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RESEARCH ARTICLE OPEN ACCESS

Studying the effect of the discharge modes of DBD plasma on the treatment of wool textiles

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ABSTRACT
In the present study a comparison between the influence of the atmospheric pressure glow discharge APGD mode and the filamentary discharge FD mode on the treatment of the wool textiles has been reported at different conditions of the current and treatment time. The induced changes in wool properties, such as the wettability, surface morphology and printability properties have been investigated. The surface characterization was performed using SEM and FTIR imaging. It has been discovered that APGD mode of DBD plasma is more efficient than FD mode. It has been found that the concentration of nitrogen excited species that are the responsible for the surface activation of the wool textile in APGD is more than its concentration in FD mode.

KEYWORDS: DBD modes, Atmospheric pressure glow discharge APGD mode, Filamentary discharge FD mode, wool treatment

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I. INTRODUCTION

Recently, the dielectric barrier discharge DBD plasma can be used as an effective technique for modifying the surface properties of wool fabric such as its whiteness, wettability, shrinkage, printability, surface morphology, mechanical properties, dyeability, and fastness properties without much alternation to the interior of the fiber. In this work, two different modes of DBD have been examined to discover the best conditions for modifying the wool fiber properties. These modes are the filamentary mode FD and the atmospheric pressure glow discharge APGD mode.

In filamentary FD plasma the electrical breakdown starts simultaneously at many points of the surface as a short-lived microdischarges of about 100 μm radius, each one generated from a streamer breakdown. Due to the short duration of the microdischarges and their small volume in comparison with the entire gas gap, plasma remains strongly non-thermal. When a filamentary discharge occurs multiple current pulses per half cycle are observed [1].

In the glow APGD plasma a uniform region extending uniformly over the whole electrode surface. Some conditions should be verified to obtain the glow mode at atmospheric pressure: a high number of seed electrons, an ionization mechanism under low fields, a high value of the dielectric secondary emission coefficient. In order to avoid streamers formation electrons should be produced in small electric field.

As an intense space-charge is not generated, ions have time to reach the cathode to promote the secondary electrons emission, so an increase of ion density is also necessary. Besides the electron-neutral collisions, metastables species play an important role in promoting ionization by Penning effect. An increase of the ionization and of the number of electrons emitted from the cathode is not only sufficient to ensure a glow regime but also the number of electrons remaining in the gas before the occurrence of a new discharge is quite important. As the electrons arrive at the insulator surface under low electric fields, they are slightly trapped: this is called memory effect. These electrons can be easier released in the next half period enhancing the value of secondary cathode emission and thus enhancing the probability of obtaining a glow discharge [1] and [2].

II. EXPERIMENTAL DETAILS

2.1. Plasma Set up

Figure (1) represents two discharge cells of APGD and of FD plasmas. These two cells have the same dimensions except for the type of the dielectric barrier. In the APGD cell the dielectric barrier is a commercial porous fiber while in the FDBD cell the barrier is a Pyrex glass. It has been found that changing the type of the dielectric barriers acquires the discharge different properties. The two parallel metal electrodes in the two cells are made of steel of a square shape and their dimensions is 20 cm. The gap distance between the two dielectric is 2 mm. A high voltage transformer (1–10 kV), which generates sinusoidal voltage at a frequency of 50 Hz, was used as an electric power supply to derive the discharge system.

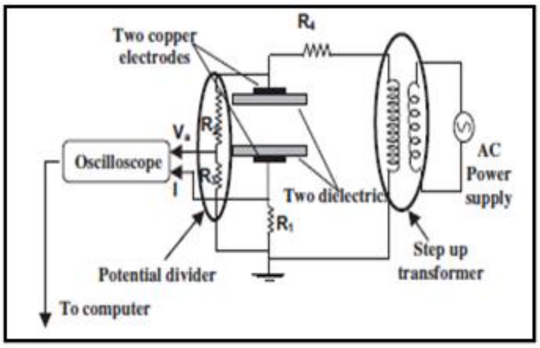

Fig. (1) Schematic diagram of the discharge cell

The discharge was operated in open air under atmospheric pressure. The applied potential difference (Va) across the electrodes and the current (I) passing through the system were recorded using a digital oscilloscope (Regol —100 MHz). The current was measured by the voltage drop across the resistance R_1 (=100 Ω) connected in series with the discharge system to the ground as shown in figure 1. The voltage across the two electrodes was measured using the potential divider of the resistance system R_2, R_3, where $R_2/R_3 = 500$. An optical emission spectroscopy (OES) technique consists of a McPherson scanning monochromator [mo- del 270] with a grating of 1200 grooves mm−1 and resolution of less than 2 Å has been used to study the nitro- gen spectra in a wavelength range of 300 - 420 nm. The monochromator was then connected to photomultiplier tube (PMT) type 9558 QB, which has a resolution time of less than 1 nanosecond, working at voltage of 1200 volts.

2.2. Materials

Substrate Wool:
Mill scoured wool fabric (100%), plain wool fabric of 200g/m^2 .

2.3 printing

Wool fabrics were printed, after printing and drying at room temperature, samples were fixed by saturated steam at 105ºC for 30 min. The printing paste formulation which used throughout this study is shown below:

30 gm Acid dye (Suncid Blue N-RH)
50 gm Thiourea
50 gm Urea
5 gm Ammonium sulfate
500 gm Guar gum
X gm water
1000 gm

2.4. Scanning Electron microscope (SEM)

The SEM photomicrographs were recorded using JEOL, JXA-840A Electron probe microanalyzer, to study the changes in the surface morphology of plasma treated fabrics.

2.5. Infrared

Infrared (IR) microscopic analysis was carried out by using Nicolet 380 FT-IR, crystal ZnSe, Thermo electron corporation , using Attenuated total reflection to obtain transmission IR spectra.

2.6. Wettability

water-drop test was applied according to AATCC test method 39-1980 **[3].** The time required for the drop of water to be absorbed into the fabric will be referred to as absorbency values.

2.7. Color strength

The color strength (K/S) of the printed samples was evaluated by Ultra scan pro spectrophotometer, Hunter Lap, by light reflectance technique and The K/S values of the prints were automatically calculated according to Kobelka-Munk equation. [4]

2.8. Felting shrinkage of wool fabric A felting shrinkage test was carried out according to the ISO/FDIS 6330 method (IWTO-20-69, 2000) for APGD-treated, FDBD-treated and untreated fabrics. The washing machine used was an A-type washer and followed the process of normal TM-31-5A method. Felting area shrinkage Sa (%), was obtained as follows:

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

Where OM= original measurements (before felting), FM= The measurements after felting.

III. RESULTS AND DISCUSSION

3.1 The discharge characteristics

3.1.1. The voltage - current waveform

Figure 2(a) and (b) represents the voltage and current oscillograms of the FD and the APGD plasmas respectively in air at applied voltage 1.2 kV.

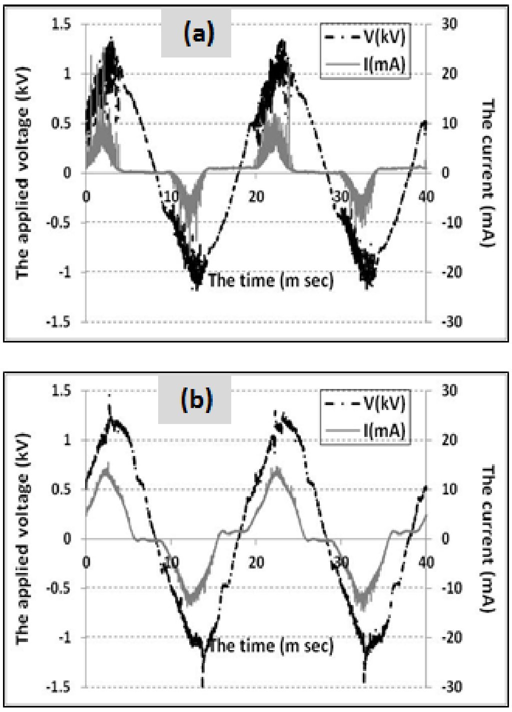

Fig. (2) I-V characteristic curve for the two modes at applied voltage 1.2 kV where a) FD mode and b) APGD mode.

It can be noticed that at the same applied voltage the discharge current in the FD plasma is characterized by short lived filaments of duration about tenth of nanoseconds. However, the APGD plasma is characterized by a large hump of duration in milliseconds with small component of microfilaments that are superimposed on the glow component.

APGD plasma is formed by using porous dielectric barrier sheets [5]. Because of the special configuration of the porous fiber sheets, which are characterized by the existence of micro holes. An internal discharge takes place inside the micro holes of the porous fiber. This internal discharge provides seed electrons sufficient for the initiation and growth of the discharge in the APGD form inside gas between the two porous fiber sheets as it was stated in [6].

3.1.2. The consumed power in the discharge cell

The average consumed discharge power for the FD and APGD reactors that are calculated from the Lissajous figures are plotted versus the applied ac voltage in Figure (3)

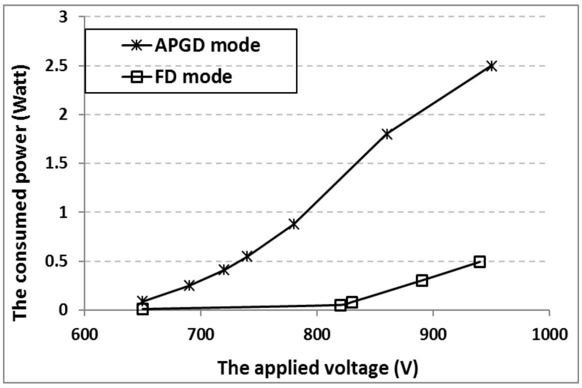

Fig. (3) The average consumed discharge power for the FD and APGD reactors as a function of the applied voltage.

From the figure (3) it can be noticed that the consumed power in the APGD reactor is greater than that consumed in the FDBD one at the same voltage. This behavior may be related to the large resistivity of the FDBD since the conductivity is due to the displacement current only. However, in the case of the APGD the conductivity resulted from both the drift and the displacement currents.

3.1.3. Optical emission spectroscopic (OES) characteristics

Typical emitted spectra of air discharge from the FD and APGD reactors at the same conditions have been observed as shown in Figure (4)(a and b). The discharge current is I= 7 mA. The range of the wavelengths is (300-500 nm). The dominant emitted spectra are the nitrogen second positive systems (N_2 2P system) bands at wavelengths; 313.6, 337.1, 353.6, 357.6, 375.5, 380.49, 399.8, 405,9nm that are seen in both figures [7]. The nitrogen second positive bands are corresponding to the transition of the nitrogen molecular from the ($C^3\Pi_u$) excited electronic state (ν' = 0-2) to the low-laying ($B^3\Pi_g$) excited state (ν'' = 0-4) according to the following equation [8] i.e.:

$$N_2(C^3\Pi_u) \rightarrow N_2(B^3\Pi_g) + h\upsilon \qquad (1)$$

The nitrogen first negative systems (N_2 1N system) are also observed for both modes.

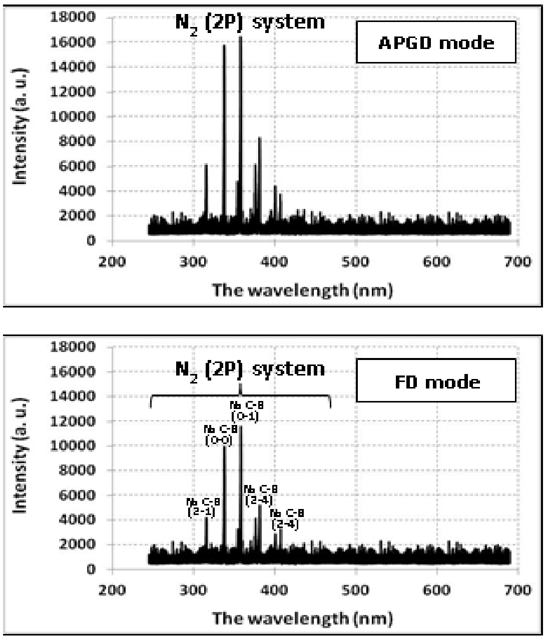

Fig. (4) Typical optical emitted spectra for APGD and FD modes of DBD respectively

It can be noticed that the peak intensities of all wavelengths of all nitrogen bands in APGD plasma are greater than that of the FD plasma. This reflects the importance of APGD in treatment of the textiles than the FD plasma.

3.2. The textile surface properties

3.2.1. Scanning Electron Microscope (SEM)

Figure (5) represents the photos of the surface morphology of the wool fabric that are taken by using scanning electron microscope for the untreated, FD and APGD treated wool samples respectively. The wool samples are treated by FD and APGD plasmas at the same conditions; the treatment time is 15 minutes and the discharge current is 20 mA.

From the figure (5a) it can be noticed that the outermost layer of the untreated wool fibers is the cuticle cells that are overlapped to each other to create a directional frictional coefficient. However, the treatment of the wool by plasma changes the morphology of the wool as it seen in figures (5b and c).

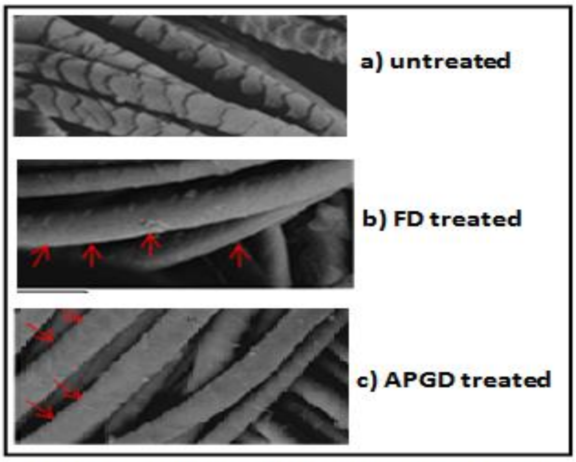

Fig. (5) SEM pictures of a) untreated, b) FD treated and c) APGD treated wool samples respectively.

In the case of FD plasma, (Figure 5b), the treated samples were exposed to a nonhomogeneous plasma that consists of locally micro discharges on the surface of the fabric. The local micro discharges that are usually called microfilaments cause a strong local oxidation and etching of the cuticle cells in some regions as it seen in figure (5b). The etching process removes the scales of the cuticle cells that it makes the fibers of the FD treated wool sample seems to be softer than that in the untreated one (represented by arrows). However, the strong oxidation process creates microcracks and microholes in the surface that harms the textile in some cases of the high-power plasma.

From Figure (5c) it can be noticed that the morphology of the APGD treated wool sample differs from the previous samples. The APGD plasma is homogeneous and causes the oxidation of the cuticle cells and formation of the microcracks and microholes but the dimensions of the microholes are very smaller than that created in the FD. So, it doesn't harm the fabric but it improves the wettability of the wool and its absorption of the dye as it will be seen later. Moreover, the etching process makes the fabric softer and free from the scales.

3.2.2. Wettability

The results in the Table (1) represent the wettability measurements using the water drop method for the untreated, the FD and APGD treated wool samples respectively at the same conditions.

Type of plasma	Condition of plasma treatment	Wettability (Sec.)	Area Shrinkage %
Untreated		332.4	6.40
APGD mode	5 mA-10 min.	111.6	4.94
	10 mA-10 min.	93.6	3.96
FD mode	5 mA-10 min	95.4	4.47
	10 mA-10 min.	45	4.94

Table (1) The Wettability and area shrinkage % properties for the untreated, APGD treated and FD plasma treated wool samples at different conditions of the current.

From the results, it can be noticed that the plasma improves the wettability of the wool treated samples irrespective the type of plasma used under the investigation. The wetting time of the untreated sample is 332.4 sec while it reaches about 45 sec for the FD plasma treated samples and 93.6 sec for the APGD plasma treated samples. The wettability improves by increasing the plasma discharge current. This behavior can be interpreted by removing of the highly hydrophobic covalently bound fatty acids surface of the cuticle cells [9] via plasma treatment, the ions and other species in plasma bombards the textile surface and causes an oxidation and a partial removing of the hydrophobic cuticle cells, also the plasma treatment has a strong effect on oxidizing the disulfide bonds of the epicuticle layer and reducing the cross-link density. As a result of this process, the surface of the wool fabric changed to be more hydrophilic. The hydrophilic groups such as (-COH, - COO, -C=O) will be formed on the surface of fibres [10] after the treatment of the wool by air plasma that contains about 20% O_2. The wetting time of the FD treated sample is smaller than that of the APGD treated sample. This may be related to the fact that dimensions of the created voids and microholes in the case of the inhomogeneous FD plasma is larger than that created by APGD plasma. As a result, the large voids absorb the water drop very quickly.

3.2.3. The area felting Shrinkage

Felting shrinkage is a mechanism of shrinkage that is confined to wool fabrics and it is a direct consequence of the presence of scales on the wool surface [11]. Felting is related to the directional frictional effect (DFE) which is found in wool fibers. The results that shown in the Table (1) represent the area felting shrinkage percentage of the wool fiber for the untreated, FD and APGD plasma treated samples respectively at different conditions of the treatment time and discharge current. It can be noticed that the area felting shrinkage percentage of the wool is decreased from 6.4% to about 4.94% when it was treated by FD plasma and to about 3.39% when it was treated by APGD plasma at the same discharge current and treatment time. This behavior reflects the important effect of the plasma in the reduction of the scales and the directional frictional effect as it was seen by SEM. Moreover, after plasma treatment the fiber is more hydrophilic and then a layer of the water can be formed among the fibers during the washing procedure that reduces their friction and causes felting reduction.

3.2.4. FTIR measurements

Infrared (IR) spectroscopy is a chemical analytical technique, which is mainly used to determine the functional groups in the treated sample. Figure (6) shows a typical IR spectrum of untreated, FD and APGD treated wool fabric surfaces.

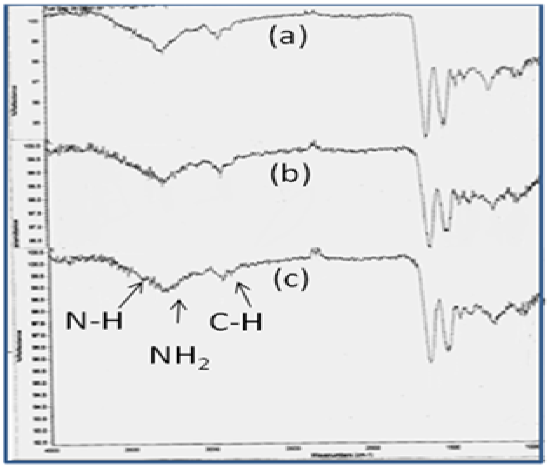

Fig. (6) FT-IR absorption spectra for the untreated, FD and APGD treated wool samples respectively at I= 1 mA and t = 10 min.

It is clear that, from the spectrum an oxidation process in the treated sample is occurred. The Oxygen containing groups such as C-O and C=O is obviously observed. This finding indicated that, an air plasma treatment is a highly efficient in incorporating Oxygen on the surface due to the fast reaction between the radical ions and the fabric surface. The produced chemical groups are increased active sites on the treated surface which led to improve its properties. There is a slight increase in the N-H and C-H groups at wave numbers 3373 and 2923 cm^{-1} has been observed in the APGD plasma fig. (6c) rather than in the untreated and the FD treated samples. The NH_2 groups are not increased in the treated samples. This may be due to the etching process in the plasma at high current.

3.2.5. Color strength

Figure (7) represents the effect of the plasma discharge current on the color strength of the printed wool fabric after treatment by FD and APGD plasma respectively at the same treatment time t= 10 min.

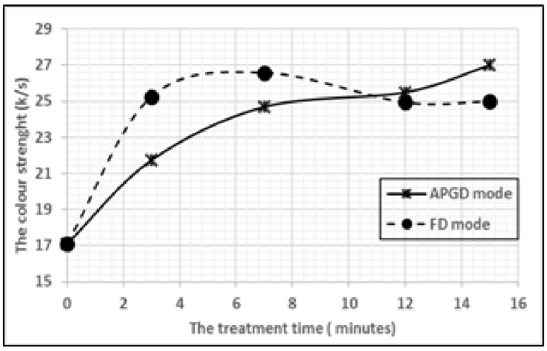

Fig. (7) The color strength as a function of the treatment time for a) FD and b) APGD treated wool at discharge current I= 10 mA.

An obvious increase in the color strength has been observed for the two cases of plasma treatment. However, the K/S still increases by increasing the treatment time of the APGD plasma while it saturates in the case of the FD plasma. This behavior is related to the following reasons; Increasing the treatment time in FD causes the increasing of the number of the plasma microdischarge filaments and its dimensions. This in turns led to further absorption of the printing dyes. Therefore, the concentration of the dye on the fabric surface is lower than that in its bulk. On the other hand, increasing the treatment time in the APGD plasma causes the increasing of the number of the microholes in the fabric without the enhancement of its dimensions (APGD plasma is homogenous). Therefore, the concentration of the printing dye still increases by increasing the treatment time. As it had been seen previously in the total scan of the emitted spectra from the FD and the APGD plasma, the intensity of the peaks that related to the excited nitrogen second positive systems that emitted from the APGD reactor is greater than that emitted from the FD one. So, the introduction of the NH_2 groups into the fiber is greater in the case of APGD plasma which is responsible to increase the absorption of the anionic dye (acid dye) [12].

IV. CONCLUSION

The comparison between the FD and the APGD plasma shows that APGD mode is more homogeneous than the FD mode. The optical spectra of the two types of plasma show the increasing of the peaks intensities that related to the excited nitrogen species and that are responsible in the activation of the wool surface and the formation of oxide and nitride group on the wool surface. The wettability has been increased by the treatment current of the wool by the two types of plasma. Also, the color strength and the other proprieties of the textile have been improved by plasma treatment. The APGD plasma is more efficient than FD in color strength properties of the wool.

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REFERENCES

[1]. U. Kogelschatz, , B. Eliasson, W. Egli, Pure Appl. Chem., Vol. 71, No. 10, pp. 1819-1828, **(1999**).

[2]. F. Massines, A. Rabehi, P. Decomps, R. Ben Gadri, P. Ségur, C. Mayoux, Journal of Applied Physic, Vol. 83, No. 6, 2950-2957 (**1998**).

[3]. American Association of textile chemists and colorists, AATCC, technical manual, research Triangle park, USA, 75(2000).

[4]. D.B Judd and G. Wyszecki, Color in business science and industry ,3 rd edn., John Wiley Sons, Cleveland, USA,(1975).

[5]. Garamoon,A.A., and El-zeer,D.M., Plasma Sources Sci. Technol. Vol. **18**, no. **3, pp.** 194–201,(2009).

[6]. F. Massines, G. Gouda, J. Phys. D: Appl. Phys. Vol. 31, pp. 3411–3420, (**1998**).

[7]. D.M. El-Zeer, N. Dawood, F. Elakshar, and A.A. Garamoon, 'The influence of the addition of argon gas to air DB discharge', Eur. Phys. J. Appl. Phys., vo. 58, no. 30801, (2012).

[8]. I.A. Kossyi, A.Y. Kostinsky, A.A. Matveyev, V.P. Silakov, Plasma Sources Sci. Technol. 1, 207 (1992).

[9]. Onar N., Sarıışık M. Use of Enzymes and Chitosan Biopolymer in Wool Dyeing Fibers & Textiles in Eastern Europe 2005;13(1) 54-59.

[10]. C. Canal, R. Molina, E. Bertran, P. Erra,' Polysiloxane Softener Coatings on Macromol. Macromol.Plasma-Treated Wool: Study of the Surface Interactions', Macromol. Mater. Eng. 2007, vol. 292, 817–824.

[11]. B P Saville, Physical testing of textiles, WOODHEAD PUBLISHING LIMITED Cambridge England,2000,page 173-174.

[12]. E. M. El-Khatib, W. M. Raslan, A.A. El-Halwagy and S. Galab, 'Effect of Low Temperature Plasma Treatment on the Properties of Wool/Polyester Blend', RJTA Vol. 17 No. 1, pp. 124-132, (2013).