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Effect of Atmospheric pressure glow discharge plasma on the surface modification and the printing properties of Wool/polyamide blend

Doaa M. El-Zeer[1-2] and Asmaa A. Salem[3]

[1]Taif University, Faculty of Science and Education in Al-khurma (girls branch), Applied Phys. Dep., Al-khurma, KSA

[2]Center of plasma technology, Al-Azhar Univesity, Nasr City, Cairo, Egypt

[3]Faculty of Applied Arts , Textile Printing Dyeing and Finishing department, Helwan University, Egypt

ABSTRACT: In this study, atmospheric pressure glow discharge plasma was used to modify the surface properties of wool/polyamide fabric, the effect of air plasma treatment on the printing properties of wool/polyamide blend was also discussed. Three dyes were used namely acid dye, basic dye, and reactive dye. Different exposure time and discharge current of air plasma treatment were investigated to impart changes in wool/polyamide properties, such as whiteness, wettability, tensile strength, elongation, felting shrinkage, color intensity, and fastness properties. The surface characterization was performed using FTIR and SEM analysis. The plasma treatments enhance the colour strength of the wool/polyamide blend with acid, basic, and reactive dyestuffs as well as the fastness properties, and represent an approach to printing the blend with single dye.

KEYWORDS: Atmospheric pressure, plasma, printing properties, Wool/polyamide blend.

1 INTRODUCTION

Plasma technologies play a vital role in materials processing especially in textiles treatment and biomedical industries. Chemically reactive plasma discharges are widely used for etching, thin film deposition, surface activation and bio-sterilization. Recently, there has been increased interest in using atmospheric pressure plasmas for textiles processing[(1)]. These plasmas do not require vacuum systems and are suitable for continuous in-line processing,. Plasma surface treatment used to modifying the functional properties of fibers possesses advantages in comparison with traditional techniques[(2)]. Plasma includes less water usage and energy consumption, with a very small fibre damage, then making plasma process very attractive. It will be used to enhance the quality of textile products in fabric preparation and in dyeing and finishing methods. As the plasma is a medium that contains ions and excited atoms besides free radicals with free electrons and photons and ultraviolet waves, all these conditions help in activation the surface of the treated textile.

Wool/polyamide is a very popular blend for woven apparel and carpets. It can show the complementary properties compared to pure polyamide or wool fibers in terms of crease recovery, durability, abrasion resistance, fast drying, and dimensional stability. Wool/polyamide blends could be printed with disperse dyes in conjunction with another class of dyes, but background staining of the fabric and poor fastness properties frequently occurs[(3)]. The use of mixtures of different types of dye can introduce difficulties in colour matching, particularly when blend properties are varied [(4)]. Acid, metal complex, reactive and basic dyes, these dyes are all theoretically suitable for printing wool/polyamide blends, application is mainly limited to acid and metal complex dyes. Acid dyes must be selected to obtain acceptable light and wet fastness for each end use, in addition to the desired brilliance of hue[(5)].

It is necessary to use metal complex dyes to get very high fastness properties, but these dyes usually produce dull prints on polyamide fiber[(3)]. Basic dyes can provide extraordinary brilliance in some cases, but do not show adequate fastness, when selecting reactive dyes, attention must be paid to staining of the ground during washing off[5].Different studies have therefore been made to improve colour fastness of anionic dyes on both polyamide [6-14]and wool[15-20] fabrics, as well as wool/polyamide blends[4,21-22].

Atmospheric pressure plasma techniques have been investigated and used for wool[23-30] and polyamide[31-41] surface modifications by several researches, but only a few concern with wool/polyamide blends[42-43].

In the present study, Atmospheric pressure glow discharge plasma is applied on wool/polyamide blend fabrics to improve their physical properties concerning on enhancing their printability with acid, reactive and basic dyes to impart maximum fixation of dyes as well as colour strength.

2 Experimental Details

2.1 Plasma Set up

A glow discharge generator was employed for the plasma treatment of the wool/ polyamide. The generator that is shown in Fig. (1) consists of two parallel steel electrodes, of diameter about 20 cm, covered with two porous alumina sheets of thickness 2 mm as hold as dielectric barrier. It was proved that the porosity of the barriers are very important for obtaining glow discharge plasma rather than obtaining the filamentary discharge plasma[44]. The discharge plasma generator is operated in open air under atmospheric pressure and powered by AC source of frequency 50 Hz. The textile is placed in the gap between the two dielectric barriers as shown in Fig. (1).

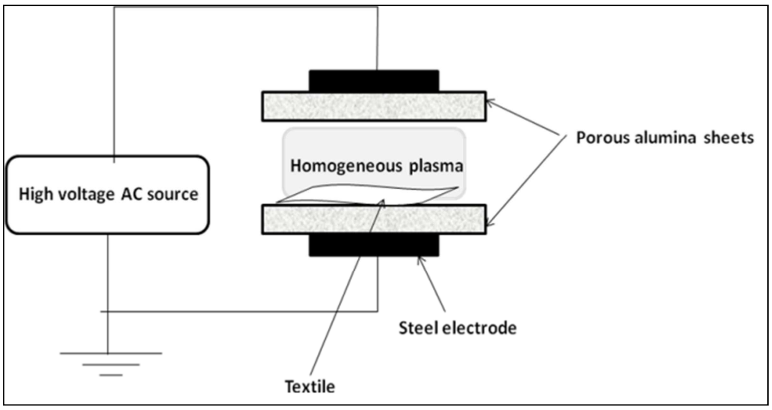

Fig. 1. Schematic diagram of the plasma generator

The consumed power of the plasma generator doesn't exceed tens of watts. In order to calculate the consumed power in the plasma generator, Voltage –current waveforms should be plotted as shown in Figure (2). From the figure (2) it can be noticed that the current represents relatively smooth humps of duration time about 5 m sec that is the evidence for obtaining the homogenous glow discharge plasma. There was small filaments that are superimposed on the glow component of the current however, the peak and the duration of these filament is relatively small and don't affect the behavior of the discharge and also cannot harm the textile. Also it can be noticed that the discharge current increased by increasing the applied voltage between the electrodes.

The reactor mean power has been evaluated by plotting the voltage versus the current the result is parallelepiped shape called Lissajous figure. The area of Lissajous figure is directly proportional to the consumed energy per one cycle. The average consumed discharge power for the plasma reactor at different applied voltages is shown in the Table (1). From the table it can be stated that the plasma reactor don't consume large power.

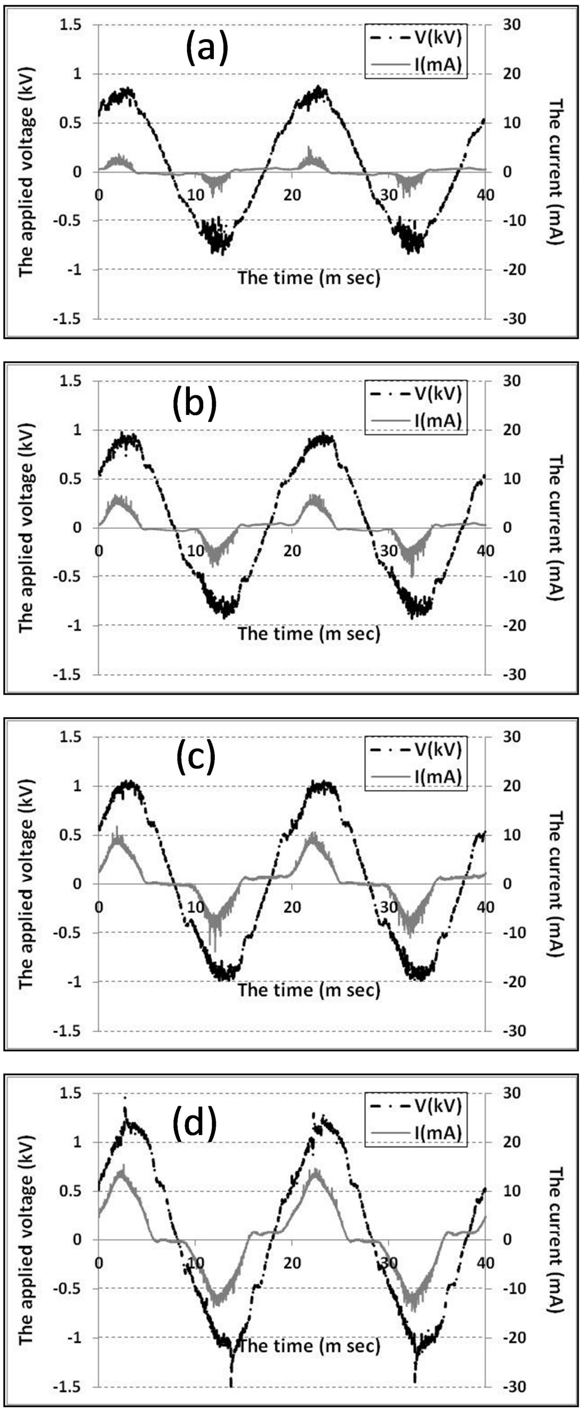

Fig. 2 I-V wave forms at different applied voltage

Table 1 The consumed power of the glow plasma reactor at different applied voltage.

Voltage (V)	Power (Watt)
650	0.088
690	0.25
720	0.41
740	0.55
780	0.88
860	1.8
950	2.5

2.2 MATERIALS

Substrate

wool/polyamide 20/80, mill scoured fabric weigth of square meter 240g/m^{2}, supplied by Golden Tex Company, Egypt.

Dyestuffs

Three different types of dyes were selected and used namely: Setazol S2B (Setazol Kimya Co., reactive dye), Ginacryl Red Violet 3RN (basic dye), Suncid Blue N-RH, C.I. Acid Blue 260, supplied from ICI Co., Egypt.

Thickening agent

Mepro gum T-8 supplied by Danisco Company, Switzerland, and Guar gum, supplied by Morgan Co. Egypt. were used as thickening agents.

Auxiliaries

trichloroacetic acid, was supplied by Riedel Haen (Germany), acetic acid, citric acid, benzyl alcohol, urea, thiourea, and ammonium sulfate, all reagents were commercial grade of purity, supplied by Al-Nasr Company, Egypt.

Scoural CA, non ionic detergent, supplied by Daico Co., Egypt, was used for washing off process for the printed samples.

-Kieralon AE 3D, (BASF),non-ionic wetting agent.

2.3 PRINTING

Samples were printed after plasma treatment by using the recipes as shown below:

Recipe (1):

30 gm basic dye (Ginacryl Red Violet 3RN 200%)

20 gm benzyl alcohol

15 gm acetic acid 30%

500 gm mepro gum

5 gm citric acd

X gm water

1000 gm

The prints were then air dried and steamed at 105°C for 30 min.

Recipe (2)[4]:

30 gm reactive dye (Setazol Red S2B)

3 gm trichloroacetic acid

500 gm meypro gum T-8

50 gm urea

10 gm non-ionic wetting agent

X gm water

1000 gm

The prints were then air dried and steamed at 120°C for 15 min.

Recipe (3):

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

The prints were then air dried and steamed at 105°C for 30 min.

Washing off

The prints were washed with cold water and soaped at 60ºC for 15 minutes with 2g/l Scoural CA, then rinsed with hot and cold water and air dried.

2.4 Measurements

2.4.1 Whiteness and color strength

The colour strength (K/S) and the degree of whiteness of the printed samples were 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.[45]

2.4.2 Wettability

water-drop test was applied according to AATCC test method 39-1980[46]. The time required for the drop of water to be absorbed into the fabric will be referred to as absorbency values.

2.4.3 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.

2.4.4 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.

2.4.5 Tensile Strength

The tensile strength test was carried out according to the ASTM standard test method D1294-95a[47]. On a tensile strength apparatus (model H5KT, Tinius Olsen Company).

2.4.6 FELTING SHRINKAGE

A felting shrinkage test was carried out according to

the ISO/FDIS 6330 method (IWTO-20-69, 2000) glow plasma treated and untreated fabrics.

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.

2.4.7 FASTNESS PROPERTIES

The colour fastness to washing, crocking, and perspiration, were determined according to the AATCC test method 61-1996, AATCC test method 8-1996, and AATCC test method 15-1997[46] respectively.

3 RESULTS AND DISCUSSION

3.1 THE TEXTILE SURFACE PROPERTIES

3.1.1 WHITENESS

The whiteness measurements as a function of the treatment time at discharge currents I=5 and 10 mA, are shown in figure (3).

It is obvious that there is a significant improvement in the whiteness degree for plasma treated samples as it compared by the untreated sample. This enhancement in whiteness increases by increasing the treatment time at discharge current 5, this behavior is attributed to the cleaning effect of plasma on the surface of wool/polyamide fabric from any contaminations by the bombardment of the ions and the excited species that are formed inside the plasma reactor. While by using 10 mA discharge current, the whiteness decreases gradually by increasing treatment time, but the values are still higher than untreated sample, higher discharge current and longer exposure time during plasma treatment may be leads to oxidation of disulphide linkage (i.e. systine bond) in wool structure[48] result in increase yellowing of blend fabric.

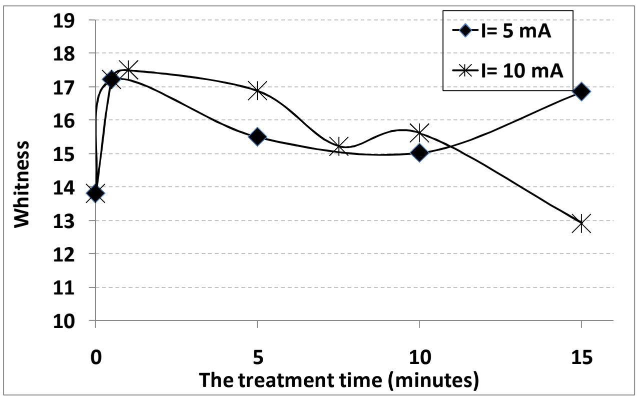

Fig.3 Effect of air plasma treatment on whiteness index of wool/polyamide fabric in relation to discharge current and exposure time.

Furthermore, this behavior may be due to increase of the surface roughness , since reflection of light of the rough surface is less than smooth ones[49].

Also the improving in the whiteness increase by increasing the discharge current i.e 5-12 mA for 10 min. as it is shown in figure (4).

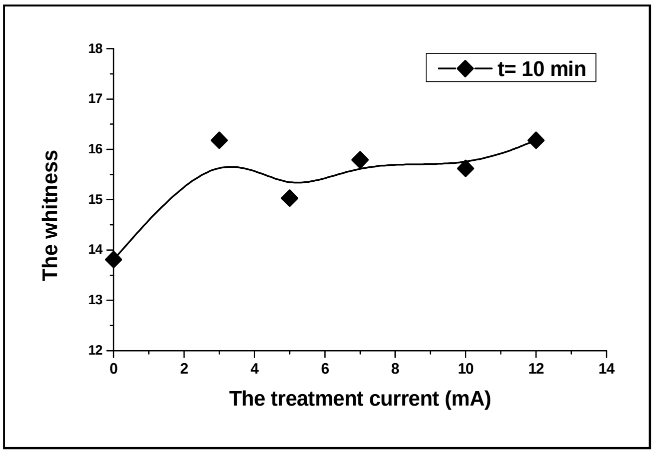

Fig.4 Effect of air plasma treatment on whiteness index of wool/polyamide fabric as a function of discharge current.

3.1.2 Scanning electron microscope (SEM)

The SEM images of untreated wool/polyamide fabric and plasma treated fabric are shown in fig.(5). The change in the surface morphology of plasma treated wool/polyamide fabric is shown in figs. (b, c), the etching and roughening effect caused by the active species bombardment of air plasma on the surface of polyamide fiber in the blend is observed in fig. (b), also remove the scales of the cuticle cell by the etching effect of plasma makes the wool fibers look more smooth than untreated one. It can be noticed from fig. (c), that the cuticle layer of treated wool is partially removed and visible holes and cracks are formed.

3.1.3 FTIR analysis

Surface chemical modifications induced by air plasma treatment of the wool/polyamide fabric are determined by an FTIR analysis. Figure (4) shows the FTIR spectra obtained for the untreated (a) and air plasma treated blend fabrics (b). In the spectrum of the air plasma treated fabric (b), some new absorption band with peak intensity at 1062.82 cm^{-1}, characteristic of –COOH carboxylic acid group, 1234.30 cm^{-1}, characteristic of asymmetric –C=O stretching vibration, and 3635.66 cm^{-1}, characteristic of primary alcohol OH stretch groups are observed. This result indicates that an oxidation process in the treated sample is occurred, and this attributed to the fast reaction between the radical ions and the fabric surface with air plasma treatment. So air plasma treatment is considers a high efficient process in incorporating oxygen on the fabric surface(50).In addition, a band with beak intensity at 3307.23 cm^{-1}, characteristic of aliphatic secondary amine NH stretch group, and the absorption at 1392.27 cm^{-1}, which due to ammonium salt NH_4 group were also observed

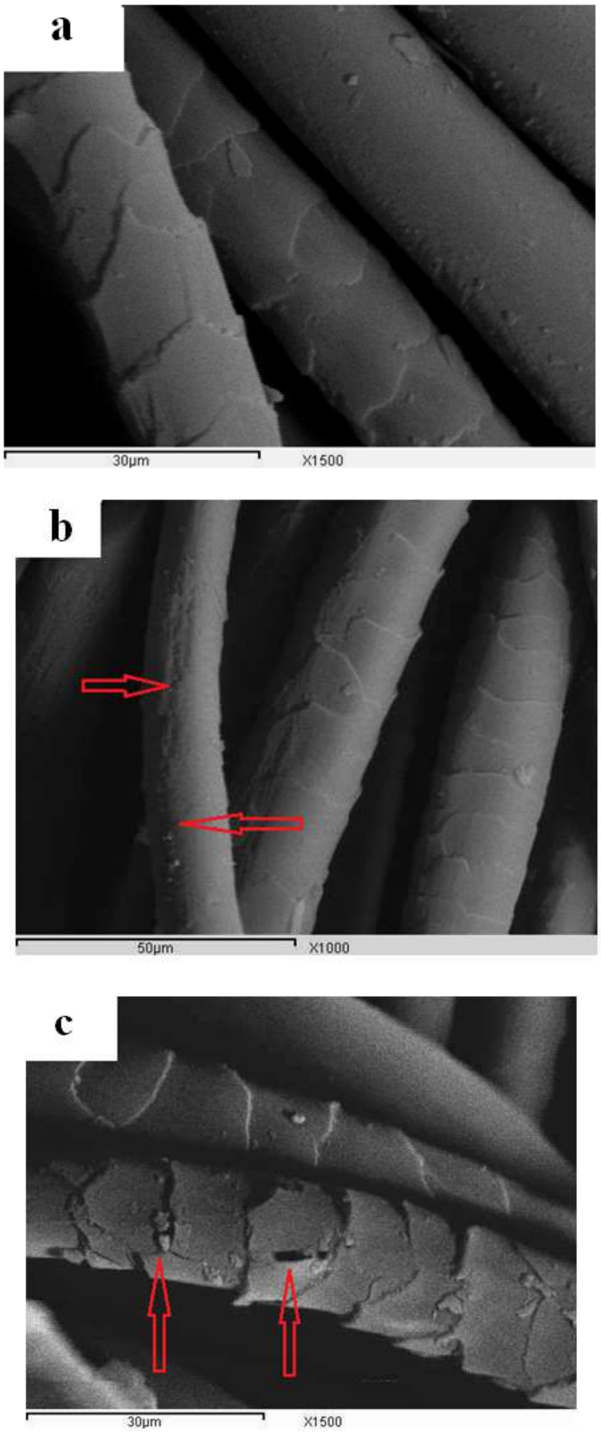

Fig.5 SEM pictures of (a) untreated, (b, c) air plasma treated wool/polyamide samples at 10 mA for 10 min.

3.1.4 Tensile strength and Elongation

Tensile strength and elongation to break of the wool/polyamide blend before and after plasma treatments are given in table (2) one can notice from the results that plasma treatment causes a slight decrease in the average tensile strength as compared to the untreated samples . This decrease is believed to be related closely to the inter- fiber inter- yarn frictional forces induce by the plasma treatment as reported by Yip,J.&el.[51] Both tensile strength and elongation are found to be discharge current and exposure time dependent, the maximum decrease in the tensile strength does not exceed than 13.78% by using 10 mA current for 10 min. Ren,Y.&el [52] has reported that a long DBD plasma treatment generates deep cracks on the fiber surfaces leading to reduce the tensile strength of the fiber, furthermore, higher discharge current or longer treatment time during plasma treatment may lead to lose in tensile properties due to excessive etching[53].

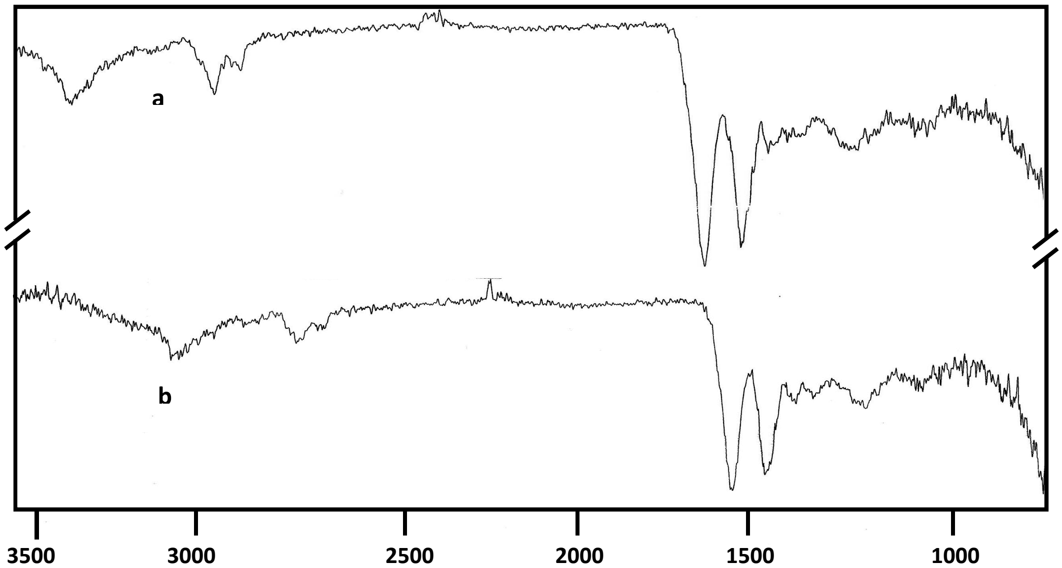

Fig.6 IR spectra of woo/polyamide fabric, (a) untreated, (b) air plasma treated fabric for 10 min., at 10 mA.

3.1.5 SHRINK RESISTANCE

Wool/polyamide blends fabrics show much lower shrinkage values than wool, due to the presence of polyamide in the yarn, the results shown in table (2) reveal that plasma treatments under different conditions of the treatment time and discharge current reduce the area shrinkage of wool/polyamide blend to about 1% with respect to untreated sample 2.5%, the shrink resistance effect induced by the DBD plasma is attributed to changes in the wool surface, such as progressive removal of fatty acids covalently bonded to the outermost surface of the fiber (Fatty layer)[54]and etching off the exocuticle that contains the disulfide linkages which increase cross linking and contribute towards shrinkage[55]. These changes could result in a reduction in the differential friction effect of the fibers and, hence, in a decrease of the natural tendency of wool to shrinkage.

3.1.6 WETTABILITY

The effect o plasma treatment on the wettability of wool/polyamide blend expressed by wetting time is investigated. The results are illustrated in table (2), it can be seen that the wetting time decreases with increasing the discharge power and also by increasing plasma treatment time. This improves in the wettability is attributed to the ion bombardment on the fabric surface which leads to formation of surface-free radicals and increase in the amount of active species formed on the surface[1]. The plasma also enhances the wetting properties by etching off the hydrophobic epicuticle from the wool surface, as well as introducing surface polar groups such as (-COH,-COO,-C=O,NH) as it is evident from FTIR analysis, in addition etching process increase the grooves on the fiber surface and hence enhance the wettability of fabrics[48].

Table 2 The physical properties of untreated and plasma treated wool/polyamide blend at different conditions of the current and the treatment time.

Condition of plasma treatment	Wettability (Sec.)	Tensile Strength of Wool fabric Kg/mm2	Elongation To Break %	Area Shrinkage %
Untreated	366.6	46.79	28.69	2.49
5 mA-7.5 min.	307.2	45.41	27.92	1.99
5 mA-10 min.	258.6	44.46	26.87	1.49
10 mA-7.5 min.	166.8	41.87	25.80	0.99
10 mA-10 min.	148.8	40.34	24.44	1.00

3.2 PRINTING PROPERTIES

3.2.1 COLOR STRENGTH

Figures 7-9 represent the effect of exposure time to the air plasma treatment at different discharge curents on the colour strength of treated wool/polyamide fabric printed with acid, basic, and reactive dyes respectively. The obtained results indicate that a significant increment in colour strength of plasma treated fabric was occurred, irrespective of type of dye used, this increase in the colour strength could be due to the following reasons: plasma treatments have a significant influence on the surface morphology and result in unsaturated and/or free radicals on the fibre surface and consequently enhance the fibre dyeability and printability. On the other side, plasma etching might open up newly accessible domains for the diffusion of dye molecules, which results in increased dyeability(56).

Printing of plasma pretreated wool/polyamide blend with acid dye:

The effect of air plasma treatment on the colour strength of wool/polyamide fabric printed with acid dye is presented in figure 7, It can be noticed that the colour strength increase by increasing the exposure time for both current used 5 mA, and 10 mA, this increase in the colour strength may be is due to the oxidation of the disulfide bonds and converts cysteine moieties in the lipid layer to cysteic acid (57,58,59). And thus introduce NH_2 groups into the fibre, which reflects an increasing absorption of anionic dyes (acid dye), on the other hand, etching of the epicuticle layer by plasma, the partial removal of wool surface scales and the reduction in the amount of covalently bonded fatty acids, lead to an increase in the hydrophilicty and hence the printability of the fabrics.

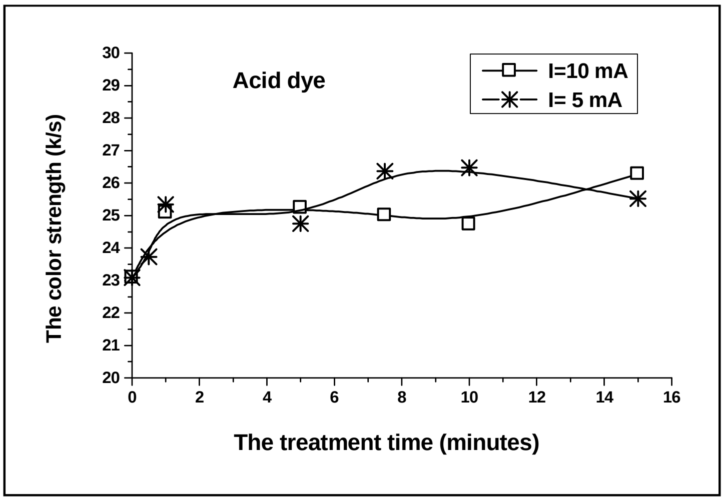

Fig.7 Effect of air plasma treatment time on the colour strength of wool/polyamide fabric printed with acid dye.

Printing of plasma pretreated wool/polyamide fabric with basic dye:

Figure 8 represent the relation between plasma exposure time and the colour strength of untreated and plasma treated wool/polyamide fabric printed with basic dye.

As in the case of acid dye, the K/S values increase as the exposure time of plasma treatment increases until it reaches 29.76% for 15 min. exposure by using 10 mA, these results can be explained in terms of wool surface modification, that is, an increase in the cysteic acid content of the wool fabric surface and thus increase sulphonate and carboxylic acid groups which act as active sites for basic dyes[60]. On the other side, plasma treatment cause the breaking of the chain molecules of polyamide, resulting an increase of carboxyl and amine end groups, the amino and carboxylic acid groups will be ionized, aionic attraction operating between basic dye and carboxylic acid groups of polyamide, also similar interaction observed between anionic acid dye and amino end groups of polyamide.

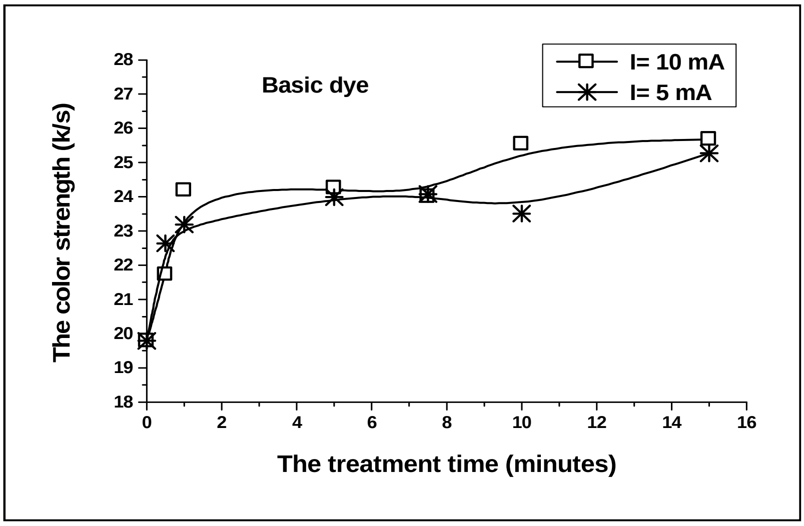

Fig.8 Effect of air plasma treatment time on the colour strength of wool/polyamide fabric printed with basic dye.

Printing of plasma pretreated wool/polyamide fabric with reactive dye:

The impact of plasma treatment time on the colour strength of wool/polyamide fabric printed with reactive dye is illustrated in figure 9, Similar to acid and basic dyes plasma treated wool/polyamide fabric printed with reactive dye gave higher K/S values than that of the untreated sample. This increase in the K/S values is correlated with the increase in exposure time of plasma treatment. This results may be attributed to the effect of air plasma treatment on increase the dye sites on the wool/polyamide fabric surface, which react with the dye and produce a variety of oxygenated functional groups[59], such as C=O, -OH and –OOH. Theses groups enhance the dye fibre interaction between the reactive dye sites and the wool/polyamide fabric by forming covalent ionic and hydrogen bonds.

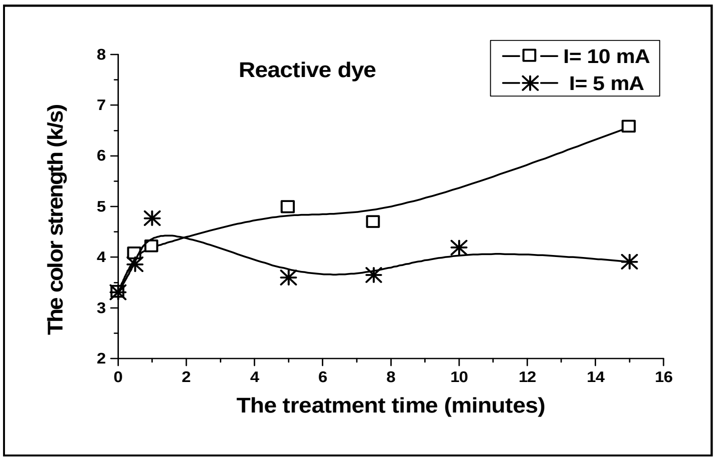

Fig.9 Effect of air plasma treatment time on the colour strength of wool/polyamide fabric printed with reactive dye.

The effect of discharge current of plasma treatment on the colour strength of treated wool/polyamide fabric printed with acid, basic, and reactive dyes is shown in figure 10, it can be concluded from the results that on using reactive dye there was a significant increase in the K/S value in comparison with acid, and basic dyes for the same discharge current, for example, the increase in the K/S for the sample treated with 7 mA for 10 min. was 14.59 %, 23.09 %, and 80.36 % for the samples printed with acid, basic, and reactive dyes respectively.

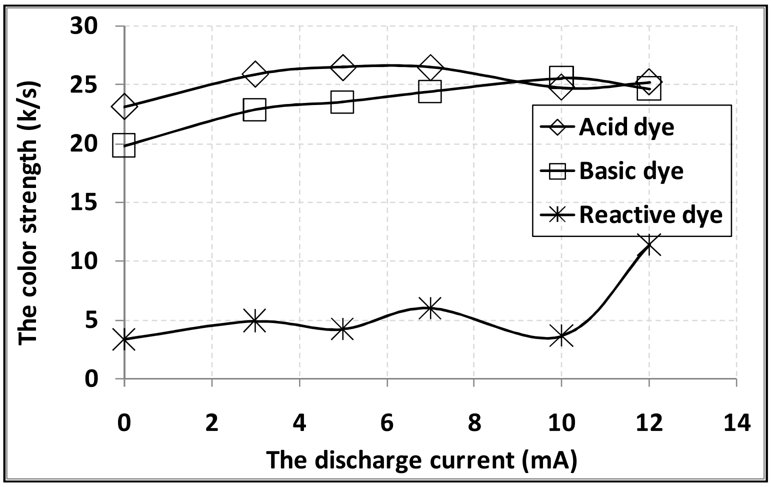

Fig.10 Effect of plasma discharge current on the colour strength of wool/polyamide fabric printed with acid, basic and reactive dyes.

3.2.2 FASTNESS PROPERTIES

The improvement in the colour fastness of the plasma treated and printed fabrics in comparison with the untreated ones is shown in table 3. It was observed that plasma treatment improves the washing and perspiration fastness of wool/polyamide fabric printed with acid, basic, and reactive dyes. Also the rubbing fastness show some enhancing after plasma treatment by using all the dyes under the investigation.

Table 3 Fastness properties of untreated and plasma treated wool/polyamide fabric under different conditions

Dyestuffs	Condition of plasma treatment	Wash Fastness			Rubbing Fastness		Perspiration Fastness					
							Alkaline			Acidic		
		Alt.	St.		Dry	Wet	Alt.	St.		Alt.	St.	
			Cotton	Wool				Cotton	Wool		Cotton	Wool
Acid dye	Untreated	5	3-4	3-4	4	2-3	4-5	3-4	4	4-5	4	4-5
	5 mA-7.5 min.	5	4	4-5	4-5	3	4-5	4	4-5	4-5	4-5	4-5
	5 mA-10 min.	5	4	4-5	4-5	3	4-5	4	4-5	4-5	4-5	4-5
	10 mA-7.5 min.	5	4	4	4-5	3	4-5	4	4-5	4-5	4-5	4-5
	10 mA-10 min.	5	4	4	4-5	3	4-5	4	4-5	4-5	4-5	4-5
Basic dye	Untreated	5	3-4	3-4	3-4	3	4-5	3	4	4-5	4	4-5
	5 mA-7.5 min.	5	4	4	4	3-4	4-5	3-4	4	4-5	4-5	4-5
	5 mA-10 min.	5	4	4	4	3-4	4-5	3-4	4	4-5	4-5	4-5
	10 mA-7.5 min.	5	4	4	4	3-4	4-5	3-4	4	4-5	4-5	4-5
	10 mA-10 min.	5	4	4	4	3-4	4-5	3-4	4	4-5	4-5	4-5
Reactive dye	Untreated	5	4-5	4-5	5	4	4-5	4	4-5	4-5	4-5	4-5
	5 mA-7.5 min.	5	5	5	5	4	4-5	4-5	4-5	4-5	5	4-5
	5 mA-10 min.	5	5	5	5	4-5	4-5	4-5	4-5	4-5	5	4-5
	10 mA-7.5 min.	5	5	5	5	4	4-5	4-5	4-5	4-5	5	4-5
	10 mA-10 min.	5	5	5	5	4	4-5	4-5	4-5	4-5	5	4-5

4 CONCLUSION

The influence of atmospheric pressure plasma treatment on the physical and printing properties of wool/polyamide blend fabric by using three dyestuffs; acid, basic, and reactive dyes, is studied. The results revealed that the plasma treatment was an effective technique to increase wettability, whiteness, colour strength of printed wool/polyamide fabric, these increase are discharge current and time exposure dependent. The results of SEM images provided a visual evidence that the plasma treatment can improve the wettability of wool/polyamide fabric because the surface etching and roughening effect, and partial removal of scales on wool fiber in the blend, The FTIR spectre indicated the presence of oxygen-containing functional groups and aliphatic secondary amine group, which enhance the dye fiber interaction. Also the tensile strength and elongation at break of the treated fabrics have slightly decreased, not exceed than 13.76%. Additionally , The fastness properties of treated wool/polyamide printed fabrics are enhanced irrespective of the dyes used under the investigation.

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