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Evaluation of Alpaca Yarns Dyed with Buddleja Coriaceous Dye and Metallic Mordants

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Abstract: The objective of this research was to evaluate the effect of dye obtained from *Buddleja coriacea* and metallic mordants on the chromatic properties, textile characteristics, spectral profiles, and color stability in alpaca fibers. The dye extraction technique involved boiling in an aqueous solution, followed by filtration. Subsequently, alpaca yarns were dyed using the resulting extract following a standard protocol. The applied mordants included sodium sulfate (Na_2SO_4), aluminum sulfate and potassium dodecahydrate ($KAl(SO_4)_2{\cdot}12H_2O$), and oxalic acid ($C_2H_2O_4$). Spectroscopy UV-Vis and FTIR spectrophotometry methods were used for the characterization of the dyed samples and analysis of the dye during the dyeing process. The findings revealed the formation of four distinct color tones. Additionally, it was determined that the mordants influenced the chromatic properties of the fibers dyed with *Buddleja coriacea* extract without modifying their textile characteristics. The identified spectral bands corresponded to keratin, the structural protein of the fibers. Changes in the intensity of these spectral bands were observed in the dyed samples, attributable to the presence of different mordants. Wet rub fastness was found to be inferior to dry rub fastness, which has implications for textile maintenance. In conclusion, *Buddleja coriacea* flowers provide an effective yellow dye, and when combined with various mordants, they allow for a variety of shades and hues in alpaca fiber yarns.

Keywords: alpaca yarns; natural dyes; Buddleja coriacea; metallic mordants; colorimetric properties

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1. Introduction

The contemporary textile industry is increasingly oriented toward more ecological and sustainable practices. In the Peruvian context, the richness of natural resources is remarkable, and South American camelids such as llamas, alpacas, guanacos, and vicuñas play a significant role in the economy and culture [1]. Alpaca, valued for its fiber, exhibits qualities such as durability, fineness, softness, and a range of natural shades, with keratin being the primary component of the fiber, composed of three layers: cuticle, cortex, and medulla [2–5]. Indicators such as mean fiber diameter (MFD) and comfort factor (CF) are crucial for assessing its quality [6]. However, the current scientific literature lacks extensive research on the application of natural dyes, specifically Buddleja coriacea, in alpaca fiber dyeing. A recent study by Morales and Perez (2022) indicates that Polylepis shows effective adherence to textile substrates, with or without mordants, under controlled conditions. The dyed samples exhibit colorfastness against washing, dry rubbing, and light exposure, with chromatic variations when using biomordants such as lemon juice or sodium chloride [7].

Other researchers have explored dyeing alpaca fibers using a variety of natural resources, both plant-based and animal-based [8,9].

The shrub *Buddleja coriacea* (herbarium code B084), endemic to the Peruvian Andean highlands between 3000 and 4000 m above sea level, is harvested between March and June for the extraction of a natural yellow dye. This dye is used in dyeing alpaca yarns destined for textile and artisanal production [10–12]. Natural dyes offer ecological and aesthetic benefits compared to synthetic dyes, which often contain harmful chemical agents [13–19]. According to Oxelman et al. (2004), flavonoids such as apigenin, luteolin, and 6-hydroxyluteolin are the main pigments in the Buddlejaceae family, capable of producing a wide range of colors from white to yellow, orange, pink, and purple [20]. However, there is limited research on the process of dyeing alpaca fibers with natural dyes and mordants. In a recent study, Quispe et al. (2020) extracted the yellow dye from *Buddleja coriacea* flowers to dye alpaca fibers, evaluating colorfastness through 16 treatments with variations in concentrations, dyeing times, and temperatures. They concluded that time and temperature are determining factors in colorfastness, explaining 91.52% of the variability in resistance to sunlight [21].

This study focuses on the application of natural dyes, specifically Buddleja coriacea, in the dyeing of alpaca fiber. The importance of this research lies in the search for ecological and sustainable alternatives to synthetic dyes, taking advantage of Peru's wealth of natural resources. The novelty of this study lies in the comprehensive investigation of the dyeing process with Buddleja coriacea, including the evaluation of textile characteristics, colorimetry parameters, chemical bands, colorfastness, and the identification of key factors in this process.

The conventional method of dyeing through heat treatment involves immersing yarns in an aqueous solution containing both the dye and the mordant. In this process, the dye adheres to the fiber's surface through covalent bonds [11]. Due to the limited affinity between the fiber and the dye, mordants are commonly necessary. These mordants facilitate the formation of stronger chemical bonds between the dye and the fiber, ensuring a more durable union. This process is referred to as complexation, where the mordent's metal ions bind to the functional groups of both the fiber and the dye, creating more stable dye complexes [22].

In this research, mordants such as sodium sulfate (Na_2SO_4), aluminum sulfate and potassium dodecahydrate ($KAl(SO_4)_2 \cdot 12H_2O$), and oxalic acid ($C_2H_2O_4$) were used. These compounds are essential for optimizing dye fixation on the fiber and for generating different shades of yellow dye. Previous studies have shown that the combination of metallic mordants with natural dyes improves colorfastness in naturally dyed alpaca fibers [23]. A study by Figueroa (2011) highlights that the use of metallic mordants increases the affinity between the fiber and the dye, resulting in a variety of hues. Color variations during dyeing with natural dyes depend on the dye concentration and the selected mordants. The choice of mordant also influences the L*, a*, and b* coordinates of the final color in the dyed textile [24]. On the other hand, Stanciuc (2020) investigated mordanting and dyeing natural fibers with walnut leaf extract on untreated sheep wool. The best results were achieved at 100 °C for 30 min, using different mordant salts to achieve brown colors with yellow or reddish undertones [25]. Additionally, Canelo and colleagues (2016) analyzed color stability in natural fibers dyed with dyes such as cochineal, purple onion, cocoa, and sunflower, concluding that domestic washing and acid resistance are the most important factors [26]. On the other hand, the study by Jabar et al. (2023) revealed that a catechin-rich dye, extracted from cocoa leaves, can dye wool fabrics uniformly and stably, enhancing their UV resistance and antimicrobial activity without altering the chemical composition of the fibers. This method promotes sustainable practices in the textile industry by utilizing agricultural by-products and reducing dependence on synthetic dyes [27].

Despite the growing interest in textiles dyed with natural dyes, there remains a gap in the scientific literature regarding the detailed evaluation of colorfastness and other properties of alpaca yarns dyed with plant-based dyes. More comprehensive studies are needed to understand the impact of these dyes on colorfastness and textile durability. While synthetic dyes dominate the industry, research on the effect of mordants in dyeing alpaca fibers to achieve various chromatic tones is still limited, and their influence on textile characteristics, dye stability, and color properties remains unknown.

Therefore, the main purpose of this research is to examine the chromatic properties, textile characteristics, and colorfastness in alpaca yarns dyed with *Buddleja coriacea* dye and metallic mordants. This study aims to provide valuable information for the textile industry and advance our understanding of using natural dyes in alpaca fibers, contributing to sustainable dyeing practices.

2. Materials and Methods

2.1. *Buddleja coriacea* Flowers, Alpaca Threads, and Mordants

During the period from April to June 2023, the flowers of *Buddleja coriacea* were collected in an advanced flowering state. The collection process took place in forest ecosystems located in the provinces of Abancay and Andahuaylas, within the Apurímac region of Peru, at an elevation ranging from 3500 to 4200 m above sea level. Simultaneously, white alpaca fibers intended for dyeing experiments were obtained from the community of Iscahuaca, situated in the Cotaruse district of the Aymaraes Province, also within the Apurímac region of Peru, at an altitude of 4100 to 4800 m above sea level. The fiber sample was specifically extracted from the dorsal region of the animal and underwent a process of fiber selection, washing, rinsing, and drying, followed by fiber discarding and traditional manual spinning using a spindle [3,21]. For the post-mordanting process, chemical compounds such as sodium sulfate (Na_2SO_4, commonly known as "ccollpa"), alum (aluminum sulfate and potassium dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$), and oxalic acid ($C_2H_2O_4$, also known as lemon salt) were acquired from a specialized commercial establishment that sells textile yarns, dyes, and mordants, located in the city of Cusco, Peru.

2.2. Extraction of Dye from the Flower of *Buddleja coriácea*

According to Figure 1, The flowers of *Buddleja coriacea* were subjected to a dehydration process in a forced-air oven at a constant temperature of 60 °C for a period of 12 h. Subsequently, for dye extraction, the boiling technique was applied. In this method, 30 g of dried flowers were introduced into 1 L of distilled water contained in a beaker. The system was heated under controlled conditions until it reached the boiling point (93 °C), maintaining these conditions for 30 min. The resulting solution, containing the dye, was filtered using a 100% pure cotton mesh. The filtered dye solution was stored in amber-colored glass containers for preservation and subsequent use.

2.3. Characterization of the Extracted Natural Dye

The extract obtained from *Buddleja coriacea* flowers underwent characterization to determine the chemical composition of the dye. Initially, the pH was measured to classify the dye in terms of its acidity or basicity. Subsequently, Fourier-transform infrared spectroscopy (FTIR) was employed to acquire the spectrum and identify the predominant functional groups in the dye. For this purpose, an FTIR spectrophotometer (Thermo NICOLET iS50, Waltham, MA, USA) was operated within a spectral range of 400 to 4000 cm^{-1}. Each obtained spectrum represents the average of 64 scans.

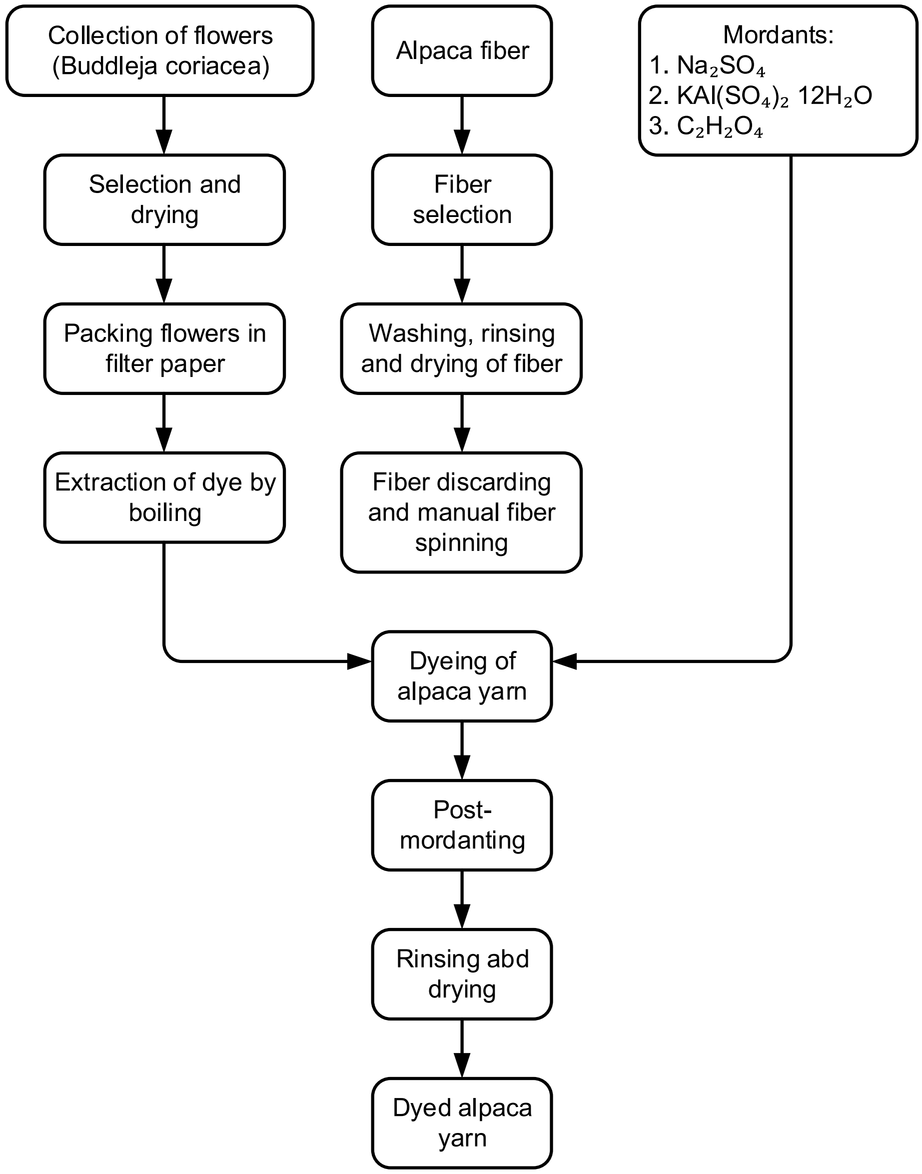


Figure 1. Dyeing process diagram of alpaca fiber with Buddleja coriácea dye.

2.4. Dyeing Process with Post-Etching

According to Figures 1 and 2, the dyeing process of alpaca fibers with *Buddleja coriacea* extract followed a protocol that included a primary dyeing phase followed by post-mordanting, as referenced in the literature [11]. Four beakers were prepared, each containing 0.25 L of aqueous solution and 5 g of pre-spun alpaca fiber. The beakers were exposed to a temperature of 95 °C for 30 min, resulting in fibers exclusively dyed with *Buddleja coriacea* dye (T2). Subsequently, different mordants were applied to the remaining beakers: 3 g of aluminum sulfate and potassium dodecahydrate ($KAl(SO_4)_2{\cdot}12H_2O$) for T3, 2 g of oxalic acid ($C_2H_2O_4$) for T4, and 2 g of sodium sulfate (Na_2SO_4) for T5. These mixtures were boiled for an additional 30 min. After completing this process, the fibers were washed and dried at room temperature, protected from direct light. A white fiber sample was labeled as T1 for reference measurements.

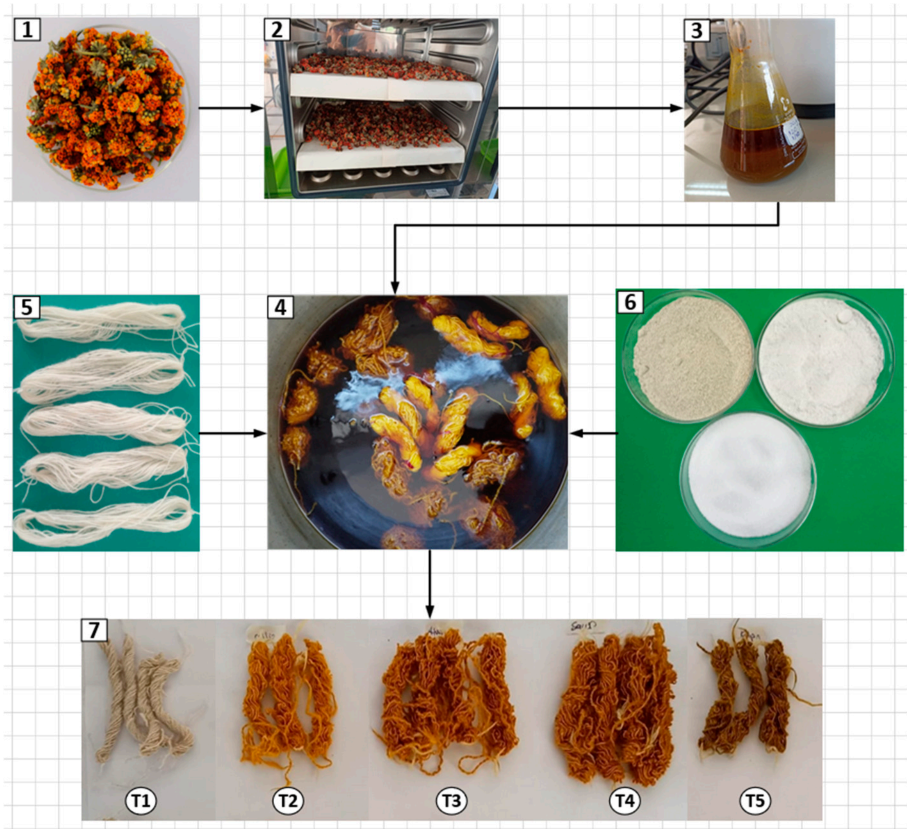


Figure 2. Dyeing procedure for alpaca fiber threads using *Buddleja coriacea* flower extract and mordants. 1, *Buddleja coriacea* tree; 2, *Buddleja coriacea* flowers; 3, drying of flowers in a forced air circulation oven; 4, extraction of natural dye from the flowers; 5, alpaca fiber threads; 6, metallic mordants; 7, alpaca fiber thread dyed with *Buddleja coriacea* flowers and mordants. T1, white fiber; T2, dyed with *Buddleja coriacea*; T3, *Buddleja coriacea* + $KAl(SO_4)_2 \cdot 12H_2O$; T4, *Buddleja coriacea* + C_2H_2O; T5, *Buddleja coriacea* + Na_2SO_4.

2.5. Color Parameter Measurement

The colorimetric parameters of the untreated yarn samples (T1) and those treated with mordants and without mordants (T2, T3, T4, and T5) were quantified using a ULS2048CL-RS-EVO spectrometer (Avantes, Apeldoorn, The Netherlands) coupled to an integrating sphere illuminated by a 50 mm halogen source. The CIELab system parameters (L*, a*, b*, C*, and h°) were applied for chromatic evaluation. L* (luminosity) reflects the brightness (100) or darkness (0) of the sample. The a* values indicate the hue from green (negative) to red (positive). The b* values represent the range from yellow (positive) to blue (negative). Chroma (C*) and hue angle (h°) were also included in the analysis. Additionally, the reflectance spectra R (%) of the samples were determined.

To calculate the chroma (C*), the following mathematical Equation (2) was used [28]:

$$C^* = \sqrt{(a^*)^2 + (b^*)^2}, \tag{1}$$

To determine the hue angle (h°), Equation (2) is applied [28]:

$$h^\circ = \tan(b^*/a^*) \tag{2}$$

2.6. Evaluation of Textile Characteristics

The textile characteristics of the samples were analyzed using an (OFDA 2000, BSC Electronics, Bismarck, ND, USA) instrument in the textile fiber laboratories of the National

University of San Antonio Abad del Cusco. The parameters examined included the average fiber diameter (DMF), comfort factor (FC), and curvature index (IC). Furthermore, the spinning fineness (FH) was approximated by employing the mathematical Formula (3) [29,30].

$$FH = 0.881DMF\,[1 + 5(CV/100)^2]^{0.5} \tag{3}$$

where (CV%) represents the coefficient of variation of fiber diameter.

2.7. Fourier-Transform Infrared Spectroscopy (FTIR) Analysis

The chemical bands of the samples were recorded using an FT-IR spectrometer (Thermo NICOLET iS50, Waltham, MA, USA) operating in the spectral range of 400 to 4000 cm^{-1}. Each spectrum was acquired through 64 scans using the FTIR-ATR technique, following the protocols established in references [31,32]. This analytical procedure was determined to assess the impact of dyeing treatment and mordant application on the structural properties of the fibers.

2.8. Colorfastness Tests

The colorfastness tests were conducted according to ISO 105 C06:2010 [33] and ISO 105 X12:2016 [34] standards at the Textile Industry Laboratories of the National University of Engineering in Lima, Peru. Standardized procedures were applied for colorfastness to washing and rubbing, using a light cabinet with D65 illumination and a grayscale rating from 1 to 5. Colorimetric measurements were taken before and after the colorfastness tests using a spectrophotometer to calculate the color difference. Subsequently, the obtained data were processed using Datacolor Tools vs. 1.1 software to determine the corresponding numerical value on the grayscale.

2.9. Statistical Analysis

To evaluate the colorimetric parameters (L*, a*, b*) and textile properties (DMF, FC, IC, and FH) of the dyed fibers, a statistical analysis was performed using R software, version 4.1.3. Data were expressed as mean ± standard deviation, using a sample size with replicates. Analysis of variance (ANOVA) was applied to compare quantitative variables between treatments. In addition, Tukey's test was used to analyze significant differences, considering a p-value < 0.05 as statistically significant and a p-value > 0.05 as non-significant.

3. Results and Discussion

3.1. FT-IR and UV-Visible Spectral Analysis of the Extracted Dye

In Figure 3, results from infrared spectroscopy (FTIR) are shown, where several significant chemical bands are identified. The band at 3342 cm^{-1} is related to N-H and O-H absorption, while the bands at 2917 cm^{-1} and 2840 cm^{-1} correspond to stretching vibrations of C-H in methylenes and aliphatics, respectively. Additionally, the band at 2157 cm^{-1} is attributed to C≡C bonds in alkynes, the one at 1628 cm^{-1} to stretching vibrations of C=O and C=C, the one at 1324 cm^{-1} to deformation or stretching vibrations of C-N, the one at 1364 cm^{-1} to deformations in CH_3 groups, and the one at 664 cm^{-1} to out-of-plane deformations of C-H in aromatics. Similar results were obtained by Safapour (2023) in a study on sawdust extract from M. laurentii. In that study, relevant chemical bands were identified in the FT-IR spectrum, primarily located at 1622 cm^{-1} (stretching vibrations of the –C=O aromatic bond in flavonoid cores with quinone-like structures) and at 1449 and 1213 cm^{-1} (stretching and bending vibrations of the O–H group in the polyphenolic system). These bands characterize flavonoids, which are responsible for the coloration present in many plants [35,36].

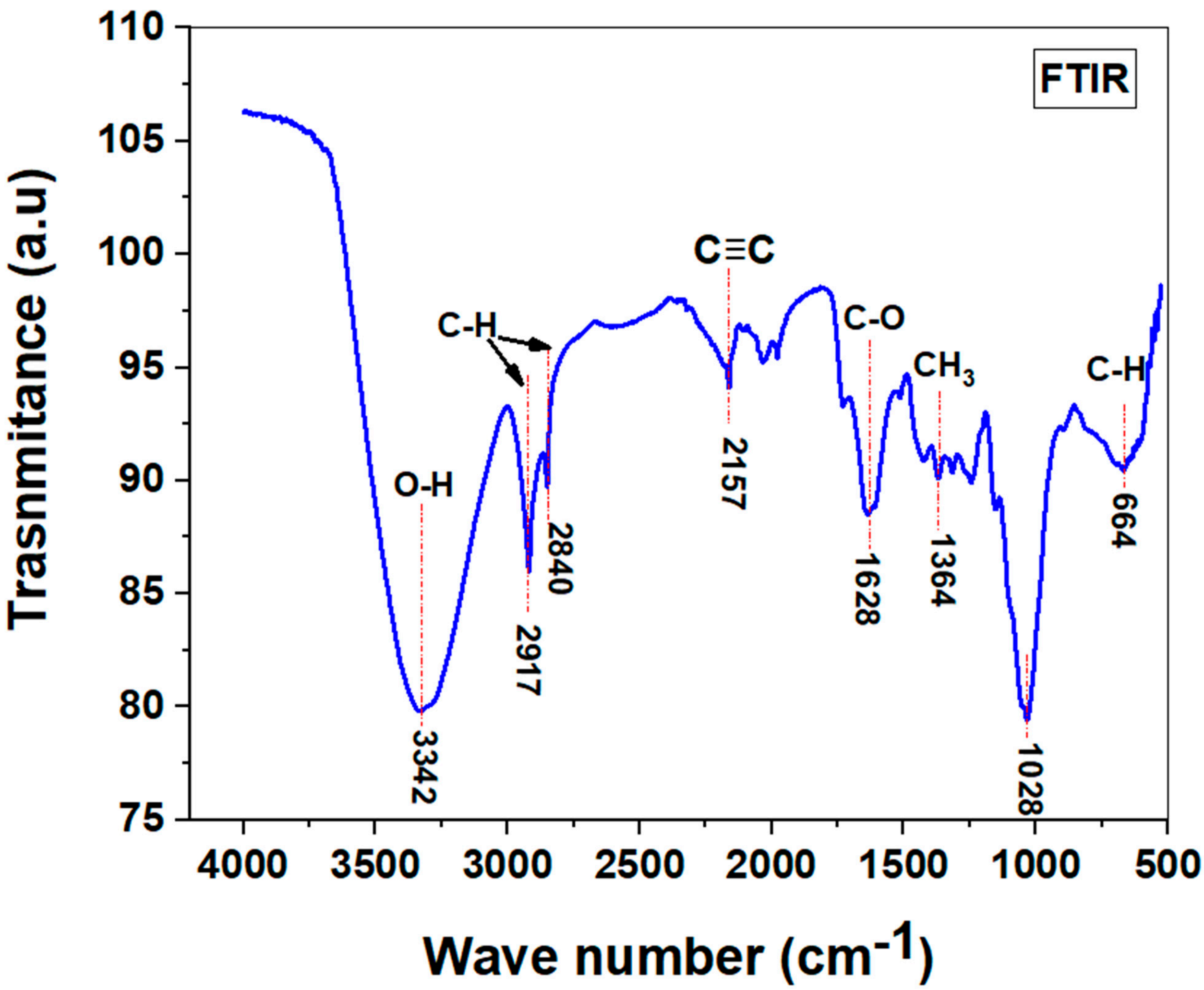


Figure 3. FTIR analysis of *Buddleja coriacea* flower extract.

The band at 3342 cm^{-1} in infrared spectroscopy, attributed to the absorption of O-H groups, is related to the flavonoids responsible for the yellow coloration in Buddleja coriacea. Flavonoids are known for their phenolic structures, and O-H absorption is characteristic of these compounds. Additionally, the band at 1628 cm^{-1}, associated with stretching vibrations of C=O bonds, could also be related to flavonoids, as these bonds are present in the carbonyl structures of flavonoids that contribute to pigmentation [37].

Table 1 displays the results of alpaca fibers dyed with different mordants, highlighting how mordants affect the pH of the dyeing solutions. In Treatment T2, which uses *Buddleja coriacea* without mordants, the average pH is 5.22, indicating a more neutral condition compared to other treatments and potentially resulting in less effective color fixation. On the other hand, Treatment T3, which includes *Buddleja coriacea* and $KAl(SO_4)_2 \cdot 12H_2O$ (alum), shows an average pH of 3.18, suggesting that alum lowers the pH and could enhance color fixation in wool. Treatment T4, with *Buddleja coriacea* and $C_2H_2O_4$ (oxalic acid), records the lowest pH at 2.58, indicating significant acidity that may benefit color fixation and intensity due to oxalic acid. Finally, Treatment T5, combining *Buddleja coriacea* and Na_2SO_4 (sodium sulfate), maintains a pH of 3.18, equal to that of Treatment T3, implying that sodium sulfate has no significant impact on the dyeing solution's pH [37]. Additionally, future research is needed to evaluate different concentrations of the extract, crucial for defining the depth and saturation of color in the dyed fibers.

Table 1. Quantification of pH of *Buddleja coriacea* dye with different mordants.

Sample	Variable	n	Mean ± D.E.
T2	pH	3	5.22 ± 0.11
T3	pH	3	3.18 ± 0.01
T4	pH	3	2.58 ± 0.03
T5	pH	3	3.18 ± 0.01

3.2. Color Property Analysis

The results shown in Table 2 and Figure 4 indicate that tests were conducted with a variety of mordants, including $KAl(SO_4)_2{\cdot}12H_2O$, Na_2SO_4, and $C_2H_2O_4$, to modify the colorimetric attributes of alpaca threads dyed with *Buddleja coriacea* extract. This study focuses on the influence of these mordants on the chromatic characteristics of the fibers. Modifications in color parameters were observed within the CIELab* and CIELCh° color systems. Regarding Lightness (L)*, it is noteworthy that Sample T1, corresponding to white fiber, exhibits the highest clarity. Treatment T2 (Buddleja coriacea) achieved a Lightness (L*) value of 68.98, indicating a lighter color compared to samples treated with mordants. In contrast, post-mordant treatments with T3 ($KAl(SO_4)_2{\cdot}12H_2O$) and T4 ($C_2H_2O_4$) resulted in decreased Lightness (L*) values of 61.84 and 60.58, respectively, indicating darker tones. Similarly, Treatment T5 (Na_2SO_4) showed the lowest Lightness (L*) value at 50.14, significantly darker. For Chromaticity (a* and b*), positive values of a* in T2, T3, and T4 indicate a tendency toward red, while negative values in T1 and T5 suggest a tendency toward green. As for b*, positive values in all treatments indicate a tendency toward yellow. Treatment T2 exhibits the highest Chromaticity, possibly related to the colorant concentration and its interaction with mordants. Regarding Color Saturation (C)*, values range from 25.92 to 40.59. The lowest saturation (25.92) is observed in T5 (Na_2SO_4), representing a moderately saturated color (less vibrant). Conversely, the highest saturation (40.59) corresponds to Treatment T2 (Buddleja coriacea), resulting in a more vivid and saturated color with greater intensity. Consequently, post-mordant application during the dyeing process affects color brightness, resulting in less saturated colors. Regarding hue angle (h°), values vary among treatments. T2 (Buddleja coriacea) and T4 ($C_2H_2O_4$) exhibit the lowest values, indicating hues closer to red-yellow. T1 (white fiber) and T5 (Na_2SO_4) have higher values, suggesting hues closer to yellow-green. Similar results were reported by Montazar et al. (2007) in their study on wool dyeing with marigold, where wool threads were pretreated with alum as a colorless mordant. Subsequently, the threads were dyed with marigold and treated with different ammonia solutions, resulting in decreased L* values for all ammonia-treated samples, while a* and b* values varied based on the ammonia solution used [38]. Color strength varies with mordant treatments. T5 (Na_2SO_4) showed the highest K/S (5.6), followed by T4 (C_2H_2O, 5.3), T2 (Buddleja coriacea, 4.6), and T3 ($KAl(SO_4)_2{\cdot}12H_2O$, 4.3). These changes reflect the intensification of color in the dyed fibers.

The statistical analysis of chromatic parameters for treatments (T2, T3, T4, and T5) yields highly reliable results, with p-values < 0.05 for color indices L*, a*, b*, C*, and h°. This suggests that the color alterations remain consistent and are directly linked to the treatments applied, ruling out the chance of being mere random fluctuations.

Table 2. Quantification of color on alpaca fiber threads dyed with *Buddleja coriacea* flower extract and color tone modifiers.

Treatment	L*	a*	b*	C*	h°	K/S
T1	80.36 ± 4.83^{d}	-0.86 ± 0.44^{a}	09.14 ± 3.38^{a}	09.19 ± 3.80^{a}	95.39 ± 2.12^{c}	---
T2	68.98 ± 7.20^{c}	2.59 ± 0.43^{c}	41.39 ± 7.76^{c}	41.48 ± 7.74^{c}	86.29 ± 0.95^{a}	4.6
T3	61.84 ± 8.04^{b}	1.81 ± 0.44^{b}	40.54 ± 8.52^{c}	40.59 ± 8.49^{c}	87.22 ± 1.28^{a}	4.3
T4	60.58 ± 5.22^{b}	2.67 ± 1.11^{c}	38.94 ± 6.28^{c}	39.04 ± 6.32^{c}	86.63 ± 1.83^{a}	5.3
T5	50.14 ± 4.71^{a}	-0.52 ± 0.49^{a}	25.91 ± 4.85^{b}	25.92 ± 4.86^{b}	91.07 ± 0.79^{b}	5.6

Average ± SD (n = 3). T1, white fiber; T2, dyed with *Buddleja coriacea*; T3, *Buddleja coriacea* + $KAl(SO_4)_2 \cdot 12H_2O$; T4, *Buddleja coriacea* + $C_2H_2O_4$; T5, *Buddleja coriacea* + Na_2SO_4. Superscripts with different letters per column are statistically significant ($p < 0.05$).

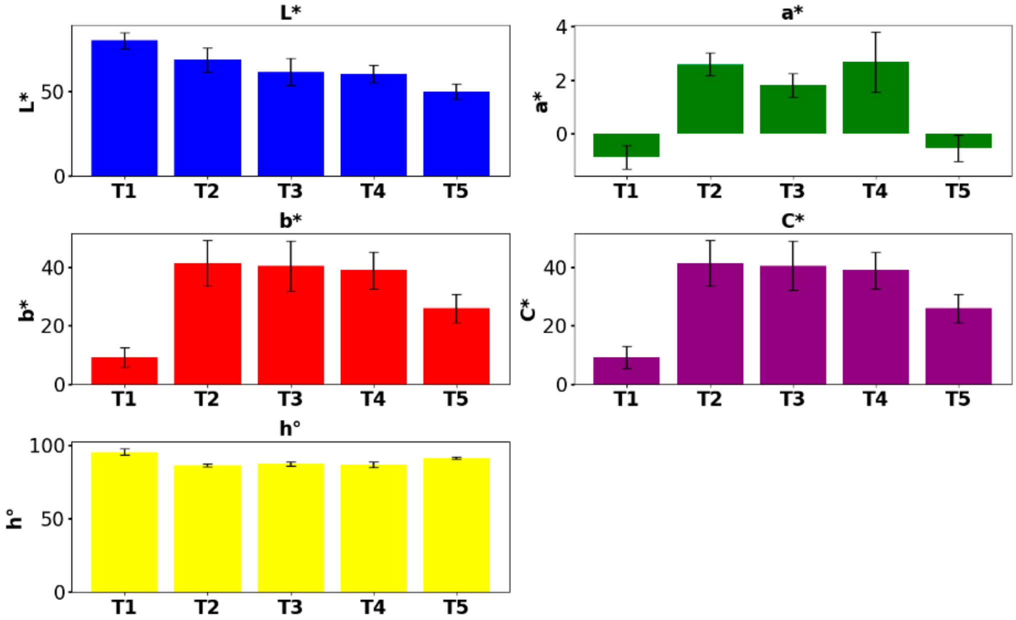


Figure 4. Colorimetry parameters after treatment.

The incorporation of various mordants with *Buddleja coriacea* extract leads to noticeable changes in the coloration of alpaca fibers, affecting saturation and hue. This study is essential for the proper selection of mordants that yield specific shades and for understanding how post-mordanting processes impact fiber characteristics. These findings are directly relevant to the textile industry, particularly sustainable fashion, where natural dyes and environmentally friendly dyeing methods are prioritized. Furthermore, these advancements promote the development of innovative dyeing techniques that enhance color quality and durability in organic textiles. Understanding the impact of mordants on alpaca fiber coloration is crucial for textile manufacturing, and the inclusion of metallic mordants such as $KAl(SO_4)_2 \cdot 12H_2O$, $C_2H_2O_4$, and Na_2SO_4 can significantly expand the range of colors and hues in the resulting textiles, underscoring the importance of meticulous mordant selection for achieving desired chromatic effects.

3.3. Reflectance Spectrum R (%) According to Figure 1

According Figure 5 the incorporation of mordants such as $KAl(SO_4)_2 \cdot 12H_2O$, Na_2SO_4, and $C_2H_2O_4$ in the dyeing process of alpaca fibers with *Buddleja coriacea* extract leads to a decrease in sample reflectance, following the sequence R% (Na_2SO_4) < R% ($KAl(SO_4)_2 \cdot 12H_2O$) < R% ($C_2H_2O_4$) < R% (Buddleja coriacea). Additionally, these mordants alter fiber absorbance and reduce light resistance. The use of mordants modifies the optical properties of alpaca fibers, affecting both absorption and reflectance, which in turn impacts their lightfastness. During post-mordanting, mordant molecules bind to both the dye and the fiber, influencing reflectance. Previous research, including the study conducted by Montazer et al. (2007), confirms that treatments like ammonia dyeing affect reflectance spectra. These findings are crucial for the textile industry, as mordants influence not only color intensity but also the interaction of light with dyed threads, facilitating the creation of chromatically diverse textiles [38]. It is recommended that future investigations explore how different mordants affect reflectance spectra and colorimetric parameters, as well as other factors that may impact light absorption in dyed alpaca threads, such as mordant concentration or dyeing process duration, in order to optimize dyeing processes more effectively.

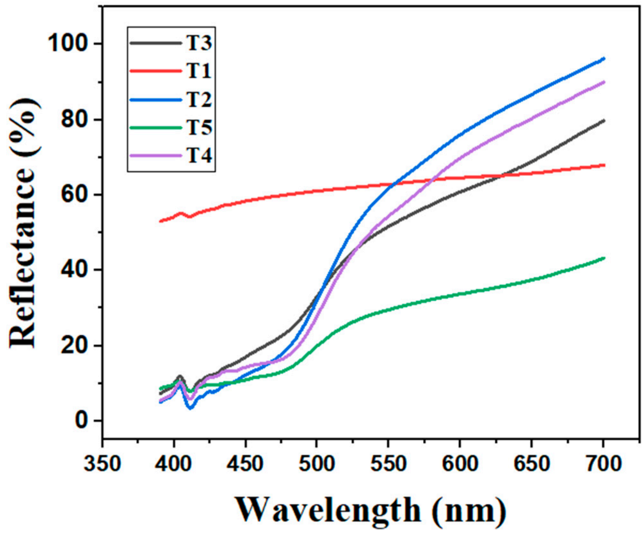


Figure 5. Reflectance spectrum R (%) of alpaca fiber dyed with *Buddleja coriacea* flower extract and mordants.

3.4. Textile Characteristics

According to the data presented in Table 3, and Figure 6 an analysis was conducted on the textile properties of alpaca yarns dyed with *Buddleja coriacea* dye and subsequently treated with various mordants ($KAl(SO_4)_2 \cdot 12H_2O$, Na_2SO_4, and $C_2H_2O_4$). Using white fiber (T1) as a reference, which had the lowest average fiber diameter (DMF) of 21.93, it was observed that treatment T2 (Buddleja coriacea) resulted in a DMF of 22.06. On the other hand, dyeing followed by post-mordanting (T3, T4, and T5) yielded MFD values of 22.87, 22.79, and 22.90, respectively. These slight variations in MFD are attributed to the diffusion of mordant and dye within the fiber. Regarding comfort factor (CF), samples T2 (Buddleja coriacea), T4 ($C_2H_2O_4$), and the white reference sample (T1) exhibited the highest CF values, with 94.40, 94.84, and 94.26, respectively. This indicates greater softness and reduced skin irritability compared to samples T5 and T3. With the addition of mordants T3 ($KAl(SO_4)_2 \cdot 12H_2O$) and T4 ($C_2H_2O_4$), CF values decreased slightly to 93.30 and 92.97, respectively. The results show that yarn fineness (FH) remains relatively constant across the samples, with values ranging from 21.39 µm to 22.30 µm. Dyeing with *Buddleja coriacea* does not significantly affect yarn fineness, as the dyed sample (T2) exhibits fineness similar to that of the white fiber (T1). The mordants used ($KAl(SO_4)_2 \cdot 12H_2O$, $C_2H_2O_4$, and Na_2SO_4) have a slight effect on increasing yarn fineness, but not significantly. In contrast, dyeing with *Buddleja coriacea* dye (T2) increased the curvature index (CI) to 62.24, suggesting more pronounced curvature. However, the addition of mordants $KAl(SO_4)_2 \cdot 12H_2O$ (T3) and $C_2H_2O_4$ (T4) reduced the values to 59.51 and 58.72, respectively, indicating a return to moderate curvature. The mordant Na_2SO_4 had minimal effect on the curvature of alpaca fiber yarns. Therefore, the changes in textile characteristics are mainly due to the diffusion of the dye and mordants through the pores inside the alpaca fibers, affecting the fiber diameter, comfort factor, yarn fineness, and curvature.

Table 3. Textile characteristics in yarns of Alpaca fiber dyed with *Buddleja coriacea* flower extract and mordants.

Treatment	MFD (µm)	CF (%)	CI (°/mm)	FH (µm)
T1	21.93 ± 0.30 [a]	94.40 ± 0.97 [a]	52.83 ± 2.77 [a]	21.42 ± 0.38 [a]
T2	22.06 ± 0.42 [a]	94.84 ± 0.66 [a]	65.24 ± 6.29 [c]	21.39 ± 0.39 [a]
T3	22.87 ± 0.56 [a]	93.30 ± 0.88 [a]	59.51 ± 2.73 [b]	22.10 ± 0.41 [a]
T4	22.79 ± 1.26 [a]	94.26 ± 2.70 [a]	58.72 ± 4.25 [b]	22.30 ± 1.32 [a]
T5	22.90 ± 1.24 [a]	92.97 ± 1.92 [a]	62.39 ± 3.44 [bc]	22.15 ± 1.03 [a]

Average ± SD (n = 3). T1, white fiber; T2, dyed with Buddleja coriacea; T3, *Buddleja coriacea* + $KAl(SO_4)_2 \cdot 12H_2O$; T4, *Buddleja coriacea* + $C_2H_2O_4$; T5, *Buddleja coriacea* + Na_2SO_4. MFD, mean fiber diameter; CF, comfort factor or fibers < 30 µm; CI, curvature index; FH, yarn fineness. Superscripts with different letters per column indicate statistically significant differences ($p < 0.05$).

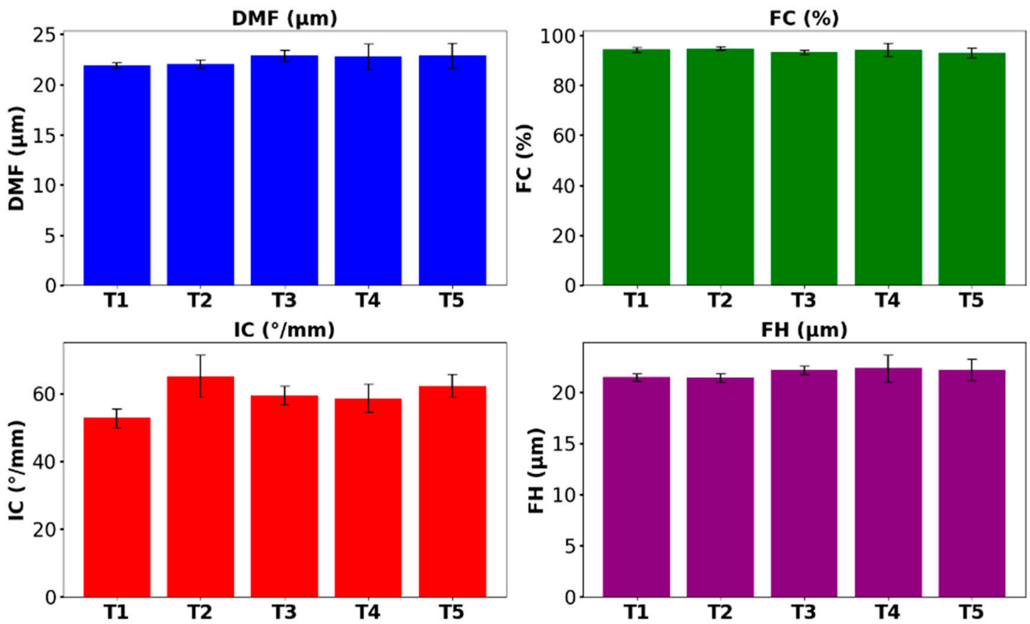


Figure 6. Change in colorimetry parameters due to treatment.

The statistical analysis applied to the textile properties of dyed alpaca fibers revealed statistical significance in the curvature index (CI) with a p-value < 0.05. However, for parameters such as average fiber diameter (MFD), comfort factor (CF), and yarn fineness (FH), no statistically significant differences were detected ($p > 0.05$). These findings align with previous studies, such as the work of Lozano et al. (2024), which determined that the application of mordants in dyeing alpaca fibers with tankar stem extract did not result in significant variations in textile characteristics [9]. Therefore, it can be concluded that the different treatments did not exert a considerable impact on the textile properties of alpaca fibers, supporting the feasibility of using natural dyes and metallic mordants in the dyeing process. However, further evaluation of these parameters in future research is recommended to enhance our understanding of the effects of dyeing on the textile qualities of alpaca fibers.

3.5. FTIR Spectroscopy

Figure 7 illustrates that the untreated white fiber sample (T1) and the sample treated with potassium aluminum sulfate dodecahydrate ($KAl(SO_4)_2 \cdot 12H_2O$), commonly known as alum (T3), exhibit higher spectral intensity in Fourier-transform infrared spectroscopy

(FTIR) compared to samples T2, T4, and T5, which show lower intensity. This indicates that the mordants involved in the dyeing process alter the chemical composition of alpaca fibers.

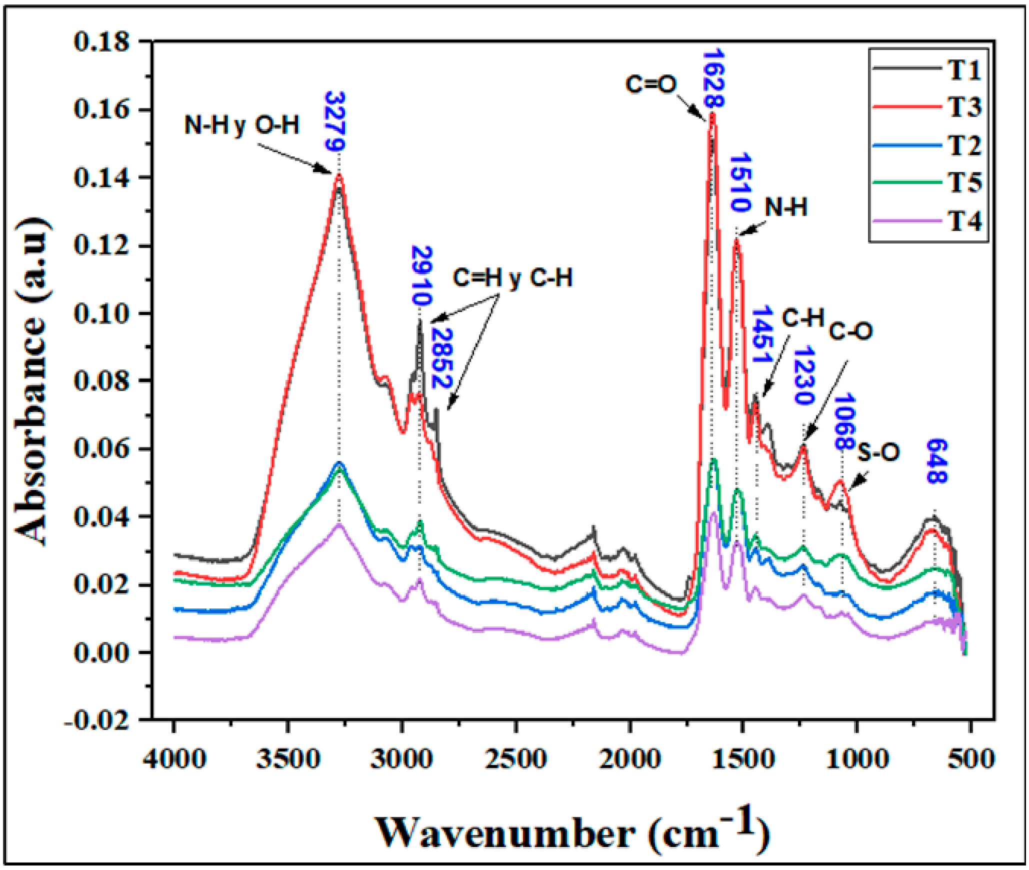


Figure 7. FTIR analysis of alpaca fiber dyed with *Buddleja coriacea* flower extract and mordants.

Alpaca fibers, being inherently proteinaceous, contain amide groups A (I, II, and III). The Amide A band, located in the 3100–3500 cm^{-1} range of the FTIR spectrum, is associated with vibrations of N-H and O-H bonds. In the region of 2850–3000 cm^{-1}, stretching vibrations of C=H and C-H bonds are detected. The Amide I band, between 1600 and 1700 cm^{-1}, corresponds to the stretching vibration of the carbonyl group (C=O) in the peptide bond. The Amide II band, around 1500–1480 cm^{-1}, relates to vibrations of N-H and C-N bonds, characteristic of protein structure. The Amide III band, in the 1361–1470 cm^{-1} interval, is primarily linked to vibrations of the C-N bond and is sensitive to differences in amino acid side-chain conformations and protein secondary structure. Additionally, the region of 1210–1290 cm^{-1} corresponds to vibrations of the C-O bond present in proteins. Alterations in the intensity of these bands in the FTIR spectrum result from the post-mordanting process, reflecting changes in the optical properties of alpaca fibers [39]. FTIR analysis provides a detailed view of the chemical composition of fibers and how it is affected by dyeing and the mordants used. Research such as that by Lozano et al. (2024) has reported similar findings, observing that both the dye and mordants influence the intensity of FTIR spectra in alpaca fibers dyed with natural extracts [9].

The results obtained can be attributed to the interaction of infrared radiation with the molecular structure of the fiber, leading to variations in the intensity of Fourier-transform infrared spectroscopy (FTIR) spectra. The presence of dye and mordant molecules adhered to the fiber has the potential to alter its molecular conformation, thus affecting the intensity of spectral bands in FTIR spectra. Therefore, the observed changes in band intensity serve as indicators of transformations in the chemical composition of alpaca fiber. Future research could use X-ray diffraction and Raman spectroscopy to determine the chemical functionality of alpaca fibers, which is essential for understanding the mordant dyeing process.

3.6. Mechanism of Dye Interaction with Fibers and Metallic Mordants

3.6.1. Dye–Fiber Interaction

According to the FTIR spectroscopic analysis shown in Figure 2, *Buddleja coriacea* dye molecules contain functional groups such as hydroxyls and carbonyls. These groups can form hydrogen and ionic bonds with the proteins in alpaca fiber, mainly keratin. These chemical interactions are essential for ensuring the durable fixation of the dye in the fiber. Additionally, future research could use LCMS profiling (High-Resolution Liquid Chromatography coupled with Mass Spectrometry) to obtain a more exhaustive analysis of the available dye molecules. It is recommended to perform an LCMS profile to gather detailed information about the available dye molecules.

3.6.2. Effect of Mordanting and Mechanism of Interaction with Fiber and Dye

Mordants interact with the functional groups of the dye, such as hydroxyls and carbonyls, modifying their chemical structure. This interaction can change how the dye absorbs and reflects light, resulting in variations in tone and hues. Mordants like potassium aluminum sulfate ($KAl(SO_4)_2 \cdot 12H_2O$) form metal complexes with the dye molecules, altering the electronic and optical properties of the dye and creating different colors. Oxalic acid ($C_2H_2O_4$), being a chelating agent, can form complexes with metal ions present in the fiber or dye, thus modifying the resulting color.

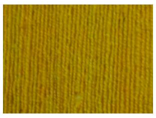
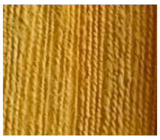
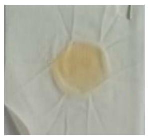
These findings have important implications in the field of natural textiles due to their ecological and sustainable focus. However, this study presents some limitations. There is a need for more research and additional testing using different mordanting techniques, such as pre-mordanting and meta-mordanting, and evaluating the toxicity of metallic mordants in the environment. Future research should address these limitations to gain a more comprehensive understanding of the dyeing process. It is crucial to explore biodegradable mordants, such as citric acid, due to their lower toxicity and potential to reduce heavy metals and contaminants.

3.7. Colorfastness Properties of Dyed Alpaca Yarns

Table 4 presents the colorfastness properties of alpaca yarns dyed with *Buddleja coriacea* extract and treated with mordants. Sample T2 (Buddleja coriacea), dyed solely with the extract, and sample T4, combined with oxalic acid ($C_2H_2O_4$), achieved a rating of 4/5, denoting excellent colorfastness. In contrast, dry rubbing fastness received a rating of 3/5, considered suitable for wet washing. Post-mordanted samples T3 ($KAl(SO_4)_2 \cdot 12H_2O$) and T5 ($C_2H_2O_4$) were rated 3/5, indicating acceptable color resistance, albeit with some fading. Wet washing resulted in a score of 2/5, suggesting limited colorfastness.

It can be inferred that *Buddleja coriacea* extract acts as a substantive dye, capable of adhering to the fiber without the need for mordants to enhance its washfastness. However, the addition of mordants during post-mordanting appears to weaken the bonds, decreasing wash resistance. Previous research, such as that by Sucasaca (2020), reports that *Buddleja coriacea* extract can produce a range of yellow shades in alpaca fiber with colorfastness ratings of 3 to 5 on the grayscale for color change [25]. These findings are relevant for selecting materials or products that require specific colorfastness under various usage conditions. However, it is important to note that these conclusions are based on the data from this particular study and may vary under different circumstances. Therefore, further studies and comprehensive testing are recommended for a more accurate assessment of the rubbing fastness of these samples. Additionally, future research is necessary to develop and validate lightfastness tests, evaluating the resistance of samples and highlighting their implications in natural textiles. Research could also focus on analyzing the surface properties of alpaca fiber, such as its texture and structure, to better understand how the dye adheres to and distributes within the fiber.

Table 4. Fastness tests on alpaca fiber yarns dyed with *Buddleja coriacea* flower extract and mordants.

Treatment	Dry Cleaning	Wet Wash	Dye Transfers			
			Sample	Cotton Witness	Dry Rub	Wet Rub
T2	4/5	3/5				
T3	3/5	2/5				
T4	4/5	3/5				
T5	3/5	3/5				

T2, water content of Buddleja coriácea; T3, water content of Buddleja coriácea + $KAl(SO_4)_2 \cdot 12H_2O$; T4, water content of Buddleja coriácea + $C_2H_2O_4$; T5, water content of Buddleja coriácea + Na_2SO_4.

4. Conclusions

This research focused on coloring alpaca fibers using a natural dye derived from *Buddleja coriacea* and metallic mordants. The selected mordants, including sodium sulfate (Na_2SO_4), potassium aluminum sulfate dodecahydrate ($KAl(SO_4)_2 \cdot 12H_2O$), and oxalic acid ($C_2H_2O_4$), significantly influenced the luminance and chromatic saturation of the dyed fibers. After the post-mordanting process, a decrease in luminance and brightness was observed, resulting in softer tones and a shift in hue angle toward warmer colors. Minimal variations were detected in the average fiber diameter (MFD), comfort factor (CF), and curvature index (CI), suggesting a moderate impact of dyeing and mordant application on the textile properties of alpaca fiber. Fourier-transform infrared spectroscopy (FTIR) revealed alterations in the intensity of post-mordanted fiber spectra. Colorfastness to washing, both dry and wet, depended on the mordant used, with superior quality (4/5) without mordants and decreased quality (3/5 and 2/5) when mordants were applied, affecting color permanence and textile quality. Statistical analysis showed a significant correlation (with $p < 0.05$) between the colorimetric parameters L*, a*, b*, C*, and h°. The use of different mordants allowed for three distinct shades from the original yellow of *Buddleja coriacea extract*. These findings endorse the use of metallic mordants and natural dyes in dyeing processes, highlighting their sustainability as renewable resources. In summary, the significance of Buddleja coriaceas natural dye and the role of mordants in creating diverse hues during alpaca fiber dyeing are emphasized.

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A.Q.-Q.; project administration, A.Q.-Q. All authors have read and agreed to the published version of the manuscript.

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