



## AFM and SEM Analysis of Polystyrene Surface Treated by DC Pseudo Plasma Discharge

D. Moubarak<sup>1</sup>, M. Abd Al-Halim<sup>2</sup>, A. Abu-Hashem<sup>2</sup> and Y.H. Elbashar<sup>3\*</sup>

<sup>1</sup>Thebes High Institute of Engineering, Cairo 11434, Egypt.

<sup>2</sup>Physics Department, Faculty of Science, Benha University, Qalyubia, Egypt.

<sup>3</sup>Egypt Nanotechnology Center ((EGNC)), Cairo University, Giza, Egypt.



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**I**N THIS work, the surface treatment of polystyrene using the DC Pseudo-Plasma is studied. First, the DC Pseudo-plasma was employed as a function of plasma device parameters under different operating conditions including time (t) (30 s), current (I) (15mA), distance (d) (3 mm) between inter-electrodes, mesh anode transparency (T) (19%), distance between the polystyrene sample and the mesh anode (D) (2mm) and air pressure in plasma exposing system (P) (2 torr). The best optimization of these parameters were performed by the water absorbency, which supported by the Scanning Electron Microscope (SEM) test Atomic Force Microscope (AFM) test.

**Keywords:** DC pseudo plasma discharge, Polystyrene, Surface treatment.

### Introduction

Plasma is defined as , the ‘fourth state of matter’, is an electrically neutral ionized gas (quasi-neutral) and contains a significant number of free electrically charged particles, these free electrically charged particles make electrically conductive [1]. DC Pseudo Plasma Discharge is a special type of discharge in which the plasma is performed using a bulk cathode and mesh anode, where the discharge (plasma) takes place behind the mesh anode [2-4]. Cold (non-thermal) plasma is become one of common technique which used in surface modification of materials specially the textile surface as it is found that most textile materials are heat sensitive polymer [5-7]. In this type of plasma, the electrons temperature is much higher than ions temperature, these low-energy molecular species and high-energy electrons initiate reactions in the plasma volume without excessive heat causing substrate degradation. Textiles treatment using plasma technology is a significant method as it interact with the surface

of the textile only without modifying or affecting the bulk properties of the materials [8], so it’s wettability increased, which allows for solvent free dyes to absorb and bond very strongly, or to make the textile a hydrophobic fibre by coat the textile surface with a specialized layer with varying characteristics, this features of plasma treatment of textile is attributed to the free radicals, electrons and heavy particles inside plasma which contribute in surface modification [9]. When the textile surface exposed to the plasma an additional energy transfers from the plasma to allow for subsequent reactions to take place on the material surface [10], so the surface treatment using plasma rises the surface energy of the material to improve characteristics of the bonding [11]. These energy, which transfers from the plasma to the material, are dissipated within the solid by a assortment of physical and chemical processes to result in a unique type of surface modification that reacts with surfaces in depths from several hundred angstroms to 10 $\mu$ m without changing the bulk properties of the

\*Corresponding author e-mail: y\_elbashar@yahoo.com, Tel.: 01001141053

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material [12]. Moreover, it is considered to be anti-pollution technique and enhances the excess of micro roughness and production of radicals to obtain hydrophilic surfaces [13, 14]. Furthermore, it is economical and effective technique for many materials, which gets a scope attention in textile engineering [15]. Plasma treatments are attaining popularity in the industry of the textile for their plentiful advantages over traditional wet processing techniques. This technology shows innovative solutions to wetting and adhesion problems in many industries. The extremely crystalline structure of the polystyrene and its polarity lack which make it resist the absorbance of the water so it is classified as a hydrophobic fabric [16, 17]. The plasma treatments application for improving wettability of all possible fibre types was obtained with varied success degrees such a treatments on natural fibres like, cotton and wool, and on synthetic polymers helps to enhance their wetting properties [18].

### Experimental setup

Figure 1 shows the A schematic diagram of the experiment setup. The DC pseudo discharge plasma system consists of a 20 cm length Pyrex

tube, 5.8 cm outer diameter and 5.6 cm inner diameter. Two aluminum plates are used to maintain the Pyrex tube at its terminals with mesh anode and plate cathode, which are made of stainless steel, are enclosed inside the discharge tube. The cathode is a plane circular disk of 5.5 cm in diameter. The anode is a movable circular mesh of 5.5 cm in diameter (with different mesh transparency). The transparencies of the mesh anode are determined from the mesh wire diameters and the separation between them. The distance of the anode place in front of the cathode is chosen to be 3 mm as it is the intermediate distance we could adjust between the two electrodes. The transparencies of the anode are chosen in this work to be (19 %).

Air is taken as the working gas. A rotary pump with double stage is used to evacuate the discharge tube. A needle valve connected to the discharge cell to control the rate of flow. To apply a potential difference up to 1 kV and a current up to 250 mA a DC power supply is used, where the a rheostat of 6 k $\Omega$  is used to control the value of the current. Plasma produced by this system has properties of temperature of 2.6 eV and density of  $2.3 \times 10^{14} \text{ m}^{-3}$ .

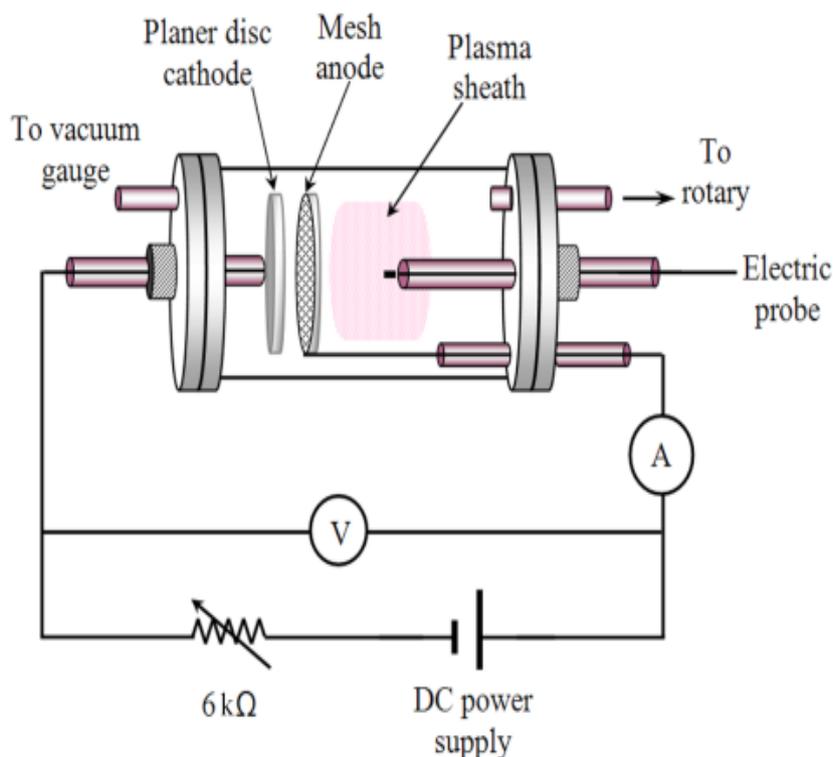


Fig.1 Schematic view of the discharge system.

### Materials used

Polystyrene samples 5.5 cm in diameter were cut from a large piece, which are maintained by a hollow cylindrical of metal, which can be introduced behind the anode at desirable distances. The polystyrene is provided from Egyptian Styrene & Polystyrene Production Company; 6 El Nabawy El Mohandis Street; East Gas Building; Nasr City; Cairo, 11211 Egypt ([www.estyrenics.com](http://www.estyrenics.com)).

### Testing and Analysis

The treated samples were undergoing many tests to know the changes of their surface after plasma treatment. The measurements are carried out on the polymer samples, the water absorbency, SEM and AFM test.

The experimental parameters which considered in the treatment are the discharge current (I), the gas pressure (P), the duration time of treatment (t), the separation distance between the two electrodes (d), the separation distance between the mesh anode and the polymer sample (D), and the mesh transparency (T). The standard considered conditions are at P=2 torr, I=15 mA, D=3 mm, T=19% and  $t_{ex}=30$  s.

### The water absorbency test

Figure (2) shows the shape of a drop of water on a treated and untreated sample of polystyrene. It's observed that the contact angle for the treated polystyrene sample gets smaller than that for the untreated one. This means that the ability of absorbing water increases by exposing the polymer to the plasma.

Moreover, that the surface tension of the water drop is high on the surface of untreated sample, because the water is not absorbed by the polystyrene surface, so it adheres weakly to the polystyrene surface and strongly to itself, while for the treated sample, the water is absorbed by the polystyrene surface so the adheres of the water drop to the polymer surface is higher than the untreated sample.

Figure (3) shows the effect of the plasma exposure time on the water absorption for polyester samples. It is observed that the relation between two times is inversely proportion. For longer plasma duration time, the surface of the sample has more ability to interact with plasma so the treatment is better hence the time of the water absorption get lower which means the wettability is higher.

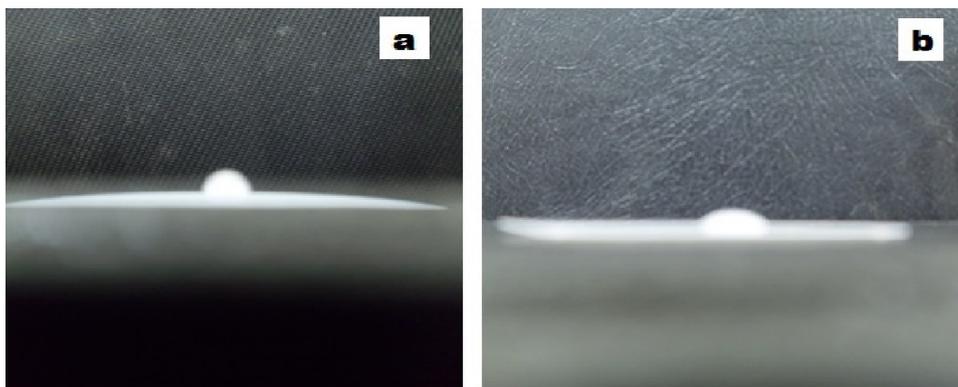


Fig.2. the drop of water on a polystyrene samples (a) untreated sample (b) treated sample.

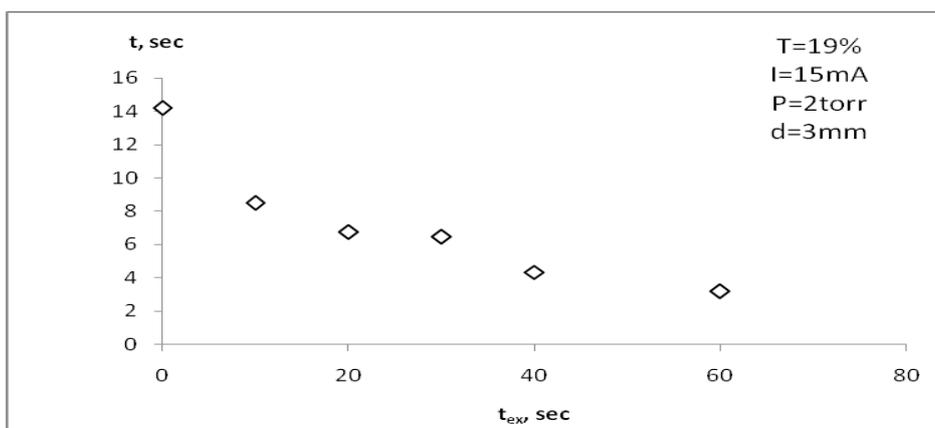


Fig. 3. Effect of the exposure time on the water absorption of a polystyrene fabric using DC pseudo discharge for air.

### The tests of Morphology

#### a- Scanning electron microscope (SEM) test

Figure 4 shows the SEM of the polystyrene samples. Images; (a) is the untreated sample while (b) is the treated sample. It is observed from Fig. (4) The exposing the polystyrene materials to the plasma, the polymer surface seem suffering more roughness due to interact between the plasma species and the sample surface which cause etching, so its wettability increases. This behavior is supported by the AFM test as shown in next topic.

#### b- Atomic Force Microscope (AFM) test

AFM test is used to detect the effect of the DC pseudo discharge plasma on the polymer surface morphology. Figure 5 shows the AFM images for two samples of polystyrene (a) untreated sample, and (b) treated sample. AFM test measures the surface roughness of the treated and untreated polystyrene samples.

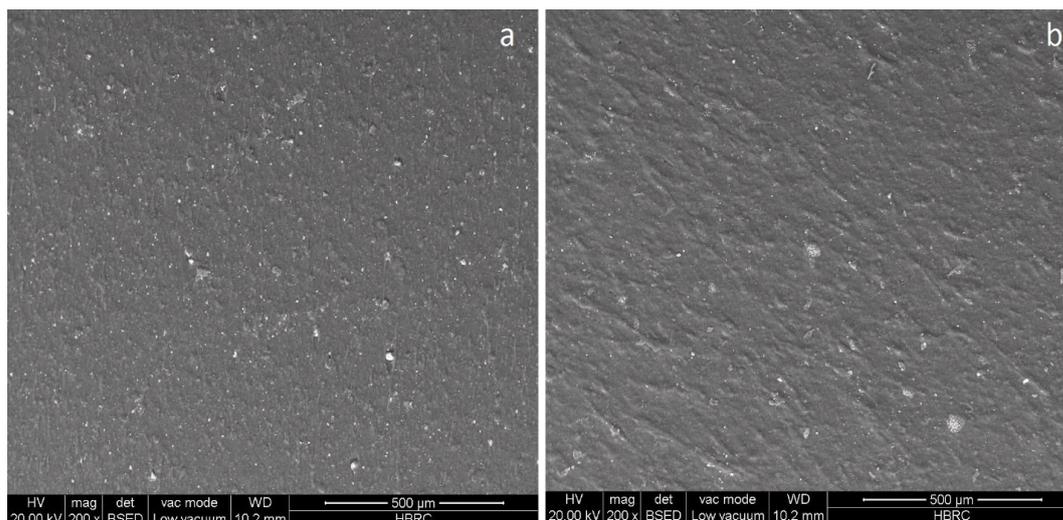


Fig.4. SEM images of polystyrene samples at different distances (a) untreated sample (b) treated sample.

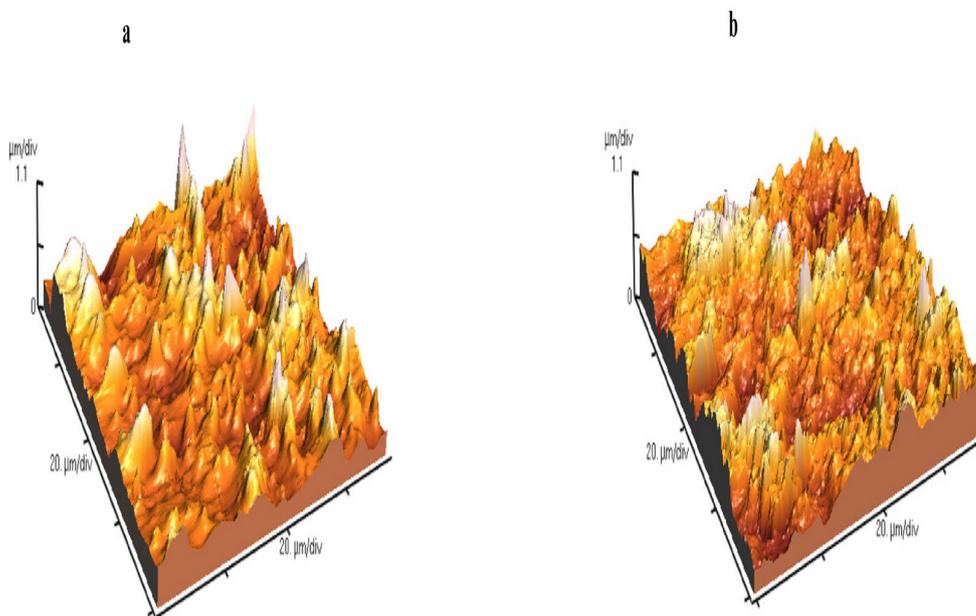


Fig. 5. AFM images of the surface morphology of polystyrene samples (a) untreated sample (b) plasma treated sample.

The untreated polystyrene sample has a 202.7 nm roughness, while the surface of the treated sample has a higher roughness of 211.5 nm. Therefore, it is found that by exposing the polystyrene materials to the plasma, their surface suffering from more roughness morphology due to the interaction between the plasma species and the molecules of the surface of the sample, which causes surface etching and the wettability, increases.

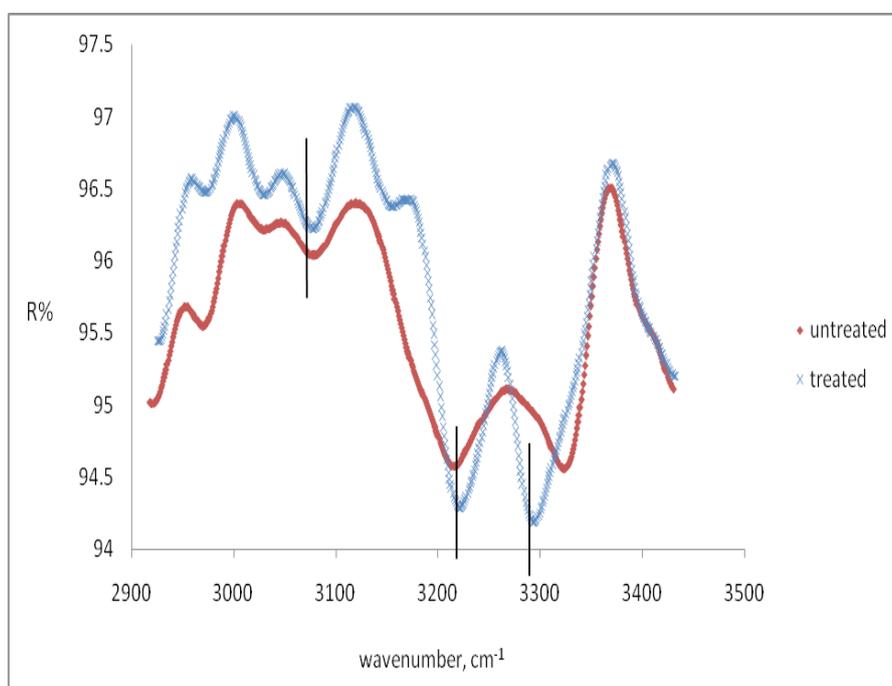
Table 1 shows the difference in the properties measured by the AFM between the untreated and treated samples. It is observed that the treated sample has higher values for both the root mean squar roughness, the average roughness, the mean hight, the median hight, the deep valley, and the surface area than those of the untreated sample.

**TABLE 1. AFM test results for the treated and untreated polystyrene samples.**

Property measured by AFM	Untreated Sample	Treated Sample
Projected Area	5625 $\mu\text{m}^2$	5625 $\mu\text{m}^2$
Rms Rough (Rq)	261.3 nm	271.7 nm
Ave Rough (Ra)	202.7 nm	211.5 nm
Mean Ht	602.2 nm	791.2 nm
Peak (Rp)	1.603 $\mu\text{m}$	1.369 $\mu\text{m}$
Valley (Rv)	-602.2 nm	-791.2 nm

The IR analysis: Infrared test is one of the important analyzing tests in this work, as it can show the bonds, which are broken and new bonds which are created at the surface of the polymer and support the AFM and SEM analysis. The treated and untreated polymer samples were tested by the IR test at the national institute for standards in Giza – Cairo.

Figure 6 shows the IR test for untreated and treated samples of polystyrene. It is observed from Fig. 6 that the hydrophilic groups have appeared on the surface of the treated polyester samples, this means that the ability to absorb water of the polyester surface is increased [93]. The O–H stretched group appears in Fig. 6 at the peaks of wave numbers of 3293.8  $\text{cm}^{-1}$ , 3221.5  $\text{cm}^{-1}$  and 3077.8  $\text{cm}^{-1}$ .



**Fig. 6. IR spectroscopy chart for treated and untreated polyester samples.**

## Conclusion

The wettability was characterized by the water drop test and the wettability was found to be increase by increasing the plasma current, The SEM and AFM tests showed more roughness in the polymer surface samples after treatment, which mean the ability of the polymer to absorb water, increased.

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## تحليل الميكروسكوب الماسح الإلكتروني و تحليل ميكروسكوب القوة الذرية لدراسة علاج سطح البولي استيرين باستخدام التفريغ الكهربائي الوهمي

ضياء إبراهيم مبارك<sup>١</sup> ، محمد عبدالحليم<sup>٢</sup> ، علاء أبو هاشم<sup>٢</sup> ويحي حمدي البشار<sup>٣</sup>

<sup>١</sup>معهد طبية العالي للهندسة – القاهرة – ١١٤٣٤ -مصر

<sup>٢</sup>قسم الفيزياء – كلية العلوم – جامعة بنها – القليوبية – مصر

<sup>٣</sup>مركز تكنولوجيا النانو – جامعة القاهرة – الجيزة - مصر

في هذا العمل تم دراسة علاج سطح البولي استيرين باستخدام التفريغ الكهربائي الوهمي. تم ضبط ظروف عمل جهاز البلازما لتكون زمن المعالجة ٣٠ ثانية؛ التيار ١٥ ملي أمبير؛ المسافة بين القطبين ٣مم؛ نفاذية الشبكة ١٩٪؛ المسافة بين الأنود والعينة ٢مم وضغط الغاز ٢تور. تم عمل إختبار إمتصاص الماء وتم دعم هذا الإختبار بتحليل الميكروسكوب الماسح الإلكتروني و تحليل ميكروسكوب القوة الذرية.