

Improvement of the Surface Properties of Wool Fabric Using Glow Discharge Plasma

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ABSTRACT: *In recent years, the use of plasma techniques has emerged as a promising approach for improving the surface properties of natural materials. This study investigates the effects of low-pressure direct current (DC) air plasma treatment on wool textiles under different exposure times. Key surface properties, including contact angle, work of adhesion, surface energy and absorption, were measured and found to improve with increased treatment duration. The water contact angle of untreated wool decreased by 47.5% after 1 h, while reductions of 48.11%, 57.78%, 74.24% and 100% were observed for samples treated for 0.5 min, 1 min, 2 min and 3 min, respectively. Notably, the plasma treatment had minimal impact on the tensile strength and shrinkage of the wool. Scanning electron microscopy (SEM) images revealed changes in surface morphology, including degreasing, increased porosity and structural alterations. Fourier transform infrared (FTIR) analysis indicated no significant differences in the functional groups of the treated wool, while DSC curves showed overlapping denaturation enthalpy peaks, suggesting negligible changes in fibre crystallinity. Overall, the plasma treatment enhanced the wettability of wool fabrics, offering an effective method for surface modification.*

Keywords: DC plasma, wool fabric, contact angle, tensile strength, shrinkage

1. INTRODUCTION

The textile industry faces several interrelated challenges, including the development and production of raw materials, processing these materials into finished products, meeting customer expectations regarding health and environmental impacts, and ensuring profitability. These concerns align with

societal priorities, particularly the need for sustainable production systems that emphasise environmental safety, support both rural and industrial communities, and satisfy consumer demands. Advancing the textile industry requires a focus on developing innovative products, alternative raw materials and environmentally friendly processing technologies that leverage natural processes and conditions.¹⁻³

Natural fabrics such as wool are widely used across industries due to their advantageous properties, including high thermal insulation. However, they also pose challenges, such as a matte appearance, pilling, gloss inconsistency and shrinkage.⁴ Plasma treatment has emerged as a promising method to enhance the surface properties of wool fabrics without compromising their inherent qualities.⁵ This technique offers significant advantages, including reduced water and chemical usage, cost savings, and an environmentally friendly, straightforward and secure process.

The efficacy of plasma-treated wool fabric has been evaluated using internationally recognised testing methods. Surface improvements can be assessed through various procedures, such as contact angle measurements, which determine the impact of plasma treatment on hydrophilic characteristics.⁶ Wool fabrics treated with an atmospheric pressure plasma jet (APPJ) under varying conditions, including treatment durations and oxygen flow rates have shown significant reductions in water absorption time and improved wettability. The distribution of water droplets on the treated wool's surface is more extensive and uniform, indicating effective plasma treatment.^{7,8}

The work of adhesion and surface energy of solids can be comprehensively evaluated by analysing the contact angle of a pure water droplet.^{9,10} Studies assessing the water absorption rate of wool fabric have also demonstrated enhancements in wettability.^{11,12} Investigations into wool fibres before and after plasma treatment reveal improved tensile properties, with plasma-treated wool exhibiting higher tenacity and elongation at the breakpoint compared to untreated fabric.^{13,14}

Additionally, low-temperature plasma (LTP) treatment has yielded positive outcomes, including reduced felt shrinkage, improved bursting strength and enhanced moisture absorption.¹⁵ Variance analysis has identified treatment duration as the primary factor influencing the extent of changes in the hygroscopic properties of wool fabric. Compared to untreated wool, plasma-treated wool exhibits distinct physical and chemical characteristics, which impact the efficiency of various textile processes, including dyeing and shrink-proofing. These modifications result in high-quality, versatile wool products.¹⁶

This recent research, we demonstrated the effects of plasma treatment on the wettability of leather and silk.^{17,18} Building on this prior work, the present study investigates the impact of low-pressure direct current (DC) air plasma treatment on the physical properties of natural fabrics, specifically examining changes in wettability, tensile strength, shrinkage and surface characteristics of wool fabrics treated for 0.5 min, 1 min, 2 min and 3 min—a topic not extensively explored in previous research.

2. EXPERIMENTAL SETUP

2.1 Plasma System

An air plasma glow discharge was generated at low vacuum pressure by applying a DC voltage between two parallel stainless-steel electrodes, each with a diameter of 5 cm and separated by a 4.8 cm gap, representing the average distance between the electrodes. This setup was housed in a cylindrical stainless-steel vacuum chamber with dimensions of 25 cm in both diameter and height. The side and back of both electrodes were insulated with ceramic materials. The upper electrode was connected to a 1,000 V DC power supply capable of delivering a current of 2 A, while the lower electrode was grounded. A rotary pump was used to reduce the chamber pressure to a base level of 10^{-2} torr, as illustrated in Figure 1.¹⁹

The system pressure was stabilised at 0.5 torr and maintained throughout all measurements. Wool samples were placed on the sample stage and subjected to DC plasma treatment at voltages ranging from 320 V–340 V for durations of 0.5 min, 1 min, 2 min and 3 min.

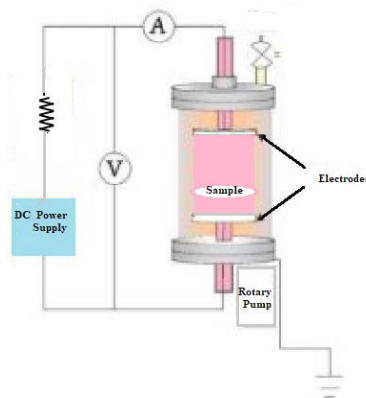


Figure 1: Schematic of the experimental setup.

2.2 Materials

Natural wool samples were procured from Charles Cayton, UK. The fabric used was suit material composed of Super 150 wool and cashmere, specifically 99% super 150 wool and 1% cashmere. It was a medium-weight fabric, approximately 330 g/m², and white in colour.

3. DIAGNOSTIC TECHNIQUES

3.1 Contact Angle Technique

The contact angle between the wool fabric and water was measured to evaluate the wettability of the wool surface. A contact angle less than 90° indicates that the liquid is wetting the surface, whereas an angle greater than 90° suggests the surface is not wetted.^{20,21}

Distilled water was applied multiple times onto the wool fabric surface using a micropipette (60 µl) positioned 1 cm above the sample. Five water drops were measured at exposure times of 0.5 min, 1 min, 2 min and 3 min at a constant voltage of 330 V. A digital camera continuously recorded the drop's behaviour over 1 h. The contact angle (θ) formed between the drop and the surface was measured using the ImageJ software for each drop.²² Adhesion work, defined as the energy required to separate two adhered surfaces, can be either positive (indicating bonding) or negative (indicating no bonding).²³ Adhesion is a critical factor in all applications and is influenced by wettability, which is governed by the contact angle. The work of adhesion between the wool surface and water was calculated using the following equation:²⁴

$$W_{SL} = \delta_L (\cos \theta + 1) \quad (1)$$

where, W_{SL} represents the work of adhesion, δ_L is the surface tension of water (72.8 mN/m), and θ is the contact angle.

Determining the contact angle is essential for calculating the free surface energy, a key property of solid materials. Generally, surfaces with higher surface energy exhibit enhanced wettability, resulting in reduced contact angles.^{25,26} Surface energy was calculated using the following equation.²⁷

$$\cos \theta = -1 + \frac{2(\delta_s \delta_L)^{\frac{1}{2}}}{\delta_L} \quad (2)$$

where, δ_s is the surface free energy of the wool sample.

3.2 Water Absorption Rate

To evaluate the water absorption properties of plasma-treated wool samples, experiments were conducted at treatment durations of 0.5 min, 1 min, 2 min and 3 min. Each measurement was repeated five times, yielding an average result with a standard deviation of 0.024 g. A control sample was included for comparison. The samples were weighed using an electronic balance before being submerged in distilled water for 5 min. Afterwards, they were suspended in the air for 1 min before being reweighed. The percentage change in sample weight was calculated using the following equation.²⁸

$$\text{weight change}\% = \frac{w - w_o}{w_o} \quad (3)$$

where, w_o and w are the weights of the wool fabric before and after water absorption, respectively.

3.3 Tensile Strength

This testing procedure provides a standardised method for evaluating the tensile properties of woven fabrics. Using the strip method, it measures maximum force, elongation, force at rupture and elongation at rupture.^{29,30} Tensile strength and elongation at break were assessed following the American society for testing of materials (ASTM) D 5034 method (Grab Test), with the reported values representing the average of three readings. An Instron testing machine equipped with a 5,000 N load cell (Instron, USA) was used for the measurements. Samples were prepared in accordance with ISO 13934-1 (ASTM Standard Test Method 1994). The tensile testing was conducted at a test speed of 100 mm/min using specimens measuring 5 cm × 20 cm.³¹

3.4 Felting Shrinkage

Felting shrinkage is a significant issue in the textile industry, occurring when fabrics undergo washing or heat exposure, leading to contraction and size reduction. This phenomenon adversely affects garment fit, appearance and quality. The root cause lies in the natural scales present on wool fibres, which tend to interlock and cause fabric shrinkage when exposed to heat and moisture. To quantify this effect, the felting shrinkage test is employed.^{32,33}

The dimensional stability test is a commonly used method to evaluate fabric shrinkage. It involves measuring the fabric's dimensions before and after washing, followed by complete drying. The final dimensions are then compared with the initial measurements to calculate the percentage area shrinkage, as shown in the equation below:^{34,35}

$$\text{Area Shrinkage}\% = \frac{A_{\text{before}} - A_{\text{after}}}{A_{\text{before}}} \quad (4)$$

where A_{before} is the original area (cm²) of the sample; and A_{after} is the area of the sample after treatment.

3.5 Scanning Electron Microscopy Measurements

The surface morphology of untreated and plasma-treated wool fabrics was analysed using scanning electron microscopy (SEM). Samples were examined after treatment durations of 0.5 min, 1 min, 2 min and 3 min using a Quanta FEG 250 electron microscope (Czech Republic) operating at 10 kV–20 kV. The equipment used included the JEOL JSM-5310 and VEGA 3 TESCAN models.

3.6 Fourier Transform Infrared Analysis

The chemical composition of wool fibres, both untreated and treated for durations of 0.5 min, 1 min, 2 min and 3 min, was analysed using a PerkinElmer UATR Fourier transform infrared (FTIR) spectrometer. The spectrum was recorded between 4,000 cm⁻¹ and 500 cm⁻¹. FTIR bands corresponding to various vibrational groups were identified and compared with existing literature.

3.7 Differential Scanning Calorimetry Measurements

Differential Scanning Calorimetry (DSC) is a thermal analysis technique used to measure heat fluxes into or out of a sample as a function of temperature or time. The calorimetric measurements were conducted using the DSC-Q2000 instrument (TA Instruments Co., Germany) under previously established procedures. The system was calibrated for temperature and heat flow as per the standard method.³⁶ All experiments were carried out in an inert nitrogen gas environment (30 mL/min) with a heating rate of 10°C/min. The temperature ranges for the measurements extended from 25°C–300°C.

4. RESULTS AND DISCUSSION

DC glow discharge plasma was generated in air at a fixed pressure of 0.5 torr. Figure 2 illustrates the current-voltage (I-V) characteristics for gap distances ranging from 3 cm–6 cm. An increase in gap distance corresponded to a rise in the discharge breakdown voltage. Additionally, the discharge current increased with higher applied voltage, ultimately reaching the glow-to-arc transition point, which also rose with increasing gap distance.³⁷ The observed outcomes align with the I-V characteristics of DC glow discharge plasma in atmospheric air, as reported in previous studies.^{38,39}

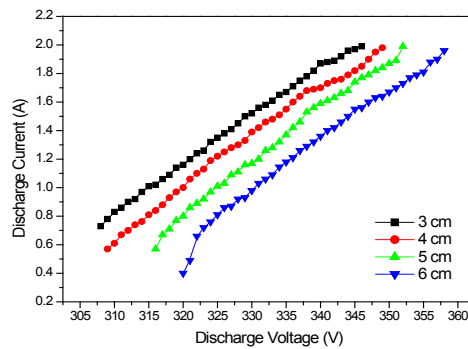


Figure 2: Voltage-current relationship of DC air glow discharge plasma for different gaps.

4.1 Contact Angle Measurement

Figure 3 shows the variation in contact angle values for untreated and plasma-treated wool samples at exposure times of 0.5 min, 1 min, 2 min and 3 min under a 0.5 torr pressure and 320 V discharge voltage, observed over a period of 1 h. The contact angle of water droplets on the wool surface decreased with longer treatment times.⁴⁰ Initially, only slight differences were observed in the angle values for treated and untreated samples immediately after the application of the water droplet. After 1 h, the contact angle of the untreated sample decreased by 47.5%, while the treated samples showed decreases of 48.11%, 57.78%, 74.24% and 100% for treatment times of 0.5 min, 1 min, 2 min and 3 min, respectively. These results indicate that plasma exposure time, enhances hydrophilicity and improves wettability (see Figure 4[A]), likely due to oxidation and the removal of the fat layer on the wool fibre surface.^{41,42}

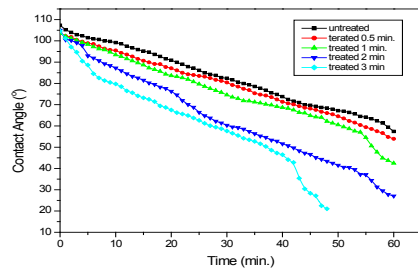


Figure 3: Contact angles of water on the surface of untreated and treated wool samples at different treatment times.

As depicted in Figures 4(B) and 4(C), longer plasma treatment times were directly correlated with increased adhesion work and surface energy. This relationship is attributed to the decrease in contact angle, which renders the surface more hydrophilic.^{43,44}

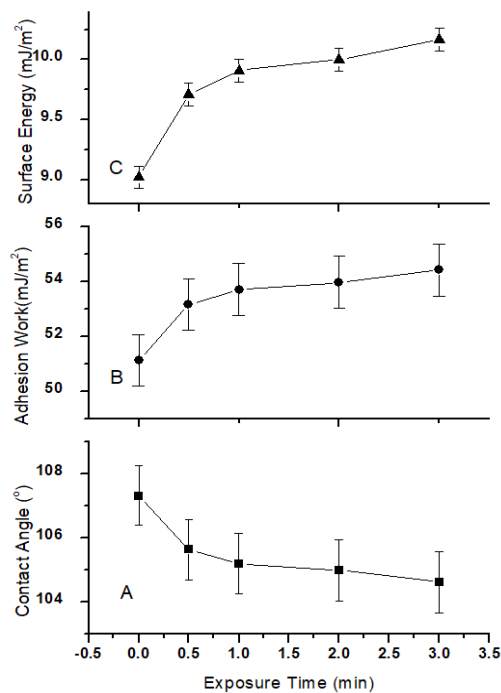


Figure 4: Surface energy, adhesion work and contact angle at different exposure times.

4.2 Water Absorption Ratio

The water absorption rate of untreated and plasma-treated wool fabric samples was measured to evaluate the effects of air plasma treatment. Figure 5 demonstrates a significant enhancement in the hydrophilic properties of the fabric after treatment for 0.5 min, 1 min, 2 min and 3 min. The treated samples exhibited greater water absorption compared to the untreated sample. This improvement in hydrophilicity is attributed to the physical etching and chemical modifications induced by plasma treatment. These findings are consistent with prior research.⁴⁵

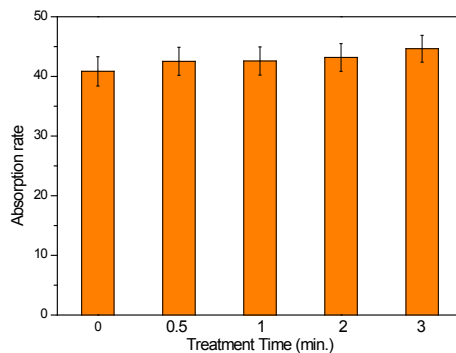


Figure 5: Water absorption rate at different treatment times.

4.3 Tensile Strength and Elongation

The tensile strength of plasma-treated wool fabrics is shown in Figure 6. A slight decrease in tensile strength was observed for most treated samples compared to the untreated sample. However, a marginal increase in tensile strength was noted for the sample treated for 2 min, which may be attributed to the effects of plasma etching. These results suggest that plasma treatment has only a superficial impact and does not significantly compromise the overall quality of the wool.^{46,47}

Similarly, Figure 6 illustrates minor changes in the elongation percentage of treated samples compared to the untreated sample. These findings indicate that plasma treatment does not substantially alter the mechanical properties of wool fibres, as supported by previous studies.⁴⁸

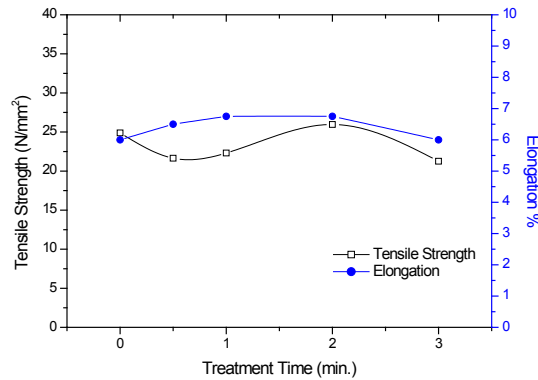


Figure 6: Tensile strength and elongation of untreated and treated wool fabrics at different times.

4.4 Felting Shrinkage Measurement

Felting shrinkage in wool fabrics is primarily caused by the scales present on the wool's surface. As noted by Hassan and Carr, this process results in significant dimensional changes.¹⁵

Table 1 presents the measured felting shrinkage percentages for untreated wool fibres and samples treated for various durations. The overall shrinkage percentage of the wool exhibited a slight decrease following plasma treatment, dropping from 2.6% in untreated samples to approximately 2.6% after 0.5 min and 1 min treatments. The felting shrinkage percentage further decreased to approximately 1.25% after 2 min of plasma treatment. However, a slight increase to 2.8% was observed in the sample exposed to plasma for 3 min compared with the untreated sample. These findings underscore the subtle yet significant impact of plasma treatment on the felting behaviour of wool fibres.^{49,50} The observed effects are attributed to changes on the wool surface, including the formation of new hydrophilic groups, the removal of fatty acids from the fiber's outermost layer, and the etching process.⁵¹

Table 1: Felting shrinkage (%) of untreated and treated wool fabric samples.

Sample	Shrinkage (%)		Felting shrinkage (%)
	Weft direction	Warp direction	
Untreated	0	2.5	Blank
Treated 0.5 min	0	0	2.40
Treated 1 min	0	0	2.60

(continued on next page)

Table 1: (continued)

Sample	Shrinkage (%)		Felting shrinkage (%)
	Weft direction	Warp direction	
Treated 2 min	0	0	1.25
Treated 3 min	0	0	2.80

4.5 SEM Analysis

Figure 7 displays SEM micrographs illustrating the surface morphology of untreated and plasma-treated wool samples. The untreated wool sample (Figure 7[A]) shows a smooth surface cuticle that is relatively dense and features intact scales. This surface exhibits hydrophobic behaviour. In contrast, the plasma-treated wool samples reveal significant surface changes due to the etching effect caused by air plasma bombardment. The SEM micrographs (Figures 7[B]–Figure 7[E]) indicate that the etching effect becomes progressively more pronounced and smoother with increased plasma treatment duration. This surface modification enhances the water absorption of the wool fibres while maintaining their overall material properties.^{52,53}

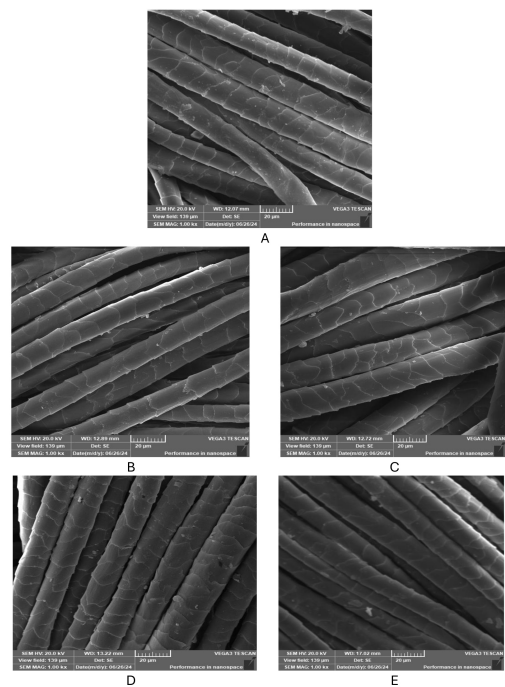


Figure 7: Surface morphology (SEM) of (A) untreated wool fibre, and (B, C, D and E) fabrics treated for 0.5 min, 1 min, 2 min and 3 min, respectively.

4.6 FTIR Analysis

Figure 8 presents the FTIR spectra of untreated wool fabric and samples treated with DC plasma for 0.5 min, 1 min, 2 min and 3 min. Small differences were observed between the spectra of untreated and treated samples. Plasma treatment alters the fibre surfaces, modifies certain compositions, and enhances hydrophilic properties by introducing polar functional groups such as $-\text{CO}-$, $-\text{C}=\text{O}$ and $-\text{COOH}$. Sharp absorption bands were located at $3,276.26\text{ cm}^{-1}$, $2,925.69\text{ cm}^{-1}$ and $1,627.7\text{ cm}^{-1}$.^{24,54,55} The peak at $1,627.7\text{ cm}^{-1}$ is associated with Amide I, which corresponds to stretching vibrations of the $\text{C}=\text{O}$ bond, while the peak at $1,514.04\text{ cm}^{-1}$ corresponds to Amide II, related to the bending vibrations of the $\text{N}-\text{H}$ bond. An increase in absorption intensity was observed in the samples treated for 0.5 min, 1 min and 2 min compared with the untreated sample.^{56,57}

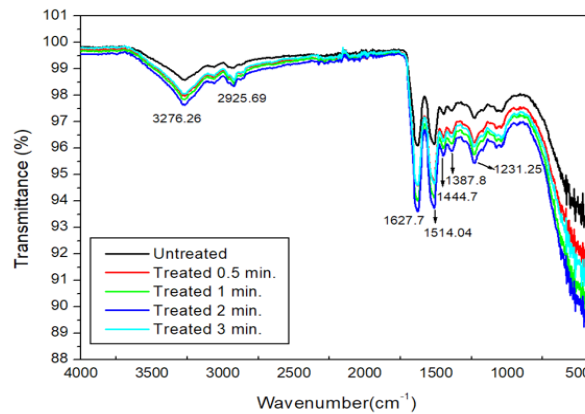


Figure 8: FTIR spectra of untreated and plasma-treated wool fabric samples.

4.7 DSC Measurements

The DSC curves of the samples, shown in Figure 9, exhibit three distinct processing phases characteristic of keratin protein. The primary difference in the DSC curves is attributed to water evaporation. For untreated samples, the glass transition temperature (T_g) of wool was 26.6°C , whereas for treated samples, it was slightly lower at 26.4°C . The glass transition typically overlaps with the initial peak of water evaporation in the DSC curve.⁹

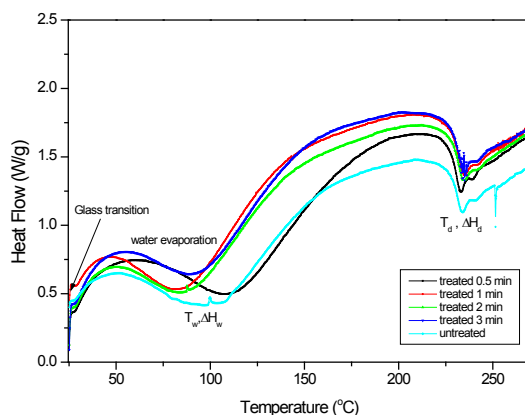


Figure 9: DSC curves of untreated wool sample, and samples treated for 1 min, 2 min and 3 min.

Table 2 summarises the DSC curve characterisation for each sample. Plasma-treated wool exhibited a higher water evaporation temperature (T_w) and greater water evaporation enthalpy (H_w) compared to untreated fabric. The water evaporation enthalpy correlates directly with the water content of the wool fibres, supporting findings that plasma-treated wool absorbs more water due to the removal of intrinsic lipids.⁵⁸

Table 2: DSC values of untreated wool sample, and samples treated for 1 min, 2 min and 3 min.

Sample ID	Glass temperature	T_w (°C)	ΔH_w (J/g)	T_d (°C)	ΔH_d (J/g)
Untreated	26.6	96.17	47.7	234.0	43.16
Treated 1 min	26.4	91.16	32.38	233.67	30.38
Treated 2 min	26.4	86.99	30.50	233.76	38.10
Treated 3 min	26.4	88.56	26.76	235.09	48.87

The DSC curves also revealed a second endothermic heat transfer peak at approximately 232°C, corresponding to denaturation enthalpy, which is dependent on the protein content of the fibres. The enthalpy of protein denaturation provides an indicator of fibre crystallinity.⁵⁸ Overlapping denaturation enthalpy peaks in Figure 9 suggest minimal changes in fibre crystallinity, consistent with the slight changes in shrinkage observed.

5. CONCLUSION

The surface of the wool fabric underwent treatment using DC plasma discharge, resulting in improved hydrophilicity, as demonstrated by contact angle measurements of water droplets on the wool surface. The contact angle decreased consistently with increasing plasma treatment time, indicating enhanced wettability of the plasma-treated wool fabric. This improved hydrophilicity was accompanied by an increase in absorptivity, surface energy and surface roughness. Notably, plasma treatment had no significant impact on the tensile strength or felting shrinkage of the wool fabric. SEM images revealed changes in the wool fabric surface, specifically the removal of fat layers due to plasma etching. FTIR analysis showed no significant differences in the functional groups of wool fabric after plasma treatment. Additionally, the DSC curves exhibited overlapping denaturation enthalpy peaks, suggesting minimal changes in fibre crystallinity. Future work will explore plasma treatments not only for their direct physical effects but also for the chemical and biological changes induced by plasma exposure. Studies will also examine extended treatment durations and improvements in dyeing operations.

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