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DBD SYSTEM OPERATING IN AMBIENT AIR FOR SURFACE TREATMENT OF POLYETHYLENE TEREPHTHALATE FILMS

BY

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Abstract. Polymers are materials widely used in many important technologies and applications because of their optical, physical, mechanical, thermal and chemical properties. Polyethylene terephthalate (PET) foil is often used as an insulating material in printed electronics, flexible circuitry, displays, industrial applications, graphic films, or for clinical and healthcare applications. A big inconvenience of PET film is the low surface tension which also implies a low wettability, resulting in poor coating adhesion and poor printing properties. In order to eliminate this disadvantage, a surface treatment was used by means of a dielectric barrier discharge (DBD) operating in ambient air at atmospheric pressure which consisted of a high voltage AC source and two circular metal electrodes 60 mm in diameter. The paper aims are to treat two different PET film substrates designed in this work as PET 1, without thermal treatment, and PET 2 which is heat stabilized. The effect of plasma exposure was assessed by determining the water contact angle by means of an Ossila goniometer and by measuring the surface tension with a special ink. The considered foils were exposed to different DBD treatment times (0.2, 0.5, 1, 5, 10, 30 s) and the

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evolution of the water contact angle overtime after treatment up to 96 hours was monitored. It was found that the water contact angle of the untreated PET 1 and PET 2 was 74° and 73.5°, respectively, and after 30 s DBD treatment time it decreased to 30.9° and 30°, respectively.

Keywords: DBD plasma; PET surface; water contact angle; wettability; surface tension.

1. Introduction

Most of the visible matter in the universe is made up of plasma, such as the stars, the sun, which is a huge sphere of hot plasma, the solar wind, the incandescent core of the Earth, which is a liquid plasma, the ionosphere (Weltmann *et al*., 2019). Only a small part of the Universe is in solid state and a smaller part in liquid form. Plasma is found naturally on Earth under the form of lightning, polar aurora, fire, corona discharge observed during storms, or is produced by humans for technological purposes through a wide variety of materials and installations (Rycroft, 1986). Plasma, because it is an ionized state in which matter has no shape or volume of its own, responds to electromagnetic forces and emits electromagnetic radiation. Any solid body can pass, through heating, into the liquid phase and then into the gas phase. By further heating the gas obtained to quite high temperatures, it is ionized and by this way a new form appears: ionized gas or plasma in the gaseous state.

The first three states of matter are composed of a very large number of molecules (or atoms), heat representing the expression of molecular motion, in which atoms move as a single unit. In contrast, the plasma state is composed of a very large number of free electrons and ions, the heat being an expression of the separate movements between electrons and ions (Burm, 2012). A gas is normally an electrical insulator, in the sense that electric currents cannot pass through it easily. However, by heating the gas to the right temperature, it was found to be a good electrical conductor. Plasma will conduct electricity, like a copper wire, because free electrons can be moved easily.

The plasma can be sort by into two groups function of electron temperature or thermodynamic equilibrium: local thermodynamic equilibrium or thermal plasma and non-local thermodynamic equilibrium or non-thermal plasma (Tendero *et al*., 2006).

Sometimes, the non-thermal plasma, used for surface treatment, is generated by applying an electric field to a DBD reactor at atmospheric pressure in ambient air (Sihelník *et al*., 2020). Either DC excitation or AC excitation at a frequency range from low frequencies to a few GHz are applied to gas at ambient pressure. Three major reactions that occur in plasma generation are excitation, ionization, and dissociation. Any volume of gas contains a few charge carriers (electrons and ions). After applying the electric field between the electrodes these free charge carriers are accelerated and may collide with the gas atoms or with the electrodes surface. As a consequence, the excitation process enhances the movement energy of the gas atoms and transitions the internal energy to a higher state.

In non-thermal plasma, the temperatures of electrons, ions and neutrons are different. Usually, the temperature of electrons is much higher than that of other particles and can achieve temperatures of 10.000 K or more, (Vandenbroucke *et al*., 2011), while the temperature of the gas can be maintained at room temperature (Petitpas *et al*., 2007). Due to the low temperature of non-thermal plasma, it is able to treat thermolabile materials such as polymeric materials or ultra-thin flexible glass (Sihelník *et al*., 2021). Moreover, non-thermal plasma is easy to adapt to complex geometries. It is also used in applications in agriculture (Scholtz *et al*., 2019), food industry (Thirumdas *et al*., 2018), medicine (Plattfaut *et al*., 2021).

In order to treat polymeric surfaces, dielectric barrier discharge (DBD) in air at atmospheric pressure has proven effective in modifying a wide range of polymers: PET (Astanei *et al*., 2022), polycarbonate (PC) (Homola *et al*., 2017), polyurethane (PU) (Kostov *et al*., 2010), polypropylene (PP) (Matoušek *et al*., 2020), biaxially oriented polypropylene (BOPP) and low-density polyethylene (LDPE) (Cretu *et al*., 2021).

The DBD discharge is established in a gas gap between different electrode configurations, but with the presence of insulating material between them, such as glass, ceramic, quartz. The DBD type discharge can be driven by an AC voltage with a wide frequency band (50 Hz-1 MHz) and also by a pulsed voltage. In some configurations with a special arrangement of dielectric material, DBD reactors can also be driven by a DC voltage (Lu *et al*., 2016). The space between the electrodes is usually filled with gas or a mixture of gases and can range from a few millimeters to a few centimeters (Zeghioud *et al*., 2020). In contrast to the corona reactors, DBDs have several advantages such as low power consumption, stability for electric discharge, low operating costs and short processing time (Sasmazel *et al*., 2021).

PET film is widely used in industrial areas due to its good barrier properties, mechanical and thermal properties being recommended for electrical and electronic applications (Narakathu *et al*., 2015). But sometimes PET is an inappropriate material because the surface energy of this film is considerably low, its surface properties such as wettability, adhesion and printability do not always meet the requirements of specific applications. For this task, ambient air atmospheric plasma treatment is an ecological technology with good results, versatile and economically suitable (Cretu *et al*., 2021). Advances in DBD reactor technology have also made it possible to treat the entire polymer surface quickly, continuously and evenly (Van Dongen *et al*., 2013).

In this work, a DBD system operating in ambient air at atmospheric pressure has been used to modify the surface of two PET films (with and without heat stabilized) in order to improve the wettability measured by a goniometer which used sessile method for analyzing the static water contact angle and to increase the surface tension evaluated by means of a special ink from Arcotest.

2. Experimental Setup

This paper analyzed the effects of exposure to DBD treatment operating in ambient air on the surface of two different PET materials designed in experiments as PET 1 and PET 2. Polymer marked with PET 1 is represented by Melinex® 506 (thickness: 125 μm) which is a transparent polyester optical film, treated on both surfaces for good adhesion to the inks. It is commonly used in the graphics industry, where excellent clarity and printing adherence are used. Melinex® ST506 is a heat-stabilized polyester film designed as PET 2 (thickness: 125 μm). It is pretreated on both sides for good grip. It can be overprinted with a wide range of solvent-based inks and silver-conducting inks. It is ideal for graphic layers and certain circuits of membrane touch switches. We want to find out if the plasma treatment also has an effect on the film that has been heat treated. Before plasma treatment, PET films were cut into smaller samples measuring 60 mm x 60 mm.

Plasma treatment was performed on only one side of the polymer by means of a DBD system that generated electrical discharges at atmospheric pressure between two circular metal electrodes with a diameter of 60 mm as shown in Fig. 1. A 3 mm thick glass plate was used to form the dielectric barrier, which covered the grounded electrode and had the role of limiting the current produced by the high voltage source. Atmospheric pressure plasma is generated by an AC high voltage power supply (HVPS) with an average discharge power of about 40 W at an effective current in the range of 6 mA. This system was operating at a 10 kHz frequency and had an effective output voltage of about 7 kV.

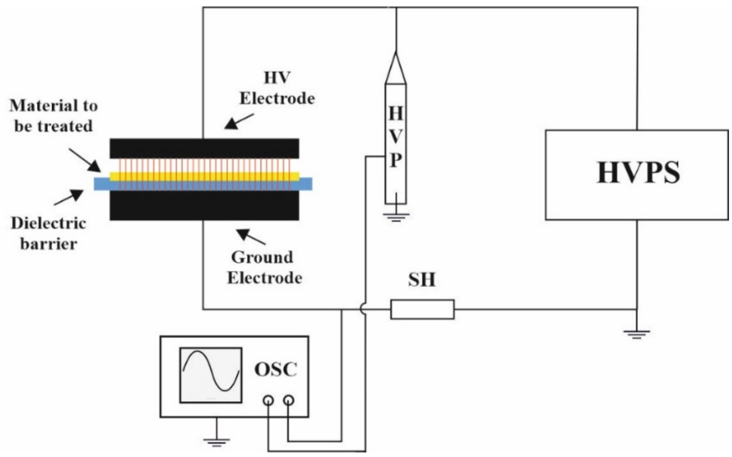

Fig. 1 – Schematic diagram of DBD system.

The material to be treated is positioned at a 4 mm gap from the HV electrode. When the applied voltage goes over the electrical breakdown threshold, it is started an avalanche ionization phenomenon so that between the electrode and the glass are generated many plasma streams that occur simultaneously and individually, with a lifespan of nanoseconds. The filamentary discharges were interrupted when the voltage passes to zero and were reinitiated when the voltage changes the polarity. The typical electrical discharge of the DBD system operating in ambient air within these electrical parameters is shown in Fig. 2.

Fig. 2 – Typical discharge by DBD system operating in ambient air.

The typical current and voltage waveforms to the DBD discharge presented in Fig. 3 were measured by means of a voltage probe (TT-HVP-2739) which has a divider ratio of 1000:1 and an impedance of 1 MΩ, and the current using a 3 Ω shunt resistor (SH). The HVP and the SH were measurement accessories that provided data recorded throughout an oscilloscope (OSC LeCroy 454) which is accurate and fast.

The wettability of the polymeric surface was evaluated using a common technique, namely the sessile drop method, which addresses flat surfaces and allows the determination of the static water contact angle (WCA) in the drop profile (Tesoi *et al*., 2020). This preferred method often used to characterize solid surfaces in terms of adhesion or hydrophobicity, has been implemented by an Ossila goniometer with a simple user interface. This goniometer has a specialized high-resolution camera that communicates with the computer through special software that determines the WCA value with great accuracy. This device contains a glass syringe with a volume of 25 µl, which has a detachable blunt needle with a 0.47 mm diameter and which allows a calibrated distilled water drop to be placed on the flat polymeric surface.

The wetting angle is also called the contact angle is formed when a drop of liquid in our case of distilled water is placed on a solid (polymeric) surface and thus the drop forms a dome shape on the material surface. The angle that forms between the polymer surface and the tangent line at the edge of the drop is called the contact angle, θ, which is WCA, as shown in Figure 4. It is observed that as the water droplet spreads on the polymer surface, the dome turn flatter and thus the WCA becomes smaller. When measuring the WCA formed by the droplet with the surface, the resulting value indicates whether the water droplet is more attracted to itself or to the surface on which it is located. The

WCA value is influenced by several factors such as surface roughness, surface tension, possible impurities or porosity.

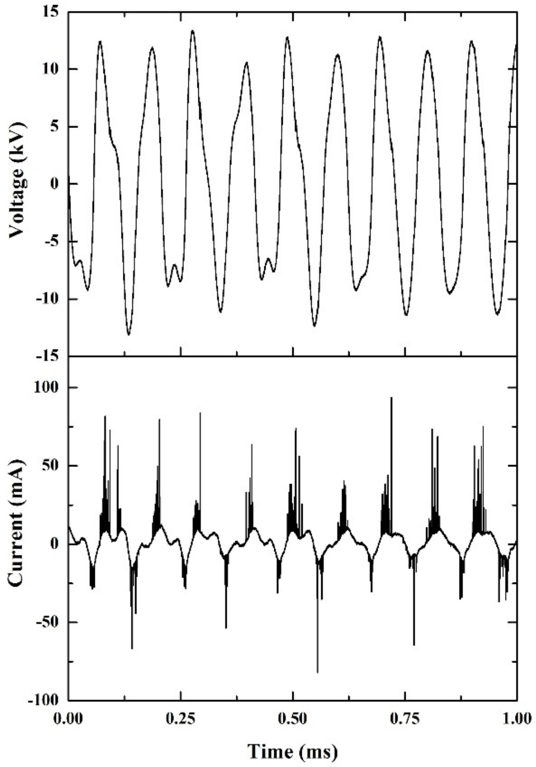

Fig. 3 – Typical current and voltage waveforms in the DBD system.

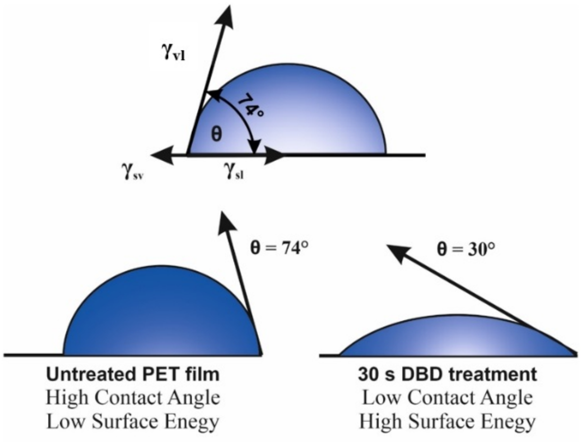

Fig. 4 – Static WCA with the sessile method on a polymeric surface.

Young's equation mathematically expresses the relationship between the contact angle of water and the surface energy of the substrate where γ_{lv}, γ_{sl}, and γ_{sv} represent the interfacial tensions of the liquid/vapor, solid/liquid and solid/vapor, respectively (Yildirim Erbil, 2021):

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \bullet \cos\theta \tag{1}$$

According to Eq. (1), when the WCA is less than 90° ($\gamma_{sv} < \gamma_{ls}$, cos θ is negative) resulting in a hydrophilic surface and for a WCA higher than 90° ($\gamma_{sv} > \gamma_{ls}$, cos θ is negative) results in a poor wetting and a hydrophobic surface.

A commercial ink from Arcotest using a set of liquids with a defined surface tension was used to measure the surface tension (σ) of the PET surfaces. Pink test inks are liquids used in this paper for surface tensions from 20 to 60 mN/m with an accuracy of ± 1 mN/m and a 4 s observation time.

3. Results and Discussion

The two polymeric PET materials with or without heat treatment with the same thickness and cut to the size of 60 mm x 60 mm were measured on average with 3 measurements on each experiment condition in the laboratory at the same temperature and relative ambient humidity. The plasma exposure time of the surface varied from 0.2 s to 30 s, time controlled by single channel 5V relay module that was programmed by an Arduino. The exposure time of the plasma resulting from the programming of the relay was checked by means of the oscilloscope and the set time was corrected in the Arduino software according to the time recorded by the oscilloscope, resulting in a quite good accuracy even for exposure times below 1 s. The PET surfaces were treated by ambient air atmospheric plasma in order to increase the wettability.

The WCA values as a function of treatment time as well as its evolution as a function of storage time after exposure to non-thermal plasma for PET 1 without heat-stabilized are shown in Fig. 5. Note that the reference value for PET 1 is around 74° with an error of 5% considered for all experiments performed. After a brief plasma exposure of 0.2 s, the WCA decreases to 50° and after 31 s changes to 31°. A treatment time of 0.5 s, 1 s, 5 s and 10 s corresponds to WCA values of 49°, 48.5°, 46° and 37.5° respectively. It is indicated that the physical properties of PET 1 change quickly and depending on the polymer residence time in the plasma zone.

The data presented in the graph suggest that the lowest value of WCA corresponds to the first measurement immediately after DBD treatment when the angle is below 50° for all times considered.

Stability of the surface condition after DBD treatment is very important if subsequent coating and printing processes require a longer waiting time.

Therefore, the aging behavior of the WCA of the treated PET films after 1-4 days was also tested.

In the case of 30 s plasma exposure of the PET 1 surface, the angle, at 0 sec time, was 31°, after 8 hours of treatment it was 41°, and after 96 hours it reached 43°. It is found that the fastest change in WCA occurred in the first 8 hours of storage. It can be seen that the time evolution takes place especially in the first 24 hours of storage time and then the WCA stabilizes for all the considered treatment times.

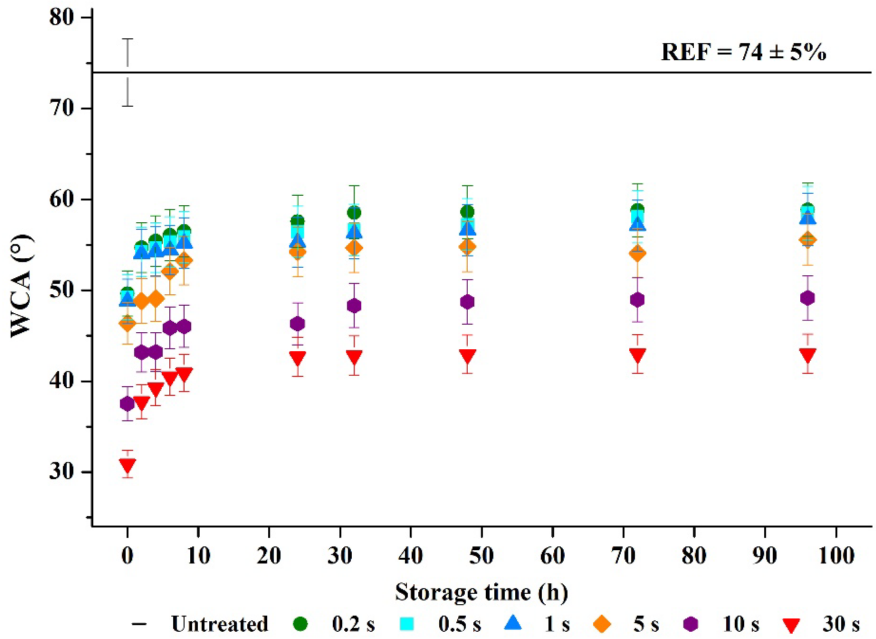

Fig. 5 – The effect of plasma exposure time on WCA on PET 1 film surface.

Fig. 6 shows the WCA measured on PET 2 (with heat-stabilized) before and after the DBD treatment at different treatment times and its evolution with storage time. WCA of untreated PET 2 is around 73.5° which corresponds to the data obtained by Homola *et al*. where it was treated Melinex® ST506 among other 2 materials by means of two atmospheric pressure plasma generators with a roll-to-roll system (Homola *et al*., 2014). Also, in the case of PET 2, the values obtained for WCA suggest that the best hydrophilic properties correspond to the initial time after plasma treatment when the angle is less than 48°.

It was found that after 24 hours of storage from plasma exposure, the WCA increased by 37% for the 30 s treated sample and 27% for the 10 s treated sample. After 96 hours of storage, the WCA increased for DBD treatment time of 10 s and 30 s by 36% and 40%, respectively. The appearance of the aging effect on the PET surface treated with non-thermal plasma is well known and is normally observed in the first hours which is explained by the decrease of polar functional groups on the surface after storage in air (Van Dongen *et al*., 2013).

After non-thermal plasma DBD treatment, no thermal damage was observed on PET 1 or PET 2 films, probably due to the thickness (125 µm). It was found that there are no significant differences between the surfaces of PET 1 and PET 2 in terms of WCA.

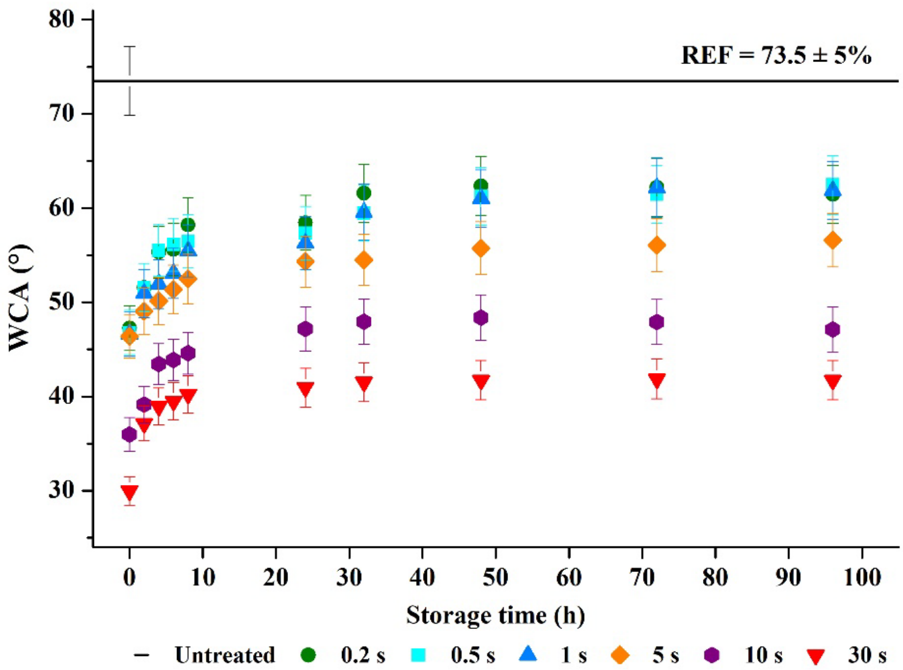

Fig. 6 – The effect of plasma exposure time on WCA on PET 2 film surface.

The surface tension can be determined by means of a specialized commercial test from Arcotest which has a wide range of inks with a defined surface tension σ. The surface is wetted when it has a surface tension less than or equal to the value indicated by the Arcotest ink. The adhesion of paints to a surface is considered sufficient when the surface energy is around equal to σ = 38 mN/m or higher (Matoušek *et al*., 2020).

Table 1 shows the surface tension for the surface of PET 1 and PET 2 measured by means of special inks from Arcotest. Untreated samples showed a surface energy below 40 mN/m (σ < 40 mN/m for PET 1, σ < 36 mN/m for PET 2). After applying the DBD treatment for 5 s, the surface tension was measured immediately, resulting, for both materials, a value between 58 and 60 mN/m.

The significance of the signs: < 36, < 40 and < 60 is that the surface tension is less than the energy that should be wetted by the ink with σ = 36 mN/m, 40 mN/m and 60 mN/m respectively.

Table 1
Surface Tension Measurements by Arcotest ink

Polymer samples	Surface tension σ Arcotest ink (mN/m)	
	Untreated	After DBD treatment
PET 1	< 40	< 60
PET 2	< 36	< 60

4. Conclusions

Two PET films (PET 1 and PET 2 with heat-stabilized) were treated by a DBD system operating in air at atmospheric pressure. The effect of plasma surface treatment was assessed by evaluating the WCA and surface tension through a set of special ink from Arcotest. In all considered cases, improvement of surface properties was achieved.

The hydrophilic properties of PET 1 and PET 2 surfaces were found to be improved upon exposure to non-thermal plasma by lowering the WCA from about 74° for the untreated sample to 50° after 0.2 s of treatment. When it is desired to use and treat foils in-line printing systems where there is speed of movement, it is necessary that the treatment time be as short as possible and with good results.

The data presented suggest that the lowest value of WCA corresponds to the first measurement immediately after DBD treatment when the angle is below 50° for all times considered.

It was found that the fastest change in WCA occurred in the first 24 hours of storage where WCA can increase by 40% compared to the value obtained immediately after treatment.

If the WCA decreases, the surface energy (Arcotest) is increased and the PET surface is wetted by 58 mN/m ink.

After plasma treatment, no thermal damage was observed on polymeric surfaces and was noted that there are no significant differences between the surfaces of PET 1 and PET 2 in terms of WCA and surface tension.

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SISTEM DBD CARE FUNCȚIONEAZĂ ÎN AERUL AMBIENTAL PENTRU TRATAMENTUL DE SUPRAFĂȚĂ AL FOLIILOR DIN POLIETILEN TEREFTALAT

(Rezumat)

Polimerii reprezintă materiale utilizate pe scară largă în multe tehnologii și aplicații importante datorită proprietăților lor optice, fizice, mecanice, termice și chimice. Folia de polietilen tereftalat (PET) este adesea folosită ca material izolator pentru circuite electronice imprimate, circuite flexibile, afișaje, filme grafice pentru aplicații industriale sau pentru aplicații clinice și de asistență medicală. Un mare inconvenient al foliei PET este tensiunea superficială scăzută, care implică o umectabilitate redusă, rezultând astfel proprietăți de imprimare și aderență slabe. Pentru a elimina acest dezavantaj, s-a utilizat un tratament de suprafață prin intermediul unei descărcări de tip barieră dielectrică (DBD) care funcționează în aerul ambiental la presiune atmosferică și care a constat dintr-o sursă de curent alternativ de înaltă tensiune și doi electrozi metalici circulari cu diametrul de 60 mm. Scopul lucrării este de a trata două substraturi diferite de film PET denumite în această lucrare ca PET 1, folie netratată termic, și PET 2 care este tratată termic de producător. Efectul expunerii suprafeței la plasmă a fost evaluat prin determinarea unghiului de contact static cu ajutorul unui goniometru Ossila și prin măsurarea tensiunii superficiale cu o cerneală specială. Foliile luate în considerare au fost expuse la diferiți timpi de tratament DBD (0,2, 0,5, 1, 5, 10, 30 s) și a fost monitorizată evoluția unghiului de contact în funcție timpul de depozitare de după tratament până la 96 de ore. S-a constatat că unghiul de contact al foliilor netratate PET 1 și PET 2 a fost de 74°, respectiv 73,5°, iar după 30 s de tratament DBD a scăzut la 30,9° și, respectiv, 30°.