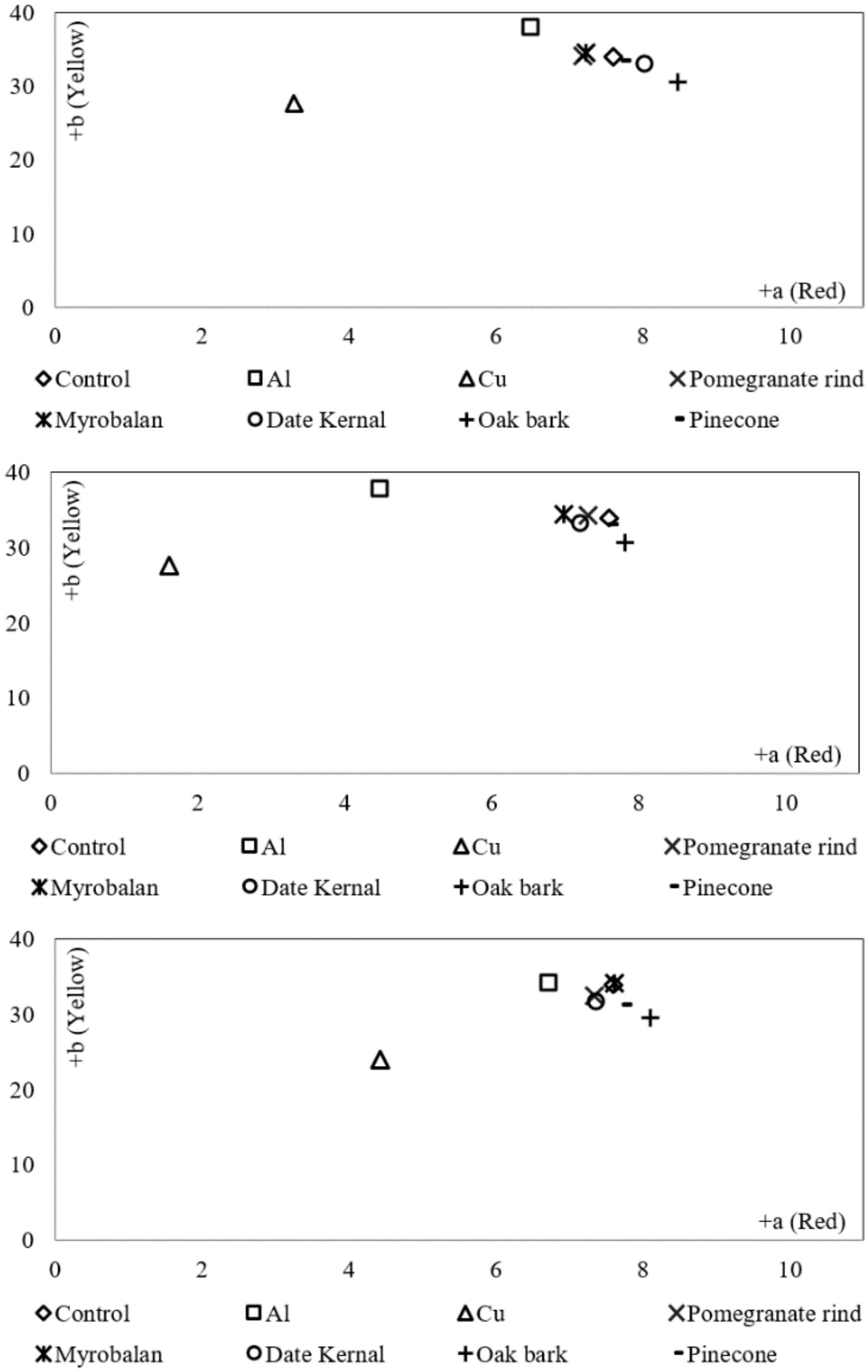

Figure 5: The a*–b* plots of the dyed wool samples with (Row 1): pre-mordanting, (Row 2): simultaneous, and (Row 3): post-mordanting methods.

Reflectance spectroscopy analysis was performed in the wavelength range of 400-700 nm to compare the color strength of the dyed samples. As shown in Figure 6a-c, the color strength of all samples decreases in the wavelength range from 400 to 700 nm for all dyeing methods. All samples show a maximum absorbance at 400 nm, corresponding to yellow.

As shown in Figures 6 and 7, the change in dyeing method and type of mordant has resulted in a shift in absorbance at different wavelengths, particularly at the maximum absorbance wavelength of 400 nm. The Cu samples show the lowest reflectance and the highest absorbance for all dyeing methods. As shown in Figure 7, the color strength of the Cu sample at the maximum absorbance wavelength is significantly higher than that of the other samples, reaching up to 32 % for the pre-mordanting, 47 % for the simultaneous, and 39 % for the post-mordanting. In the simultaneous method, the sample mordanted with Al has a lower color absorption than other samples at 400 to 550 nm wavelengths. However, at a wavelength of 550 to 700 nm, its absorbance does not differ from that of the control and bio-mordant samples. The color strength of this sample at the maximum absorbance wavelength is 24 to 32 % lower than that of the control and bio-mordant samples. The results show that in the simultaneous method, the color strength of the bio-mordant samples (at the wavelength of maximum absorbance) is relatively uniform, ranging between 12.83 and 13.72. In addition, the relative color strength ($(K/S)_R$) of the bio-mordant samples in this method is 93.51 to 100 % compared to the control sample. There is no significant difference in the absorbance of the control, Al, and bio-mordanted samples in the pre-and post-mordanting methods. The color strength of these samples at the maximum absorbance wavelength is between 13.10 and 14.40 for the pre-mordanting method and between 13.43 and 14.99 for the post-mordanting method (Figure 7 and Tables 1-3). In the pre-and post-mordanting methods, the relative color strength of the samples with bio-mordant is 90.97 to 97.77 % and 91.73 to 102.39 %, respectively, compared to the control sample.

The results demonstrate that the post-mordanting method gives the highest color strength among all the dyeing methods. In contrast, the simultaneous method gives the lowest color strength for most of the dyed samples, except for the pinecone sample (Figure 7). The post-mordanting method consistently produces dyed samples with a color strength that is 4 to 31% higher than the simultaneous method. Post-mordanting provides more control over the final color. The dye can be applied to achieve the desired shade, and then the mordant can enhance or change the shade. The simultaneous method uses a dye bath that contains both dye and mordant so that a dye-mordant complex can form. However, the size of this complex can hinder its diffusion into the fibers, resulting in reduced color strength in the dyed samples.

For textiles dyed with natural colorant, checking the color fastness properties and strength is crucial. This part of the research examines the effect of dyeing methods and mordants on color fastness to wash and light. The results of dyed wool samples' wash and light fastness are shown in Tables 1 to 3. As can be seen, the dyed wool sample with clove bud extract and without mordant (control sample) has a low wash and light fastness. As shown in Figure 4, metal and bio-mordants can form coordinate or hydrogen bonds with the ligands in the wool fibers and the clove colorant and increase the fastness properties [17]. In addition, combining mordants with the dye increases the size of the formed complex and reduces its solubility, thereby reducing its diffusion from the substrates and improving fastness [38].

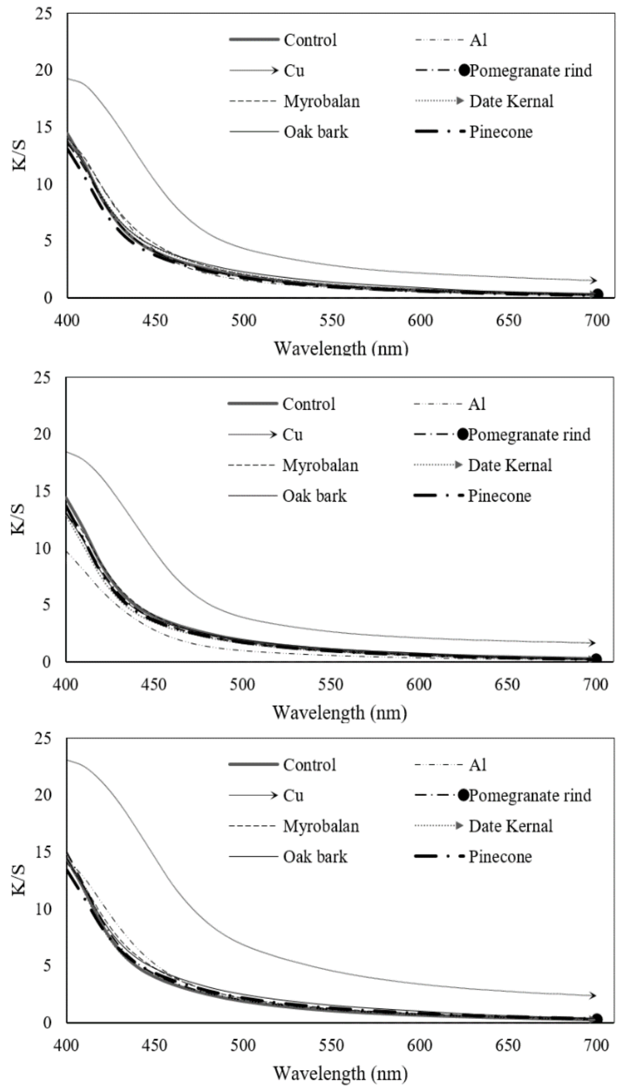

Figure 6: Color strength of dyed yarns with various mordanting methods; (Row 1) pre-mordanting, (Row 2) simultaneous, and (Row 3) post-mordanting.

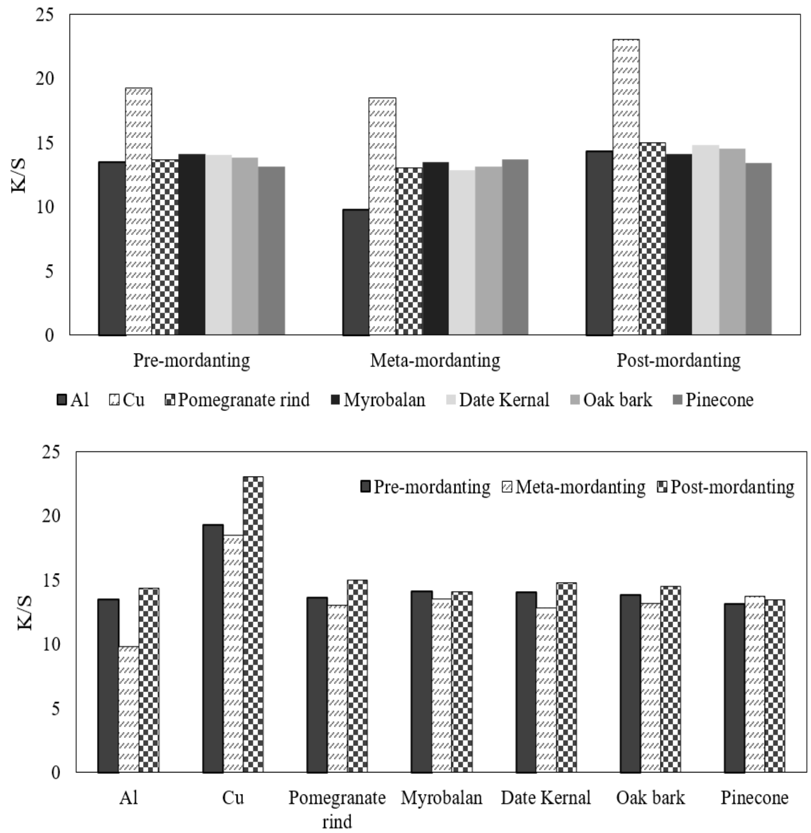

Figure 7: Color strength of dyed yarns with various mordanting methods (Row 1), as well as metal and bio-mordants (Row 2).

The results indicate that all samples dyed with clove bud extract, combined with metal and bio-mordants, have better wash and light fastness properties than the ISO standard. According to the ISO standard, a wash fastness value of more than 4 on the grayscale and a light fastness value of more than 5 on the blue scale is considered acceptable. A comparison of the fastness results of the samples dyed with metal and bio-mordants shows that all samples have a similar wash fastness for all dyeing methods, with values between 4-5 and 5 on the grayscale. Furthermore, the light fastness of bio-mordanted samples is either equal to or better than that of the metal samples. Among the bio-mordants evaluated, myrobalan demonstrated the highest light fastness. The presence of polyphenolic components within bio-mordants (as shown in Figure 4) contributes to their UV absorption properties and thus increases lightfastness [39].

4. Conclusion

This study investigated the dyeing of wool yarns using clove bud extract as a natural colorant and various bio-mordants (pomegranate rind, myrobalan, date kernel, oak bark, and pinecone) as eco-friendly alternatives to metal mordants. The colorimetric properties, including L*, a*, b*, C*, h°, color strength, washing, and light fastness, were evaluated for samples dyed using pre-mordanting, simultaneous, and post-mordanting methods. The results show that:

- The colorimetric and fastness properties of the dyed samples were influenced by factors such as the type of mordant and the dyeing method.
- Utilizing metal and bio-mordants with clove colorants produced diverse brown hues, indicating the potential for tailoring color outcomes based on specific application requirements.
- Bio-based mordants, especially myrobalan, oak bark, and pinecone,can produce dyed yarns with comparable or superior color strength and fastness properties to traditional metal mordants (Alum). All samples exhibit similar washing fastness, ranging from 4-5 to 5 on the grayscale. Furthermore, the light fastness of bio-mordant samples is either equivalent to or superior to metal samples. This finding underscores the potential

of natural materials in developing more sustainable textile dyeing processes.

- The choice of dyeing method (pre-mordanting, simultaneous, and post-mordanting) significantly influenced the color strength. Post-mordanting consistently yielded the highest color strength, indicating that the sequence of mordanting and dyeing plays a crucial role in achieving desired color outcomes. Also, all samples exhibit similar washing and light fastness across all dyeing methods.

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How to cite this article:
Tehrani M, Asadi Fasrsani T. Sustainable Wool Dyeing: Utilizing Syzygium Aromaticum (Clove Bud) Extract and Plant-Derived Mordants. Prog Color Colorants Coat. 2025;18(3):363-373. https://doi. org/10.30509/pccc.2025.167426.1343.