



Improvement of Wool Surface Characteristics by Using the Atmospheric Pressure Glow Discharge

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ABSTRACT: This study investigates the enhancement of wool fabric properties using atmospheric pressure glow discharge (APGD) plasma. The wool samples are exposed to APGD plasma under different conditions of the current and treatment time. It achieved significant improvements in the wettability (wetting time reduced from 332.4 s–93.6 s), felting shrinkage (from 6.4%–3.93%) and the whiteness improved significantly. The plasma treatment cleans the wool surface and enhances dye uptake, offering a sustainable approach for textile processing. The treated samples were examined by scanning electron microscope Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) spectroscopy that confirm the changes in its surface morphology the formation of new functional groups on the wool fabric.

Keywords: wool treatment, atmospheric pressure glow discharge APGD plasma, whiteness index, wettability, printability

1. INTRODUCTION

Chemical treatments for textiles offer a wide range of reagents for fabric modification, but they also introduce toxic chemicals and require strict process controls.¹ The finishing of fabrics can release harmful compounds into the air while consuming large amounts of chemicals.² Therefore, there has been a growing need to explore alternative techniques that modify textiles without causing environmental harm. Plasma technology has emerged as a promising “green” solution, offering a clean, dry and waste-free alternative to conventional methods. One of the commonly used plasma techniques under atmospheric pressure is the filamentary dielectric barrier discharge (FDBD) mode.³ In this mode, electrical breakdown occurs almost simultaneously

at multiple points on the surface, leading to the formation of numerous short-lived micro-discharges, each with a radius of about 100 μm , generated through a streamer breakdown process. The atmospheric pressure glow discharge (APGD) plasma is a specific mode of dielectric barrier discharge (DBD) plasma known for its uniform characteristics, which result in a relatively homogenous modified layer that covers a broad surface area.² Wool fibre, a natural fibre predominantly made of keratin (approximately 82%), is composed of 95%–98% proteins (around 80%–85% keratin), lipids (0.1%) and minerals.⁴ However, wool fibres face challenges such as felting shrinkage and difficulty with dyeing and printing processes. Plasma treatment offers a cost-effective, dry method for modifying wool properties, as it does not require water or chemicals.^{5–6} In comparison with other plasma techniques such as direct current (DC) plasma, it can be confirmed that APGD plasma is considered to be the most reliable type of plasma as it operated in open air without needing the vacuum system.⁷ Recent studies have explored the importance of DBD plasma in textile treatments.^{8–10} The APGD plasma can damage the squamae of the natural wool to improve the wettability greatly without burning the wool fibre.¹¹ In this study, the APGD mode has been employed for treating wool surfaces under varying conditions of current and treatment time. To achieve the APGD mode plasma, five conditions must be met:

1. A high number of seed electrons to initiate and sustain the discharge in the glow mode under a low electric field.
2. Ionisation should occur under a low electric field to prevent the rapid growth of electron avalanches.
3. The secondary emission coefficient should be high enough to initiate the glow discharge.
4. Increased collision processes, such as electron-neutral and metastable species collisions, to promote ionisation through the Penning effect.
5. Ion density should be high enough to promote secondary electron emission.

Enhancing ionisation and the electron emission from the cathode is vital for sustaining the glow discharge. Additionally, the quantity of electrons left in the gas before a new discharge is initiated plays a critical role. When electrons reach the dielectric surface under low electric fields, they are slightly trapped, which is known as the memory effect. These trapped electrons can be easily released in the subsequent half period, enhancing secondary cathode emission and, consequently, increasing the occurring of a glow discharge.^{8–9}

2. METHODOLOGY

Figure 1 illustrates the discharge cell setup of the APGD plasma. The cell is composed of two parallel steel electrodes, each with a circular shape and a radius of 11.5 cm. Two porous alumina (Al_2O_3) plates are pasted on these electrodes, each with a thickness of 3.5 mm, are attached. The diameter of the two plates is 20 cm with gap is 2 mm. A high voltage transformer is used for generating a sinusoidal voltage in the range of 1–10 kV at a frequency of 50 Hz. This power supply is responsible for driving the discharge within the cell.

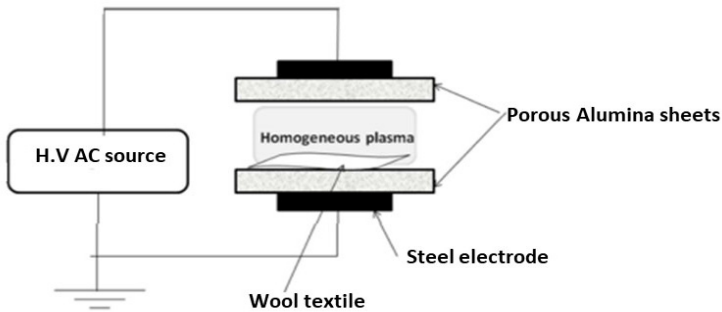


Figure 1: Schematic diagram of the discharge cell.

The discharge was operated in open air at atmospheric pressure, with the applied potential difference (V_a) across the electrodes and the current (I) passing through the system recorded using a digital oscilloscope (Regol —100 MHz), as shown in Figure 1. The voltage across the two electrodes was measured using a potential divider of the resistance system. Optical emission spectroscopy (OES) was employed to analyse the nitrogen spectra in the wavelength range of (300 nm–450 nm). It consists of a McPherson scanning monochromator (Model 270) that is connected to a photomultiplier tube (PMT) type 9558 QB. Infrared (IR) microscopic analysis was conducted with a Nicolet 380 FTIR, Thermo-Electron Corporation, utilising attenuated total reflection (ATR) to obtain transmission IR spectra. To study the surface morphology of plasma-treated fabrics, scanning electron microscope (SEM) photomicrographs were obtained using a JEOL JXA-840A Electron Probe Microanalyser. The water drops test method, following the AATCC Test Method 39-1980, was used to measure the absorbency of the fabric.¹⁰ The time for absorbing the water drop into the fabric was recorded as the absorbency value. Tensile strength of the textile was evaluated using the ASTM standard test method D1294-95a, with a tensile strength apparatus (model H5KT, Tinius Olsen Company). The degree of whiteness of the treated samples was assessed using an Ultra Scan Pro Spectrophotometer from Hunter Lab, employing the light reflectance technique.¹² We test the felting shrinkage

by the ISO 6330:2000 for both APGD plasma-treated and untreated samples. The washing machine used was A-type washer, following the normal TM-31-5A method. The felting area shrinkage (Sa)% was calculated as follows:

$$\text{Felting shrinkage } (Sa)\% = \frac{(OM - FM)}{OM} \times 100 \quad (1)$$

where,

OM = original measurements (before felting)

FM = measurements after felting.

After the felting process, wool fabrics were printed and then dried at room temperature. We apply saturated steam at 105°C for 30 min to fix the sample. The formulation for the printing paste used throughout the study is as follows: 1), 30 g Acid dye (Suncid Blue N-RH), 2), 50 gm Thiourea, 3), 50 gm Urea, 4), 5 gm Ammonium sulfate, 5), 500 gm Guar gum and 6) Equation (1) below:

$$\frac{X \text{ gm water}}{1,000 \text{ gm}} \quad (1)$$

After the wool fabrics were printed, the prints were rinsed with cold water to remove any excess paste. The fabrics were then soaped at 50°C for 15 min using a solution of 2 g/L Scoural CA. Following the soaping process, the fabrics were rinsed with both hot and cold water to further remove any residual chemicals. Finally, the fabrics were air-dried to complete the process. All measurements on the treated wool samples are repeated three times and statistically analysed by statistical deviation.

3. RESULTS AND DISCUSSION

3.1 The Discharge Characteristics

Figures 2(a) and 2(b) present the voltage and current oscillograms of the APGD plasma in air under different applied voltages. Figure 2(a) shows the oscillogram at an applied voltage of 1 kV, while Figure 2(b) displays the oscillogram at 1.2 kV. From these figures, it is apparent that the APGD is characterised by a prominent voltage and current hump that lasts for milliseconds. This main hump is accompanied by small microfilament components that are superimposed on the glow discharge component.

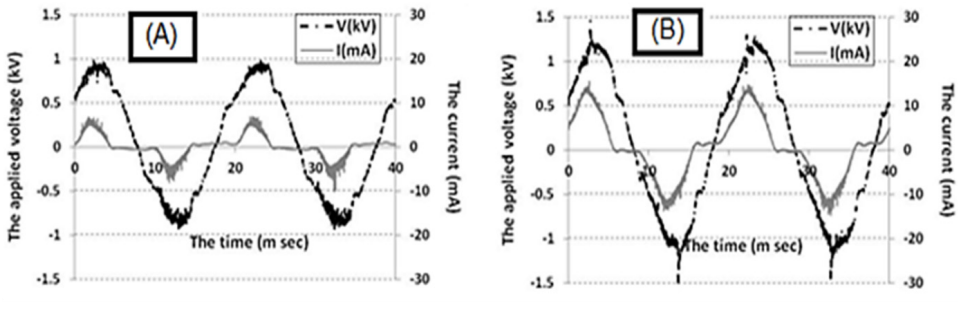


Figure 2: The voltage - current oscillograms in APGD (A and B) at applied voltages of 1 and 1.2 kV, respectively.

The use of porous dielectric barrier sheets in the reactor setup facilitates the formation of the uniform DBD mode known as APGD.¹³ The porous fibre sheets, which contain micro holes, play a key role in this process. When exposed to the applied voltage, an internal discharge occurs within these micro holes of the porous fibre. This internal discharge is crucial because it generates seed electrons that are sufficient to initiate and sustain the discharge in the APGD form within the gas space between the two porous fibre sheets.¹⁴ The mean power consumption of the reactor is evaluated using Lissajous figures, which allow the calculation of the average consumed discharge power.¹⁵ Figure 3 illustrates the relationship between the consumed discharge power of the APGD reactor and the applied AC voltage. The plot in Figure 3 demonstrates how power consumption varies with changes in the applied voltage, providing valuable insights into the efficiency and behaviour of the plasma reactor under different operational conditions.

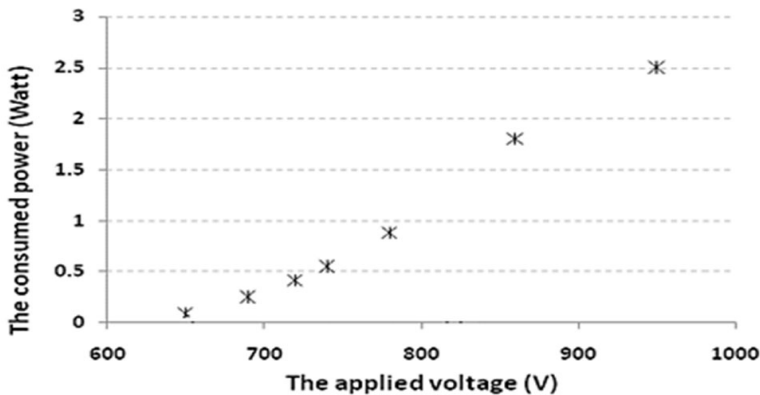


Figure 3: The average consumed discharge power of the APGD as a function of the applied voltage.

In Figure 3, it is observed that the consumed power in the APGD reactor is around 2.5 watts, which is significantly lower compared to other treatment techniques. This low power consumption highlights the APGD as an energy-efficient, cost-effective and powerful method for textile treatment, making it an attractive option for industrial applications. In the APGD plasma, the conductivity is the result of both drift and displacement currents. The emitted spectra from the air discharge in the APGD reactor are shown in Figure 4, covering the wavelength range of 300 nm–450 nm. Since the discharge is operated under open air, the dominant emissions are from the excited nitrogen molecules in the discharge. The nitrogen second positive system (N_2 2P system) bands at wavelengths; 313.6 nm, 337.1 nm, 353.6 nm, 357.6 nm, 375.5 nm, 380.49 nm, 399.8 nm, 405.9 nm are seen under all conditions of discharge.¹⁶ The nitrogen second positive bands are corresponding to the transition of the excited nitrogen molecules from the ($C^3\Pi_u$) excited electronic state ($v' = 0-2$) to the low-laying ($B^3\Pi_g$) excited state ($v'' = 0-4$) according to the following equation:¹⁷



The nitrogen first negative systems (N_2 1N system) are also observed of small intensities. The intensities of the excited peaks increased to about double values by increasing the discharge current from $I = 4$ mA to $I = 7$ mA. The presence of these observed bands highlights the crucial role of excited nitrogen molecules in modifying the wool surface. These nitrogen species are essential in driving chemical reactions on wool fibres, which can lead to changes in properties like surface energy, dye absorption and texture. The energy released by the excited nitrogen molecules aids in activating the wool fibres, making them more receptive to various treatments, without the need for additional chemicals or water. This further demonstrates the effectiveness of the APGD plasma treatment as a sustainable and efficient method for enhancing wool's characteristics.

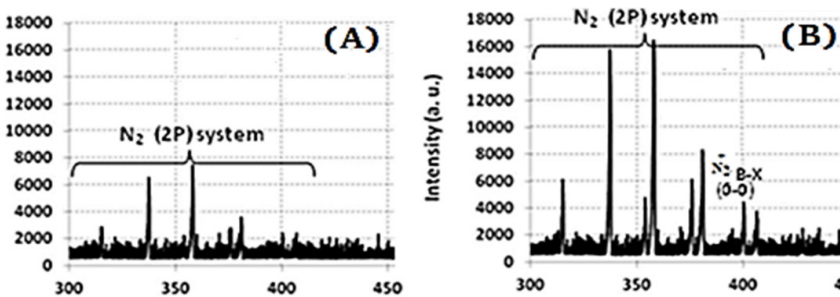


Figure 4: The typical optical emitted spectra from the APGD reactor at discharge current of 4 mA and 7 mA, respectively.

3.2 Textile Surface Properties

The morphology of the surface of untreated and APGD-treated wool fabrics were shown in Figure 5, captured using a scanning electron microscope. The wool sample was treated by APGD plasma for 10 min where the discharge current was 10 mA. In Figure 5(a), the untreated wool fibres appear an outer layer of cuticle cells that overlap, creating a directional frictional coefficient. On the other hand, after plasma treatment, as shown in Figure 5(b), the morphology of the wool undergoes significant changes. The APGD plasma oxidise the cuticle cells and form microcracks and microholes. Despite the presence of these microholes, their small size does not damage the fabric. Instead, it enhances the wool's wettability and its ability to absorb dye, as will be demonstrated later. Additionally, the etching effect softens the fabric, removing the scales and making it more pliable compared to the untreated sample.

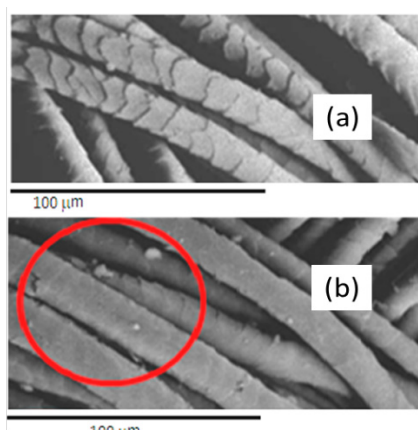


Figure 5: SEM pictures of (a) untreated, (b) APGD treated wool samples, respectively.

Figure 6 presents the whiteness measurement as a function of treatment time at two different discharge currents. The whiteness of the wool increases sharply compared to the untreated sample, and eventually reaches a saturation point as the treatment time continues to increase. This increase in whiteness can be attributed to the high concentration of excited nitrogen molecules and ions generated in the discharge reactor, as observed in the emission spectra. These excited species bombard the wool surface, effectively cleaning it by removing contaminants. Additionally, this process activates the surface, preparing it for better absorption of the printing dye, improving the dye uptake and overall appearance of the fabric.

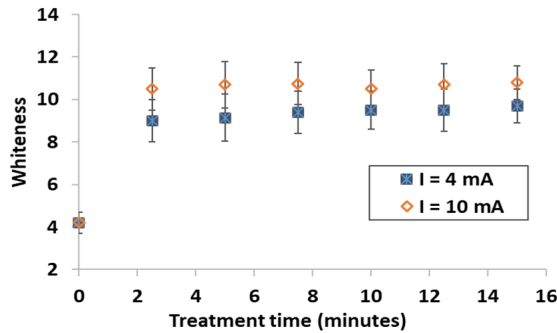


Figure 6: The witness as a function of the treatment time for APGD at discharge current of 4 mA and 10 mA.

Figure 7 presents the wettability measurements of untreated and APGD-treated wool samples using the water drop method, under two different current conditions. In the figure, it is evident that plasma treatment significantly improves the wettability of the wool. The wetting time for the untreated sample is 332.4 s, while for the APGD plasma-treated samples, it is reduced to approximately 93.6 s. This decrease in wetting time indicates that the plasma treatment effectively enhances the wool’s ability to absorb water, likely due to the surface modifications that increase its hydrophilicity. It was found that further increasing the discharge current causes increasing in the intensity of the microfilaments that superimposed on the discharge current, which in turns may cause partial damage of the porous alumina that is used as a dielectric barrier. This is the reason that we didn’t increase the discharge current beyond 10 mA.

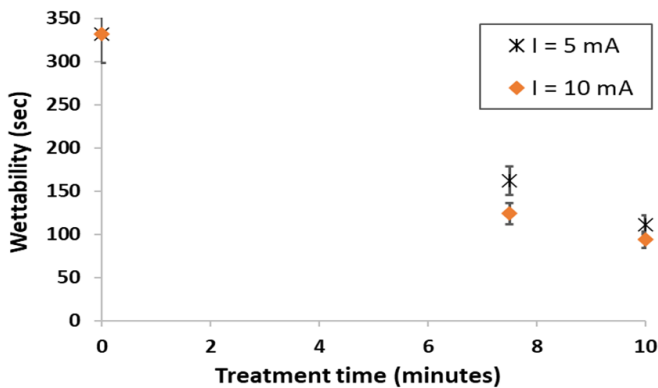


Figure 7: The wettability of wool as a function of the treatment time at discharge currents of 5 mA and 10 mA.

The wettability of the wool improves as both the plasma treatment time, and the discharge current are increased. The ions and other species generated by the plasma bombard the wool surface, leading to oxidation and partial removal of the highly hydrophobic covalent bonds such as fatty acids that are found on the cuticle cells.¹⁸ There is an obvious effect on oxidising the disulfide bonds in the epicuticle layer, reducing the cross-link density. Therefore, the treated wool sample tends to be more hydrophilic than the untreated one.

Figure 8 shows a typical FTIR spectrum for untreated and APGD-treated wool fabric surfaces. There is a slight increase in the peaks of the N-H, C-H and OH groups at wave numbers $3,373\text{ cm}^{-1}$, $2,923\text{ cm}^{-1}$ and $3,065\text{ cm}^{-1}$, respectively in the APGD plasma-treated sample (Figure 8[b]) compared to the untreated wool sample. However, the NH_2 groups do not show an increase in the treated samples, which may be attributed to the etching process occurring during plasma treatment at high currents. This etching breaks down certain surface structures, preventing the formation of NH_2 groups. The spectrum clearly indicates that oxidation and nitridation processes occurs in the treated sample, as evidenced by the appearance of oxygen and nitrogen-containing functional groups such as $-\text{COH}$, N-H and C-H. This confirms that APGD plasma treatment is highly effective in incorporating oxygen and nitrogen onto the wool surface due to the fast reactions between radical ions and fabric.

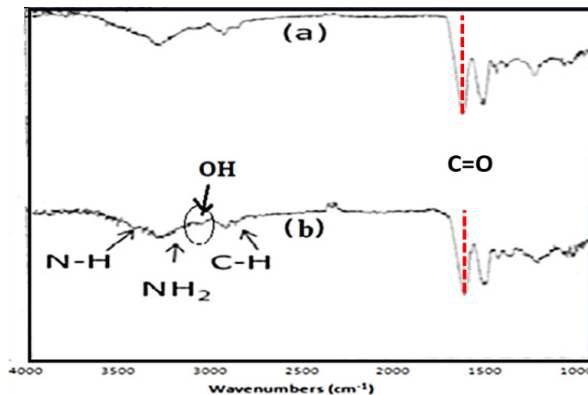


Figure 8: FT-IR absorption spectra for the untreated and treated and APG treated wool samples respectively at discharge current of 10 mA and treatment time of 10 min.

It was noticed that there was a slight decrease in the carbonyl peak ($\text{C}=\text{O}$) at about $1,620\text{ cm}^{-1}$ compared to the untreated sample. This reduction indicates the partial degradation or breaking of protein structure by plasma which is commonly observed in treatments aimed that enhances the dyeing properties of wool fibres. Previous studies have reported similar degradation effects by plasma treatment compared to enzyme

treatments, noting that enzyme treatments do not induce such degradation.¹⁹ On the other hand, Canal et al., stated that after exposure to air plasma, which contains about 20% oxygen, hydrophilic groups such as (-COH, -COO, -C=O) are formed on the fibre surface.²⁰

Felting shrinkage, which is specific to wool fabrics, occurs due to the presence of scales on the wool surface.²¹ This phenomenon is directly related to the directional frictional effect (DFE) found in wool fibres. When wool fabric is exposed to mechanical action in the presence of moisture, the individual fibres begin to move. However, due to the scales on the surface, the fibres are restricted to moving in only one direction. When washed or tumble dried, the combination of mechanical agitation, friction, moisture, pressure and heat causes the edges of one fibre's scales to interlock with the gaps between the scales of another fibre, resulting in a "ratchet" effect that leads to felting.

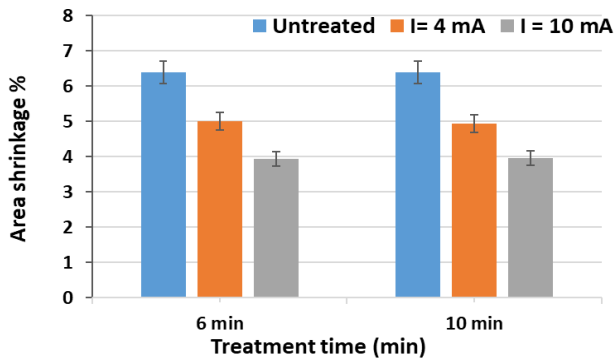


Figure 9: The area felting shrinkage % of wool as a function of the treatment time at discharge currents of 4 mA and 10 mA.

Figure 9 illustrates the shrinkage percentage of wool fibres for both untreated and APGD plasma-treated samples under different treatment conditions, including various treatment times and discharge currents. The results show a noticeable reduction in the area felting shrinkage percentage, from 6.4% for untreated wool to about 3.93% after treatment with APGD plasma at a discharge current of 10 mA and a treatment time of 10 min. This reduction highlights the significant role of plasma treatment in decreasing the surface scales and directional frictional effects, as observed by SEM analysis. After plasma treatment, the wool fibres become more hydrophilic, allowing a thin layer of water to form between the fibres during washing. This layer reduces friction between the fibres and helps further decrease felting shrinkage. While extending the treatment time from 6 min to 10 min does not have a significant effect within this small range, increasing the discharge current leads to a noticeable improvement in felting shrinkage. In comparison with the enzyme treatment on wool

fabric, protease enzyme treated wool fabric shows least area shrinkage (3.0%) followed by laccase enzyme (4.3%), lipase enzyme (4.9%) and transglutaminase enzyme (7.9%) treated fabrics, as compared to 13.3% of the untreated (blank) fabric.²² The difference in initial untreated shrinkage values suggests different fabric conditions or testing methods between the studies.

The influence of plasma treatment on the tensile strength TS and elongation percentage (E) at break of wool fibres has been improved as shown in Table 1. These improvements are attributed to the etching action of the plasma, that roughens the surface of the fibres.²³ This roughening effect creates more contact points at the microscopic level within the fibres and at the macroscopic yarn scale, potentially enhancing the wool mechanical properties. On the other hand, based on the FTIR spectroscopy results showing that plasma treatment causes partial degradation of the protein (keratin) structure in wool, increasing the plasma treatment conditions may raise the risk of wool fibres becoming brittle and more prone to tearing or breaking under tension

Table 1: Tensile strength of wool fabric and elongation to break % at different APGD plasma conditions

Condition of plasma treatment	Tensile strength of wool fabric (kg/mm ²)	Elongation to break (%)
Untreated	32.55	18.23
5 mA–7.5 min	35.12	19.92
5 mA–10 min	35.93	21.88
10 mA–7.5 min	35.62	21.53
10 mA–10 min	36.66	22.37

The increase in the number of contact points between fibres and yarns, as a result of plasma treatment, enhances the inter-yarn and inter-fibre friction. This increase in friction leads to the development of a larger cohesive force when tensile stress is applied. Additionally, the diffusion of nitrogen species formed within the plasma reactor interstitially among the fabric contributes to the improvement in TS. Figure 10 illustrates the effect of the plasma discharge current on the colour strength of the printed wool fabric after APGD treatment for a duration of 10 min.

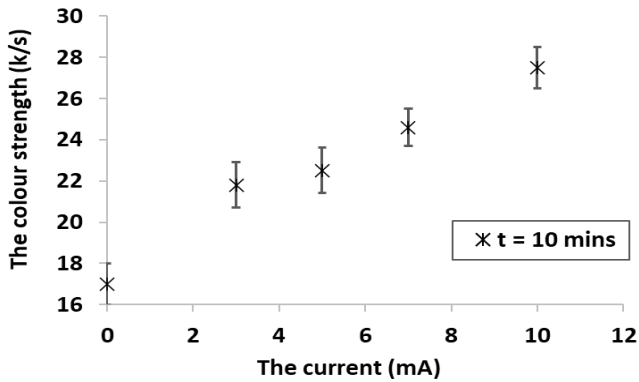


Figure 10: The color strength as a function of the discharge current APGD treated wool at treatment time $t = 10$ min.

A clear increase in colour strength (K/S value) is observed as the discharge current increases. This increase in colour strength can be attributed to several factors:

1. Surface activation: plasma treatment increases the surface area and introduces more active sites on the wool, improving the fabric's ability to interact with dyes and enhancing colour uptake.
2. Improved wettability: the enhanced wettability of the treated wool, as seen in previous measurements, allows the dye to spread more evenly and bond more effectively to the fibres, resulting in stronger colour strength.
3. Producing micro holes: the concentration of the printing dye continues to rise with the increase in discharge current, as the increased number of micro holes provides more sites for the dye to adhere to and be absorbed by the fabric.²⁴ Recent researches immobilised nanoparticles such as ZnO, TiO₂ and CuO into the wool fabric to improve its properties and the dyeing colour strength improved to about 14%.²⁵

4. CONCLUSION

This study highlights the significance of treating wool fabrics with APGD plasma and its impact on the surface characteristics of the wool. The optical spectra of the plasma reveals an increase in the intensity of peaks associated with excited nitrogen species, which play a key role in activating the wool surface. These species contribute to the formation of oxide and nitride groups on the wool, enhancing its properties. The treatment with APGD plasma improves the wettability of the wool, allowing better

dye absorption and more uniform colour distribution. Additionally, plasma treatment leads to improvements in other textile properties, such as colour strength, surface roughness and overall fabric performance. These findings demonstrate that plasma treatment is an effective method for enhancing the properties of wool fabrics without the need for harsh chemicals or extensive processing, offering a more environmentally friendly and efficient alternative to traditional textile treatments. In future research, we aim to improve the properties of the insulating material (porous alumina) so that it can withstand currents higher than 10 mA, in order to study the effect of high currents on the properties of plasma-treated wool fabric.

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