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Textile Characteristics, Medullation, and Colorimetry of Wool Fiber Dyed with *Dactylopius coccus* Using Atmospheric Pressure Plasma Jet (APPJ)

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Abstract: The industrial and artisanal textile industries necessitate the adoption of sustainable dyeing practices. Although the natural dye derived from *Dactylopius coccus* presents a viable option, its traditional application requires metallic mordants that pose environmental and health risks. This study investigates the utilization of atmospheric-pressure plasma jet (APPJ) technology for dyeing wool with *Dactylopius coccus* dye, with the objective of optimizing the process and minimizing its environmental impact. The APPJ technique was employed for wool dyed with *Dactylopius coccus* dye, and the textile properties, medullation, and colorimetry were evaluated using an optical fiber diameter analyzer (OFDA) and a spectrometer with an integrating sphere. The results demonstrated that the APPJ enhanced the color intensity and uniformity, facilitating improved dye penetration into the fibers. Plasma treatment darkened the fiber, generated reddish and yellowish tones, and increased the color saturation and intensity. The wool samples treated with plasma exhibited an increase in DMF and SF but a decrease in IC and greater size variability. The APPJ reduces total medullation in wool dyed with cochineal dye. In conclusion, the APPJ was demonstrated to be a promising method for dyeing wool with *Dactylopius coccus* dye, offering an effective and sustainable alternative to traditional methods, with enhanced color vibrancy and uniformity and reduced resource utilization.

Keywords: textile characteristics; colorimetry; *Dactylopius coccus*; wool fiber; atmospheric-pressure plasma jet (APPJ)

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1. Introduction

In the context of the artisanal textile industry in Peru, sheep wool constitutes a fundamental raw material, particularly in regions such as Cusco, Ayacucho, and Puno. This ancestral practice holds significant cultural value and drives the local economy through the production of meat and fiber. Wool, which comprises a complex protein known as keratin, is valued for its softness, thermal-insulation properties, and durability [1]. Traditionally, wool has been dyed with natural colorants, among which the dye obtained from *Dactylopius coccus*, commonly known as cochineal, is prominent. Its primary component, carminic acid, provides an intense red color [2]. However, the natural dyeing process presents challenges because of the low affinity between the fiber and dye, necessitating the use of metallic mordants to enhance adherence [3]. Various mordants such as alum ($KAl(SO_4)_2 \cdot 12H_2O$) are essential for fixing natural dyes. These mordants can alter the intensity and shade of the color; for instance, iron sulfate produces darker tones, whereas sodium carbonate produces brighter tones [4,5]. Although effective, these mordants have adverse impacts on the environment and human health [6,7]. In this context, it is necessary to identify sustainable alternatives to mitigate these impacts.

The atmospheric-pressure plasma jet (APPJ) technique presents a viable alternative to conventional methods in the textile industry, circumventing the utilization of mordants and chemical dyes that generate toxic waste. The application of an APPJ in textile dyeing is feasible because of its capacity to enhance dye absorption in the fibers, resulting in more intense and uniform colorization. This sustainable method significantly reduces the consumption of chemicals and water, thereby minimizing environmental impact. Moreover, the APPJ facilitates enhanced dye penetration and color durability, offering an environmentally benign and efficient alternative to traditional dyeing methods [8]. The APPJ modifies the properties of fibers and *Dactylopius coccus* dye without compromising material integrity, improving both color intensity and uniformity in wool [9]. This technique is characterized by its dye-penetration capability, color durability, and sustainability. Furthermore, the APPJ has emerged as an innovative solution, yielding outcomes comparable to those of traditional mordants, but without their associated toxicity [10]. This study investigated the influence of APPJ application during the dyeing of wool with *Dactylopius coccus* dye by evaluating its effects on the medullation properties, textile characteristics, and colorimetric parameters. The significance of this research lies in its potential to implement more sustainable practices in the artisanal textile industry, aligning with global trends towards sustainability and environmental responsibility [9,10].

In the field of wool dyeing research, the utilization of the atmospheric-pressure plasma jet (APPJ) represents an emerging and promising technique. Numerous studies have elucidated the benefits and mechanisms of this technology. Sajed et al. [11] demonstrated that plasma treatment and polypropylene imine dendrimers significantly enhanced the dyeing capacity of wool with *Dactylopius coccus* dye, increasing the dye's affinity for the fiber. Conversely, Quispe-Quispe et al. [12] found that the discoloration of *Dactylopius coccus* dye was caused by reactive species generated by the APPJ through oxidative degradation. Similarly, Petkevičiūtė et al. [13] investigated the impact of nonthermal plasma on the surface morphology, color characteristics, and electrical conductivity of dyed wool and observed significant changes in these properties. Additional studies, such as those conducted by H. Barani and Calvimontes [14], analyzed the effects of oxygen plasma treatment on wool fibers. Their results indicated that this treatment increased the surface roughness, modified the morphology and chemical structure of the fibers, increased the crystallinity, and improved the dye absorption capacity. Cristina Barnils [15] has also contributed to this field, investigating the impact of plasma treatment on wool and polyamide 6 fibers. Her findings demonstrate improvements in the shrinkage resistance of wool and alterations in the microstructure and dyeing properties of polyamide 6, suggesting significant potential for enhancing the functional characteristics of treated textiles.

The fundamental textile characteristics of wool, including the average fiber diameter (DMF), comfort factor (FC), and spinning fineness (FH), are critical determinants of its quality. A lower DMF content results in finer fibers and softer fabrics, which are highly valued in the market. A high FC indicates greater softness and reduced skin irritation, which are desirable attributes of garments in direct contact with the skin [3]. A high FH enables the production of fine, lightweight, and durable yarns that are essential for high-quality products [16]. These characteristics significantly influence the comfort and durability of textiles, thereby emphasizing their importance in the textile industry. According to a study conducted by Quispe-Peña et al. [17], these properties are crucial for defining the quality and ultimate application of wool products.

Regarding colorimetry parameters such as lightness (L*), chromaticity (a*, b*), chroma (C*), and color strength (K/S), research has demonstrated that these factors significantly influence the quality of wool fiber [18–20]. These parameters are fundamental in determining the visual appearance of the fabric and its subsequent market acceptance. Barani and Calvimontes [14] observed that oxygen plasma treatment can enhance these parameters, resulting in improved dye absorption and increased colorfastness.

Medullation properties significantly influence the quality of natural fibers. Vicuña fibers are characterized by their fineness and softness, owing to the absence of medullation. In contrast, sheep wool and alpaca fibers exhibit variations in medullation, which affects their application and value in the textile industry [21]. Various studies have indicated that the medullation properties of wool fibers, such as the proportion of fibers with medulla (TMED), number of fibers without medulla (NMED), presence of fibers with fragmented medulla (FMED), length and distribution of interrupted medulla (DMED), presence of intact medulla (CMED), and identification of fibers with prominent medulla (SMED), significantly impact wool fiber quality [22]. These characteristics influence the softness, strength, and appearance of the fabric, which are essential for determining its suitability for different textile applications [23]. For instance, Cruz et al. [24] evaluated the incidence and relationship between medullation types and fineness in sheep, alpaca, llama, paco-vicuña, and angora rabbit fibers, emphasizing the importance of reducing the incidence of medullation to enhance the quality of textile processing. The research by Vujasinović et al. [25] reported a bio-innovative pre-treatment of coarse wool fibers, highlighting the presence of medullation in these fibers and emphasizing the importance of utilizing the entire fleece in the context of sustainability. The use of enzymatic complexes and percarbonate bleaching results in clean, high-quality wool fibers, minimizing the environmental impact and improving energy efficiency. This approach is crucial for sustainable textile production, enabling the maximum utilization of available resources and reducing waste.

Regarding the controversial and divergent hypotheses, several studies have elucidated the advantages of plasma treatment in dyeing wool with *Dactylopius coccus* dye. For instance, Sajed et al. [11] demonstrated that plasma combined with polypropylene imine dendrimers significantly enhanced dyeing capacity, suggesting that an APPJ could surpass conventional methods. Peran et al. [26] indicated that oxygen plasma pre-treatment enhanced the dyeing capability of wool with natural extracts such as pomegranate peel. This treatment improved the dye absorption, thereby enhancing the antimicrobial and color properties of the wool fabric.

Researchers have demonstrated the potential of an APPJ to modify medullation properties, including total medullation in fibers (TMED) and non-medullated samples (NMED), as well as to enhance lightness (L*), chromaticity (a* and b*), chroma (C*), and color strength (K/S) [27,28]. Barani and Calvimontes [14] observed that oxygen plasma treatment alters the morphology and chemical structure of fibers, increasing their roughness and crystallinity, which may optimize dye absorption without the necessity for metallic mordants. However, certain experts have questioned the consistent replicability of these improvements in an industrial context. Furthermore, Barnils et al. [15] reported enhancements in shrinkage resistance and alterations in the microstructure and dyeing properties of plasma-treated wool. Nonetheless, other researchers emphasize the importance of evaluating the cost-effectiveness and long-term viability of APPJs [17]. These diverse reports underscore the necessity for further research to conclusively determine the efficacy and benefits of APPJs in comparison to traditional wool dyeing methods.

In this context, it is imperative to seek sustainable alternatives in the textile industry to mitigate the environmental impacts and promote responsible practices. The atmospheric-pressure plasma jet (APPJ) has emerged as an innovative technique that offers results comparable to those of traditional mordants, but without their associated toxicity [29]. This investigation focuses on analyzing the impact of dyeing wool with *Dactylopius coccus* dye, utilizing the APPJ, on the medullation properties, textile characteristics, and colorimetry parameters in yarns dyed with and without plasma treatment.

This research aims to evaluate the feasibility of employing an atmospheric-pressure plasma jet (APPJ) as a sustainable method in the textile and artisanal industries. Experiments were conducted to examine the efficacy of the APPJ in dyeing wool using the *Dactylopius coccus* dye. The findings help us to determine if the APPJ is a viable and sustainable alternative, contributing to more environmentally responsible dyeing techniques while preserving artisanal tradition. Colorimetric parameters, textile characteristics, color fastness, and medullation properties were assessed.

2. Materials and Methods

2.1. Materials

The spun white wool, pre-treated and in the form of a cone, was purchased from the Michell Alpaca store in the Department of Cusco, Peru. The dye utilized was derived from *Dactylopius coccus*, commonly referred to as cochineal, an insect that parasitizes the prickly pear cactus (*Opuntia ficus*). *Dactylopius coccus* specimens were collected from the community of Huayllabamba, situated in the province of Abancay, Apurímac region, Peru, at an altitude of 2500 m above sea level. After collection, *Dactylopius coccus* underwent a drying process and was subsequently ground into a fine powder for use as a natural dye. The mordant employed in the dyeing process, specifically alum ($KAl(SO_4)_2 \cdot 12H_2O$), was obtained from a local market in Abancay, Apurímac department, Peru. This method was compared with traditional dyeing techniques that utilize mordants to evaluate differences in the physical properties of wool fibers.

2.2. Preparation of Dactylopius coccus Dye and Wool Fibers

The wool yarns were divided into four samples of 2 g each; one sample was designated as white wool (LB) and utilized as a reference. The dye solution was prepared by dissolving 800 mg of cochineal powder in 1000 mL of distilled water and homogenizing the mixture via thermal treatment at 95 °C for 2 h. Prior to the dyeing process, the wool yarns were cleaned and conditioned. This process involves washing the yarns with a mild detergent solution to remove impurities and natural oils. Subsequently, the fibers were rinsed with distilled water and dried at room temperature to ensure thorough cleaning and preparation for dyeing [19–30].

2.3. Treatment via APPJ Discharge

The dyeing of fibers with cochineal dye utilizing plasma discharge was conducted using a PLASMA CLEAN-PL-5050 apparatus from Keylink Technology Co. (Wenzhou, China). The plasma equipment was operated at an electrical voltage of 140 V and airflow of 105 psi. Dyeing experiments were performed on 150 mL dye samples, with 15 min of exposure to the plasma discharge. The distance between the plasma outlet and the surface of the liquid solution containing wool fibers was maintained at 0.3 cm.

2.4. Dyeing Wool Fibers Using APPJ Discharge

One hundred and fifty milliliters of *Dactylopius coccus* dye extract and three pre-prepared fragments of white wool were introduced into a beaker. The container was positioned on a magnetic stirrer SCI-S10 Analog Multi-Position Magnetic apparatus from Scilogex (Rocky Hill, CT, USA) to ensure the uniform mixing of the solution. The nozzle of the PLASMA CLEAN-PL-5050 equipment from Keylink Technology Co. (Xiamen, China) was situated 5 cm from the liquid–air interface to ensure that the flow of the reactive species (RONS) reached the dye/fiber solution. The APPJ plasma discharge was applied for 15 min. The wool fragments were extracted from the container at 5, 10, and 15 min and subsequently washed. The resulting samples were designated LP5 (plasma-dyed fiber for 5 min), LP10 (plasma-dyed fiber for 10 min), and LP15 (plasma-dyed fiber for 15 min).

2.5. Dyeing Wool Fibers with Cochineal Dye Using Alum Mordant

The methodology described by Romero [31] was implemented for the dyeing process using *Dactylopius coccus* (cochineal) dye. A white wool specimen was initially placed in a beaker containing 150 mL of the previously prepared dye solution. To ensure a homogeneous mixture, a magnetic stirrer was used at 95 °C. Upon reaching boiling point, 1 g of alum was introduced as a mordant. The mixture was maintained at boiling temperature for an additional 15 min. Subsequently, the wool specimen was extracted, rinsed with deionized water, and designated LM0. This dyeing process, incorporating alum, was conducted to facilitate comparative analysis. The multiparameter meter PL-700AL is fabricated by GOnDO Electronic, Taipei City, Taiwan.

2.6. Evaluation of pH

A multiparameter meter PL-700AL (GOnDO Electronic, Taiwan) was used to record the changes in the pH of the dye solution throughout the dyeing process. The samples were measured at intervals of 5, 10, and 15 min during plasma treatment. To assess the acidity or alkalinity of the dye, pH measurements were conducted for both plasma-treated and untreated samples.

2.7. Evaluation of Colorimetry Parameters of Wool Samples

The CIELab system (L*, a*, b*, c*) and K/S parameters were used to evaluate the colorimetric changes in the wool yarns dyed under varying conditions. The measured color parameters encompassed lightness (L*), which indicates the degree of lightness (100) or darkness (0) of the sample; a* values, which range from negative (green) to positive (red); and b* values, which range from positive (yellow) to negative (blue). Additionally, the C* values, representing the chroma and hue angle, were measured. To quantify these properties, a ULS2048CL-RS-EVO spectrometer (AVANTES, Apeldoorn, The Netherlands) coupled with an integrating sphere with 50 mm halogen illumination and a 10° observation angle with D65 illuminants was used. This apparatus also facilitated the acquisition of the reflectance spectra, R (%), of the samples. The color intensity parameter (K/S) was calculated using the Kubelka–Munk Equation (1) [32,33].

$$\frac{K}{S} = \frac{(1-R)^2}{2R}, \tag{1}$$

The chroma (C*) was determined using Equation (2) [34,35].

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

The hue angle (h°) was determined using Equation (3).

$$h^\circ = \tan(b^*/a^*), \quad (3)$$

2.8. Evaluation of Textile Characteristics

The textile characteristics of the specimens were analyzed utilizing an optical fiber diameter analyzer (OFDA 2000 equipment, Meltzer International, Lima, Perú) at the textile fiber laboratories of the National University of San Antonio Abad del Cusco. Mean fiber diameter (MFD), comfort factor (CF), and curvature index (CI) were measured using the IWTO-62 standard [36]. Furthermore, spinning fineness (SF) was calculated employing a specific mathematical Equation (4) [37].

$$\mathrm{SF} = 0.881 \times DMF\sqrt{1 + 5\left(\frac{CV}{100}\right)^2} \quad (4)$$

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

2.9. Measurement of Medullation Properties

The medullation characteristics of wool samples dyed with plasma and mordants were evaluated utilizing an optical fiber diameter analyzer (OFDA). The proportion of medullated fibers (TMED), quantity of non-medullated fibers (NMED), presence of fragmented medullated fibers (FMED), length and distribution of interrupted medulla (DMED), presence of intact medulla (CMED), and identification of fibers with prominent medulla (SMED) were assessed [38]. An OFDA 2000 instrument (Meltzer International, Lima, Perú) was employed in the top mode to measure the wool fibers, and the analysis was conducted in accordance with the IWTO-62 standard.

2.10. Evaluation of Color Fastness to Washing

The assessment of colorfastness to washing and its transfer in samples LP5, LP10, LP15, and LM0 was conducted in accordance with the ISO 105 C06:2010 [39] and ISO 105 X12:2016 [40] ISO 105 C06:2010 Test: A1S standard. The methodology involved cutting yarns and the multifiber DW test fabric to dimensions of 4 cm × 10 cm, which were subsequently sewn together along their shorter sides to form the test specimen. A solution containing 4 g/L detergent without optical brightening agents was prepared, and 150 mL of this solution was introduced into the equipment container. The composite sample was then placed in a launderometer with the temperature set to 40 °C for 30 min. The sample was extracted and rinsed twice with distilled water (100 mL). Following drying, the evaluation was performed in a light booth using D65 illumination, employing grayscale to assess the transfer and color change. The results were validated using a spectrophotometer, comparing the samples before and after the test to determine the color difference, and the corresponding grayscale value was calculated utilizing the Datacolor Tools vs. 1.1 software.

2.11. Statistical Analysis

Statistical analysis was conducted to evaluate the colorimetric parameters (L*, a*, b*) and textile properties (MFD, CF, CI, and SF) of the dyed fibers. The analysis was performed using R software version 4.1.3. The data are presented as the mean ± standard deviation, with a sample size of three repetitions (n = 3). One-way analysis of variance (ANOVA) was employed to compare the quantitative variables between two different treatments. Furthermore, Tukey's test was utilized to identify significant differences between means. Pearson's correlation coefficient was applied to examine the relationships between the various parameters, with p-values < 0.05 considered statistically significant and p-values

> 0.05 considered not significant. A correlation heatmap was generated to visualize the relationships between the textile characteristics, colorimetric parameters, and medullation properties of the treated samples.

3. Results

3.1. Variations in Coloration of Wool Fiber With and Without Plasma Treatment

The experimental results (Table 1 and Figure 1) demonstrate that the treatment of wool fibers with plasma and mordants induces significant modifications in the colorimetric properties of these fibers. The white fiber (LB) exhibited a lightness (L*) of 89.96. Upon exposure to plasma, a progressive decrease in lightness was observed, reaching values of 60.17 to 52.10 after 15 min of plasma exposure. The addition of mordants intensified this effect, further reducing the lightness to 46.32. Regarding the a* component, the plasma-treated fibers displayed a tendency towards reddish tones, with values ranging from 5.74 to 22.91 depending on the exposure time. This pattern was also evident in the fibers dyed with the mordants. In relation to the b* component, yellow tones were observed in the samples plasma-treated for up to 10 min, as evidenced by their negative values. However, after extending the exposure time to 15 min, the values became positive, indicating blue tones. The mordanted fibers had a value of −2.54, suggesting the presence of yellow tones. The highest chroma (C*) was recorded for the LP15 sample, indicating a higher color saturation compared to the other treatments. Samples LP5 and LP10 presented lower chromas, indicative of lower saturation. The results indicated that the white fiber (LB) had a hue angle of 98.35°. Plasma dyeing significantly altered the tone to 323.13° (LP5), 350.73° (LP10), and 34.72° (LP15). Mordant dyeing (LM0) exhibited an angle of 346.36°, shifting towards blue–violet tones. The LM0 samples demonstrated intermediate color saturation. Regarding the color intensity K/S, a gradual increase was observed as the plasma exposure time was prolonged, which was associated with the alteration of the dye concentration in the fiber due to the degradation caused by the APPJ plasma discharge. In contrast, the fibers dyed with mordants exhibited a higher color intensity K/S (4.18), which was attributed to the formation of a thin layer on the fiber surface.

Table 1. Color parameters obtained from wool samples dyed with and without plasma treatment.

Treatments	L*	a*	b*	C*	h°	K/S
LB	89.96 ± 0.29	−2.13 ± 0.09	14.26 ± 0.46	14.39 ± 0.43	98.35	0
LP5	60.17 ± 0.80	5.74 ± 0.26	−4.30 ± 0.56	7.18 ± 0.54	323.13	1.05
LP10	53.19 ± 0.88	5.94 ± 0.22	−0.97 ± 0.39	6.03 ± 0.19	350.73	1.82
LP15	52.10 ± 0.82	22.91 ± 1.18	15.81 ± 0.45	27.88 ± 1.17	34.72	2.20
LM0	46.32 ± 1.26	10.60 ± 0.32	−2.54 ± 0.59	12.21 ± 0.55	346.36	4.18

White fiber (LB), plasma dyed for 5 min (LP5), plasma dyed for 10 min (LP10), plasma dyed for 15 min (LP15), and mordant dyed for 0 min (LM0).

According to the correlation heatmap depicted in Figure 2, a strong negative correlation (−0.84) exists between the parameters L* and K/S, indicating an inverse relationship between the L* value (lightness) and K/S value (absorption and scattering coefficient). This observation suggests that the specimens with higher lightness exhibited lower dye absorption. A strong positive correlation (0.72) was observed between a* and C*, signifying that an increase in the a* value (red–green component) corresponds to an increase in the C* value (chroma). This demonstrates that specimens with higher red color intensity also exhibit higher color saturation. The correlation between b* and a* is weakly positive (0.23), implying a slight tendency for the a* value to increase as the b* value (yellow–blue component) increases, although this trend is not pronounced. A strong positive correlation of 0.81 between C* and b* indicates a significant relationship, suggesting that as the C* value (chroma or color saturation) increases, the b* value (yellow–blue component in the color space) also increases substantially. For L* and a*, a moderate negative correlation (−0.70) is observed, indicating an inverse relationship between these parameters. This suggests that the specimens with higher lightness tend to exhibit a lower red component.

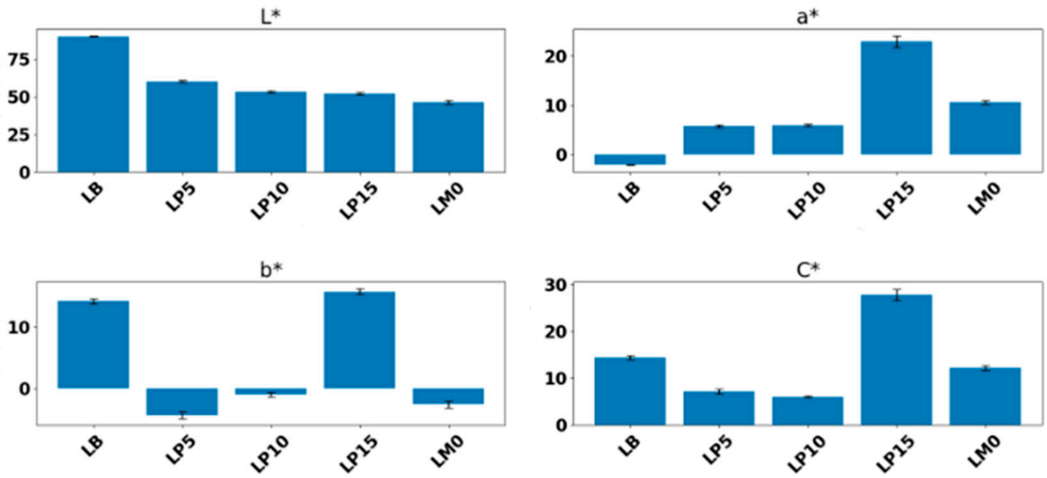

Figure 1. Bar chart of the colorimetric parameters of samples treated with and without plasma.

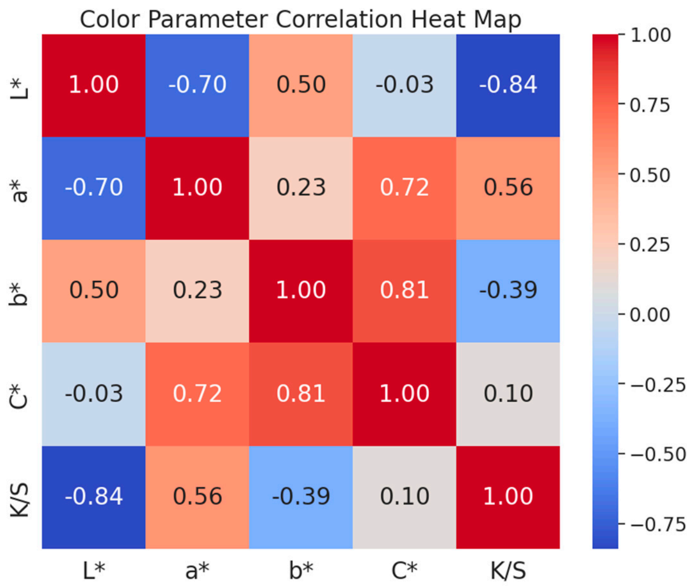

Figure 2. Heatmap correlating colorimetry parameters.

3.2. Comparison of Textile Characteristics in Samples with and Without APPJ Treatment

Table 2 and Figure 3 show the results of colorimetry parameters for samples subjected to plasma treatments (LP5, LP10, LP15) in comparison to the white fiber (LB) and mordant-dyed fiber (LM0). The mean fiber diameter (MFD) exhibits an increase correlated with the duration of plasma treatment, ranging from 26.33 ± 0.60 μm in LP5 to 28.20 ± 0.61 μm in LP15. This increase, potentially attributable to the diffusion of dye and its electrostatic adherence, demonstrates greater efficacy compared to the treatment with mordants (LM0, 24.94 ± 0.75 μm) and the white fiber (LB, 24.12 ± 0.55 μm). The coefficient of variation (CV%) also increases with treatment, indicating a greater variability in fiber size. Regarding the comfort factor (CF), a slight decrease is observed with plasma exposure time, which could indicate a negative effect of these treatments. The curvature index (CI) decreases with increased plasma exposure time, suggesting that the fibers become straighter. Finally, the spinning fineness (SF) increases with plasma exposure time, which is related to the increase in MFD and suggests that the treatments are promoting fiber height growth.

Table 2. Textile characteristics of wool fiber yarns dyed with plasma and without plasma.

Treatments	DMF (μm)	FC (%)	IC (°/mm)	SF (μm)
LM0	24.94 ± 0.75 [ab]	76.13 ± 2.06 [ab]	67.77 ± 6.62 [ab]	25.18 ± 1.54 [ab]
LB	24.12 ± 0.55 [a]	79.83 ± 1.67 [b]	61.33 ± 3.31 [a]	23.85 ± 0.50 [a]
LP5	26.33 ± 0.60 [c]	75.20 ± 3.93 [ab]	74.30 ± 10.56 [b]	26.38 ± 1.03 [b]
LP10	26.12 ± 0.91 [bc]	72.00 ± 4.89 [a]	71.60 ± 3.30 [ab]	26.09 ± 0.72 [b]
LP15	28.20 ± 0.61 [d]	76.77 ± 0.46 [ab]	74.60 ± 6.91 [b]	28.61 ± 0.31 [c]

Mean fiber diameter (DMF); comfort factor (FC); curvature index (IC); spinning fineness (SF).

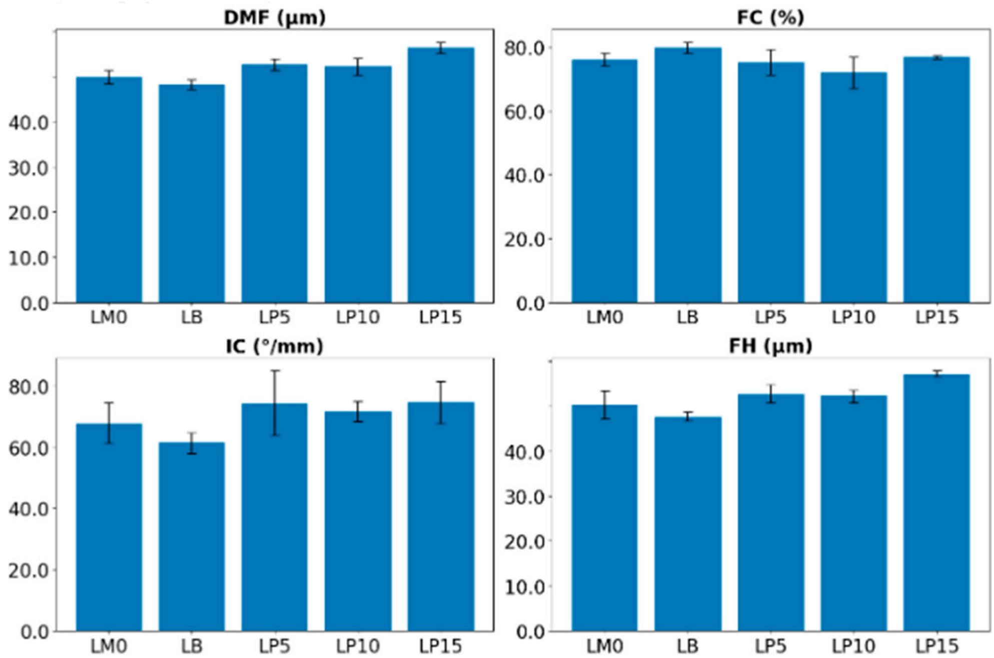

Figure 3. Bar chart of textile characteristics of samples treated with plasma and without plasma.

An analysis of variance (ANOVA) was conducted to evaluate the textile properties of the samples LM0, LB, LP5, LP10, and LP15, considering the characteristics DMF, CV, FC, IC, and SF. The results indicated that only the curvature index (IC) presented statistically significant variations among the different treatments, with $p \leq 0.05$. The variables DMF, CV, FC, and SF did not show significant changes, with $p > 0.05$. In conclusion, the curvature index (IC) was the only characteristic that reflected a notable difference due to the applied treatments, while the other properties maintained statistical consistency without marked differences.

According to the correlation heatmap depicted in Figure 4, the results demonstrate several significant correlations among the textile characteristics of wool fibers. The perfect correlation (1.00) between the mean fiber diameter (DMF) and spinning fineness (SF) indicates a direct relationship, suggesting that an increase DMF corresponds to a higher SF, which is favorable for producing fine yarns. Additionally, a strong positive correlation (0.89) between DMF and the curvature index (IC) implies that as DMF increases, so does the curvature of the fibers, influencing their handling and comfort in textile applications. On the other hand, the moderate negative correlation (−0.37) between DMF and comfort factor

(FC) suggests that fibers of greater diameter may be less comfortable, which is relevant for applications where comfort is crucial. Similarly, a moderate negative correlation (−0.35) between FC and SF indicates that more comfortable fibers tend to have lower spinning fineness, emphasizing the necessity to balance comfort and quality in the produced yarns. A moderate to strong negative correlation (−0.64) between FC and IC suggests that fibers with greater curvature tend to be less comfortable, which is significant for optimizing treatments that improve both comfort and functionality of textiles. Finally, the strong positive correlation (0.88) between IC and SF implies that a higher curvature index is associated with better spinning fineness, benefiting the production of high-quality yarns. These results highlight the importance of understanding the interactions among different textile characteristics to optimize the production and quality of final products.

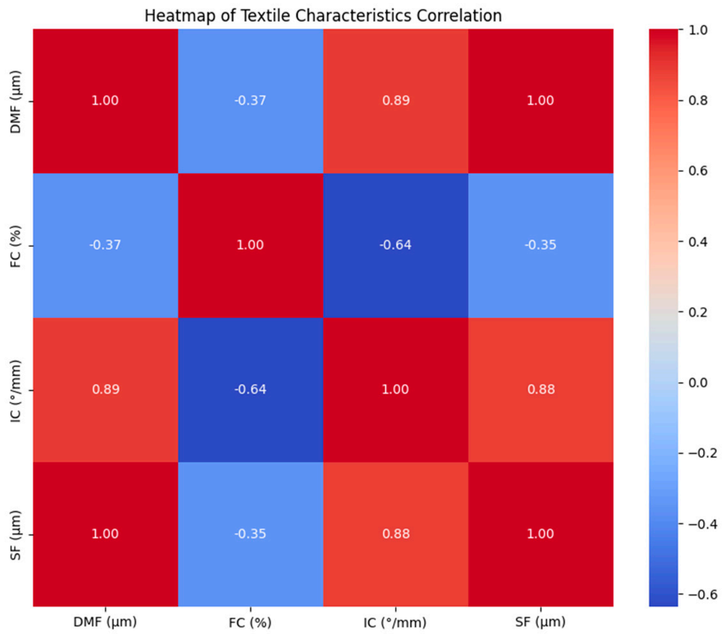

Figure 4. Heatmap correlating textile characteristics.

3.3. Comparative Study of Medullation in Fibers with and Without APPJ Treatment

According to the results presented in Table 3, and Figure 5 the DMF exhibited significant differences. Regarding TMED, the LM0, LB, and LP5 groups demonstrated a higher percentage of total medullation compared to the LP10 and LP15 groups, which exhibited lower percentages. The samples subjected to prolonged plasma discharge (LP10 and LP15) displayed a lower percentage of total medullation in the fibers. Generally, medullation is considered an undesirable characteristic as it devalues the wool. For NMED, the LM0 group samples had the lowest percentage of non-medullated wool, while the LP15 group had the highest percentage of non-medullated wool. The percentage of non-medullated wool increased with the plasma discharge treatment time. No significant differences were observed between the LB and LP5 groups. In FMED, the LP15 group presented the highest percentage of fragmented medulla, while the LM0 group had the lowest percentage. The percentage of wool with fragmented medulla was directly proportional to the increase in plasma discharge time. No significant differences were observed between the LB and LP5 groups. For DMED, significant differences were observed among the five sample groups, indicating that the treatment does not influence the variation in the percentage of fibers with discontinuous medulla. In CMED, the LM0 group had the lowest percentage of continuous medulla, while the LP15 group presented the highest percentage. The continuous medulla in sheep wool also increased with the plasma discharge treatment time. No significant differences were observed between the LB and LP5 groups. Finally, in SMED, the variations shown by the data were less clear compared to the previous columns. The LP5 group had the lowest percentage, and the LP10 group had the highest percentage. No significant differences were observed between LB and LP15 groups.

Table 3. Mean fiber diameter ± SD in wool fibers dyed with cochineal and evaluation of the percentage of medullation by medulla type.

ID	DMF (µm)	TMED (%)	NMED (%)	FMED (%)	DMED (%)	CMED (%)	SMED (%)
LM0	24.94 ± 0.75 ^{ab}	99.13 ± 0.23 ^{bc}	0.87 ± 0.23 ^{a}	0.17 ± 0.11 ^{a}	0.04 ± 0.06 ^{a}	0.59 ± 0.27 ^{a}	0.09 ± 0.12 ^{ab}
LB	24.12 ± 0.55 ^{a}	98.59 ± 0.18 ^{bc}	1.41 ± 0.18 ^{ab}	0.51 ± 0.03 ^{bcd}	0.01 ± 0.00 ^{a}	0.69 ± 0.06 ^{ab}	0.22 ± 0.08 ^{abc}
LP5	26.33 ± 0.60 ^{c}	98.49 ± 0.64 ^{bc}	1.51 ± 0.70 ^{ab}	0.52 ± 0.22 ^{bcd}	0.19 ± 0.12 ^{a}	0.76 ± 0.39 ^{ab}	0.05 ± 0.07 ^{a}
LP10	26.12 ± 0.91 ^{bc}	97.34 ± 1.09 ^{a}	2.64 ± 1.04 ^{cd}	0.71 ± 0.18 ^{de}	0.11 ± 0.06 ^{a}	1.28 ± 0.57 ^{bc}	0.53 ± 0.34 ^{c}
LP15	28.20 ± 0.61 ^{d}	97.31 ± 0.16 ^{a}	2.69 ± 0.16 ^{d}	0.83 ± 0.05 ^{e}	0.13 ± 0.18 ^{a}	1.48 ± 0.02 ^{c}	0.26 ± 0.01 ^{abc}

Identification of samples (ID), mean fiber diameter (DMF), total medullation in fibers (TMED), non-medullated wool (NMED), wool with fragmented medulla (FMED), fibers with discontinuous medulla (DMED), wool with continuous medulla (CMED), strongly medullated wool (SMED), and superscripts with different letters per column indicate significant differences ($p < 0.05$).

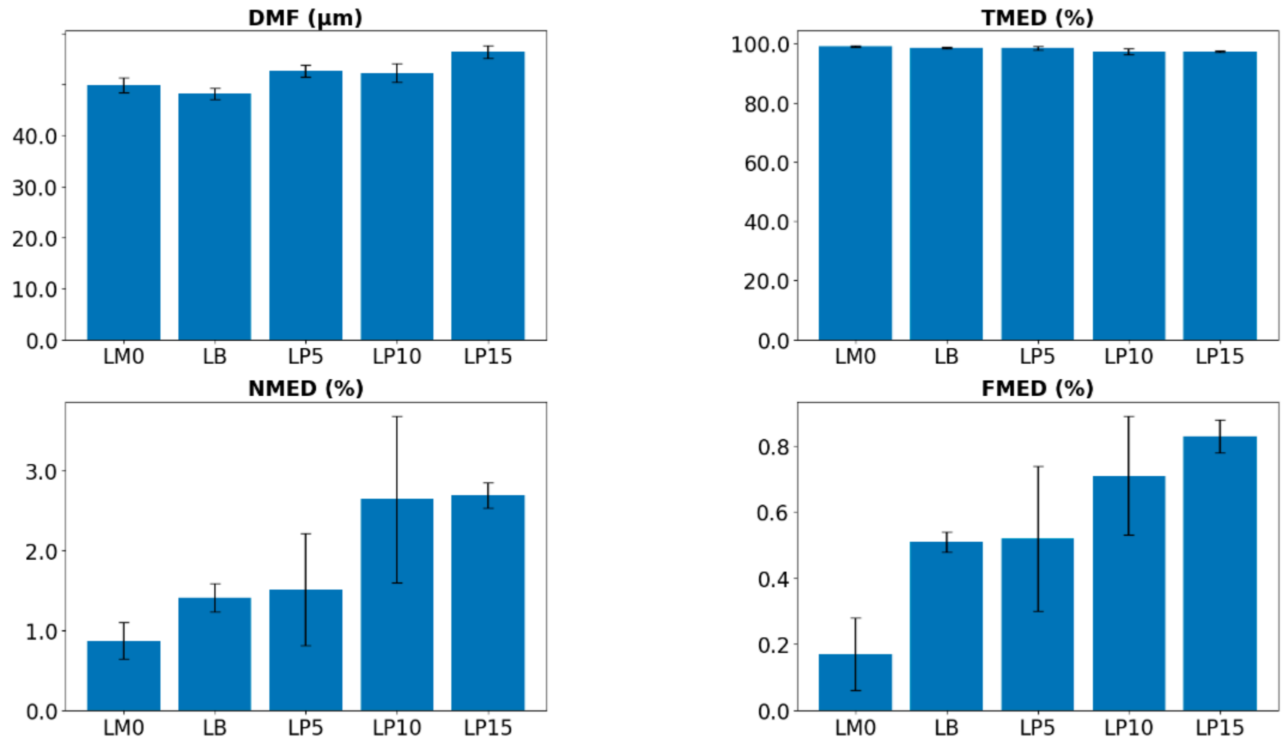

Figure 5. Bar chart of medullation properties of samples treated with and without plasma.

Statistical analysis of the textile characteristics of the samples (LM0, LB, LP5, LP10, and LP15) was conducted utilizing analysis of variance (ANOVA) for each textile characteristic (TMED, NMED, FMED, DMED, CMED, and SMED). The analysis revealed no statistically significant differences between the parameters ($p > 0.05$); however, significant differences were observed in DMF.

The results are shown in Figure 6, in which a heatmap correlates the various variables measured in the wool fiber yarns. The mean fiber diameter (DMF) had a strong positive correlation with the percentage of fibers with a fragmented medulla (FMED) (0.87), fibers with a continuous medulla (CMED) (0.87), and strongly medullated fibers (SMED) (0.4). This suggests that as DMF increases, the proportions of the mentioned characteristics also increase. However, DMF shows a strong negative correlation with the total medullation percentage (TMED) (−0.87), indicating that a larger mean diameter is associated with lower total medullation. TMED also showed a strong negative correlation with FMED (−0.94), discontinuous medulla (DMED) (−0.43), and CMED (−0.97), suggesting that higher total medullation reduces the proportion of fibers with fragmented, discontinuous, and continuous medulla. In contrast, FMED had a strong positive correlation with DMED (0.47), CMED (0.9), and SMED (0.61), implying that a higher proportion of fibers with a fragmented medulla also increases those with discontinuous, continuous, and strongly medullated fibers. DMED has strong positive correlations with CMED (0.42) and SMED (−0.055), indicating that a higher percentage of fibers with discontinuous medulla is associated with an increase in continuous and strongly medullated fibers. Finally, CMED had a strong positive correlation with SMED (0.68), suggesting that a higher proportion of fibers with a continuous medulla also increased the proportion of strongly medullated fibers.

Figure 6. Heatmap correlating medullation parameters of treated and untreated samples.

3.4. Effects of APPJ Plasma Treatment on pH During the Dyeing Process

The results (Figure 7) reveal an exponential decrease in the pH of the dye/fiber solution with exposure time to reactive oxygen and nitrogen species (RONS) produced by atmospheric-pressure plasma jet (APPJ) discharge. A significant reduction in pH was observed in the first two minutes, with the pH variation stabilizing from 2 to 10 min. The pH ranges from 4.84 to 1.97, indicative of the conversion to an acidic dye.

APPJ plasma treatment significantly influenced pH. The interaction with RONS exponentially decreases the pH, which is attributable to the generation of free radicals and hydrogen peroxide (H_2O_2) through the chemical reactions of the treatment [41] A notable change in electrical conductivity was also observed, related to the formation of radicals, such as ozone (O3), hydrogen peroxide (H_2O_2), nitrite (NO_2^-), and nitrate (NO_3^-) ions, which promote the creation of acidic dyes and alter the electrical conductivity of the dye solution. These results demonstrate that APPJ plasma treatment is an effective method for modifying the chemical and physical properties of dye solutions during dyeing. However, further studies are needed to evaluate the feasibility and efficiency of APPJ plasma treatment on a larger scale and to understand the exact mechanisms of the chemical reactions induced by the treatment.

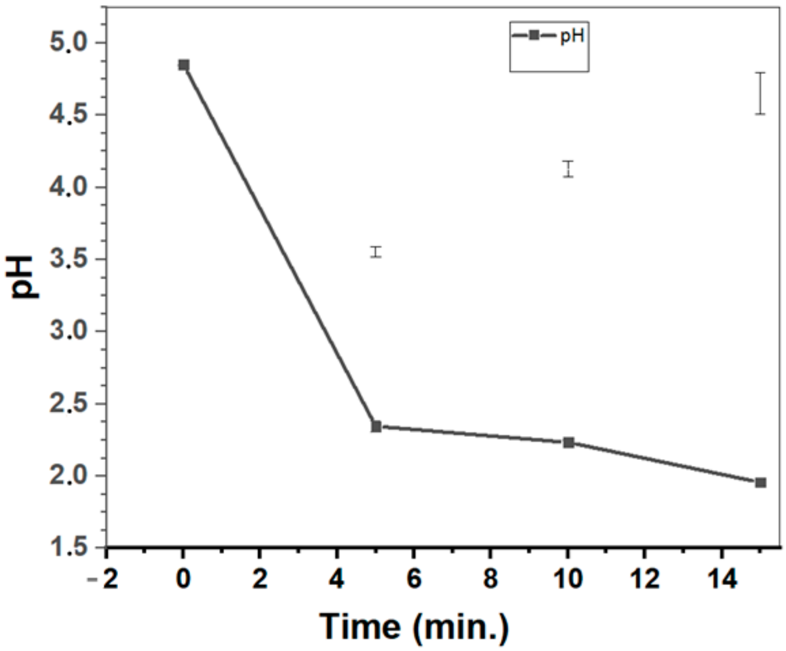

Figure 7. Changes in pH due to APPJ discharge exposure.

3.5. Analysis of Color Fastness Properties to Washing of Fibers

The results show Table 4 that the LP5, LP10, LP15, and LM0 samples exhibit high wash resistance, as evidenced by an almost imperceptible color change, with values of 1–2 on the gray scale. The evaluation of color transfer revealed that these samples have a minimal tendency to bleed onto other materials or surfaces during use or washing, achieving ratings of 4–5 and 5. Consequently, it was determined that the LP5, LP10, LP15, and LM0 samples were wash-resistant and had a low probability of color transfer. These findings are consistent with those reported by Haji et al. [42], who observed that the color fastness of natural fibers dyed with plasma treatment improved significantly owing to the effects of such treatment. It has been found that the use of plasma in textiles such as cotton intensifies the color and strengthens dye fastness properties. Plasma treatment increased the affinity of the fibers for the dye and optimized its fixation. In addition, plasma treatment has demonstrated potential in terms of uniform coloration and improved color fastness. This is crucial for the durability and quality of textile products, suggesting that the samples retain their original coloration after multiple washes and do not transfer color to other objects or garments during use. However, it is essential to consider the study's limitations, such as the omission of other color fastness factors, such as light exposure, and the restriction to only four treatments. Therefore, additional research is recommended to examine different aspects of color fastness, evaluate various natural extracts under different dyeing conditions, and study the impact of temperature and dye concentration on the color fastness of the fibers.

Table 4. Fastness test on wool fiber dyed with APPJ plasma discharge.

Treatments	Dry Cleaning	Wet Cleaning
LP5	5	5
LP10	5	5
LP15	5	5
LM0	5	5

4. Discussion

4.1. Discussion on Colorimetric Results: Comparison Between Treated and Untreated Samples

The results of this study indicate that plasma treatments and mordants cause significant changes in colorimetric parameters. The lightness (L) of the fibers decreases significantly with plasma treatment, from 89.96 ± 0.29 in the white fiber (LB) to 46.32 ± 1.26 in the mordanted sample (LM0), 60.17 ± 0.80 in the plasma-treated sample (LP5), and 52.10 ± 0.82 with plasma treatment (LP15), indicating a notable darkening. The a* parameter increases, highlighting reddish tones in the LP15 sample (22.91 ± 1.18). Conversely, the b* value shifts towards blue tones in LP10 (−0.97 ± 0.39) and towards yellow tones in LP15 (15.81 ± 0.45), reflecting significant chromatic variations induced by the plasma treatment. Furthermore, there is higher color saturation (C*) in LP15 and an increase in color intensity (K/S) with plasma exposure time, with greater intensity observed when mordants are utilized in the dyeing process.

The results of this study are consistent with those of previous research, such as that conducted by Sánchez et al. [43], which showed that plasma application decreased dye concentration as a function of treatment time, resulting in lower color intensity and a change in the lightness of the treated fibers. These findings support the hypothesis proposed at the beginning of the research that the APPJ has the ability to alter colorimetric parameters. Progressive darkening of wool was observed as the plasma exposure time increased, as reflected in the decrease in the L* value. Additionally, the APPJ generated various shades in the cochineal-dyed wool, producing reddish tones (positive a*) and yellow tones (negative b*). These results confirmed the ability of the APPJ to modify the colorimetric parameters of cochineal-dyed wool.

These findings are highly relevant to the textile industry, as plasma treatment offers a more sustainable alternative to traditional dyeing methods that often use metallic mordants. Reducing the use of metallic mordants is a crucial goal for the textile industry because these compounds can be toxic to the environment and human health. Future studies are recommended to evaluate the impact of the APPJ on the colorimetry of other types of fibers and dyes, as well as to test different plasma combinations. Additionally, it would be interesting to investigate the effect of the APPJ on color uniformity and to explore methods to optimize the plasma treatment process to achieve greater uniformity.

4.2. Interpretation of Changes in Textile Characteristics Due to APPJ Treatment

The results of this study indicate that APPJ treatment increased the mean fiber diameter (MFD) and spinning fineness (SF) while reducing the curvature index (CI). These findings are consistent with those of previous studies. For instance, a study conducted by Sun and Stylios [44] investigated the effect of plasma treatment on textile properties and characteristics. After subjecting the wool and cotton fabrics to oxygen plasma, significant changes were observed in the mechanical properties, which were measured using the KES-FB system. Additionally, plasma treatment improved the wettability and surface adhesion of the fabrics, affecting their feel and appearance. Other authors, including Barani and Calvimontes [14], demonstrated that oxygen plasma treatment modifies the surface properties of wool, such as roughness and chemical structure, without significantly affecting the fundamental textile characteristics, such as MFD, FC, CI, and SF. However, unlike these studies, the present work reveals that the APPJ has a significant impact on CI, suggesting that the APPJ might be more effective in modifying the fiber structure than oxygen plasma.

The initial hypothesis of this study was that the APPJ improves the textile characteristics of wool. These results partially confirmed this hypothesis. Although the APPJ did not improve all textile characteristics, it increased the spinning fineness and reduced the curvature index, which could be beneficial for the textile industry.

These findings are relevant to the textile industry, as they suggest that the APPJ can be a useful tool for improving the quality and durability of fabrics. An increase in spinning fineness could enhance fabric strength, while a reduction in the curvature index could improve softness and hand feel. Future studies are recommended to investigate the effect of the APPJ on the mechanical strength of the fiber and its impact on the hand feel and appearance of fabrics. It would also be interesting to explore the effect of the APPJ on the dye absorption and wash fastness of fabrics.

4.3. Discussion on the Influence of Plasma Treatment on Fiber Medullation

Treatment using an APPJ causes an increase in the average fiber diameter (DMF) compared to untreated samples, suggesting a thickening of the fibers [14]. This result is consistent with the findings of previous research, such as that conducted by Barani and Calvimontes [14,29], which demonstrated that oxygen plasma can alter the surface of wool fibers, increase roughness, modify morphology and chemical structure, and increase crystallinity [24]. These changes indicate that plasma treatment can be an effective technique for improving the physical and chemical properties of wool fibers.

The initial hypothesis of this study was that the APPJ would alter the medullation properties of wool. The findings support this hypothesis, as a reduction in the total medullation percentage (TMED) was observed in samples treated for longer periods, suggesting that the APPJ reduces the amount of medulla in the fibers. Additionally, the APPJ affects the distribution of different types of medulla, increasing the percentage of fibers with fragmented medulla (FMED), continuous medulla (CMED), and strongly medullated fibers (SMED) in treated samples [24].

Considering that medullation plays a crucial role in the thermal insulation and strength properties of wool, changes in medullation could affect the quality and performance of wool textiles, thereby affecting their thermal insulation capacity, wear resistance, and texture. These results suggest that the APPJ could be used not only to dye wool sustainably but also to improve its physical and chemical properties, which would have a significant impact on the textile industry.

Therefore, due to the limited number of studies on the effect of the APPJ on the dyeing process and its textile and medullation characteristics, further research is needed to determine the impact of the APPJ on the medullation properties of wool. Future studies could examine the effect of the APPJ on the medullation of other types of wool and its influence on the thermal insulation and strength of textiles. These investigations provide valuable information for optimizing the APPJ treatment process and improving the properties of wool for specific applications.

4.4. Evaluation of APPJ in Wool Dyeing

The use of the APPJ significantly reduces water consumption and has the capability to degrade dyes, allowing for the generation of tones and shades from the red obtained exclusively with cochineal dye, as demonstrated by the colorimetry parameters. Additionally, it optimizes processing efficiency and provides greater color uniformity and fixation than traditional methods that require mordants to achieve these results. It reduces the generation of toxic waste and, although it requires a high initial investment, could be profitable in the long term. Freire-González [45] emphasizes the importance of water use efficiency to reduce global water resource consumption. Penkov et al. [46] conclude that atmospheric-pressure plasma jets significantly improve material processes. The APPJ technique does not require metallic mordants, unlike the traditional method, where it is always necessary to fix the dye on the fiber. Wang et al. [47] underline that the APPJ improves dye uniformity and penetration in textile materials. Moreover, Lin et al. [48] demonstrated that the APPJ combined with wet fixation and drying treatments enhances dye fixation and reduces the use of harmful chemicals. Future research could focus on unveiling the interaction mechanism of the acidic medium generated by plasma reactive species (APPJ) with the medulla of the fibers, allowing for significant changes in them.

5. Conclusions

The results of this study reveal that treatment with an atmospheric-pressure plasma jet (APPJ), particularly LP15, has a significant impact on the colorimetric parameters, textile characteristics, and medullation properties of dyed wool samples. The LP15 treatment reduces lightness (L) to 52.10 $\pm$ 0.82, darkening the fiber, and presents the highest chromaticity values a* (22.91 $\pm$ 1.18) and b* (15.81 $\pm$ 0.45), enhancing the reddish and yellowish tones. Compared to mordant-dyed fibers (LM0) and white fibers (LB), LP15 produced more intense colors.

Similarly, plasma treatments (LP5, LP10, and LP15) significantly improved the mean fiber diameter (MFD) and spinning fineness (SF), with LP15 being the most efficient treatment. This treatment also increased the curvature index (CI) and comfort factor (CF), suggesting an overall improvement in the properties of the treated fibers. The LM0 and LB samples showed the highest percentages of medullated fibers (TMED) with 99.13 $\pm$ 0.23% and 98.59 $\pm$ 0.18%, respectively, while the LP10 sample had a lower percentage of 97.34 $\pm$ 1.09%. Additionally, LP10 showed the highest percentage of fragmented medullation (FMED) with 2.64 $\pm$ 1.04%, and LP15 stood out with the highest percentage of continuous medullation (CMED) with 1.48 $\pm$ 0.02%, indicating the influence of plasma on the colorimetric parameters and medullation properties of the fibers.

These findings suggest that the APPJ is an effective technology for improving the textile quality of wool and reducing reliance on traditional methods that employ toxic chemicals. This has important implications for sustainability and economic impact in the textile industry, as it allows for higher-quality products with lower environmental impact and potential cost savings in production.

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