

Effect of Barrier Plasma Pre-Treatment on Polyester Films and their Adhesive Properties on Oak Wood

Igor Novák,^a Ján Sedliáčik,^{b,*} Milada Gajtanska,^b Jarmila Schmidtová,^b Anton Popelka,^c Pavlo Bekhta,^d Tomasz Krystofiak,^e Stanisław Proszczyk,^e and Ondrej Žigo^a

A barrier plasma, created at atmospheric pressure, was used to improve the surface and adhesive properties of polyester (PES) film with respect to wood using polyurethane adhesives. The modification of PES film surfaces using barrier discharge plasma is attractive for various applications. Plasma pre-treatment initiates and participates in grafting, polymerization, or cross-linking reactions on the PES surface. This method of surface modification is clean, dry, ecological, and very efficient. The enhancement of the wettability of the polyester film was necessary for promoting higher adhesion to wood with water-based adhesives. The treatment of polyester films by barrier plasma led to a considerable increase in the surface free energy of the film and subsequently an increase in the peel strength of the adhesive joint of PES film-oak wood with polyurethane adhesive.

Keywords: Adhesive properties; Barrier discharge plasma; Hydrophilicity; Oak wood; Surface free energy; Lamination; X-ray photoelectron spectroscopy

Contact information: a: Polymer Institute, Slovak Academy of Sciences, Dúbravská cesta 9, 845 41 Bratislava, Slovakia; b: Department of Furniture and Wood Products, Technical University in Zvolen, Masaryka 24, 960 53 Zvolen, Slovakia; c: Center for Advanced Materials, Qatar University, P.O. Box 2713, Doha, Qatar; d: Department of Wood-Based Composites, Cellulose & Paper, Ukrainian National Forestry University, Gen.Chupryny 103, 79057 Lviv, Ukraine; e: Department of Wood Based Materials, Poznań University of Life Sciences, ul. Wojska Polskiego 28, 60-637 Poznań, Poland;

* Corresponding author: sedliacik@tuzvo.sk

INTRODUCTION

Atmospheric pressure plasma activation represents a technique for replacing traditional methods of surface preparation by abrasion. With sufficient exposure to the plasma afterglow, polymer and composite surfaces are fully activated such that when bonded and cured with adhesives, they undergo 100% cohesive failure in the adhesive.

Polyester (PES) films are frequently used as laminates in various industrial applications, *e.g.*, in the automotive and furniture industries, because of their excellent properties. The surface free energy (SFE) of polyester is insufficient for many applications, *e.g.*, bonding, lamination, and printing (Černáková *et al.* 2001; Shenton *et al.* 2001; Bekhta *et al.* 2015; Novák *et al.* 2015; Liu *et al.* 2016). Plasma processing can lead to improvements in the wetting, permeability, conductivity, adhesion, and biocompatibility of polymer materials. Plasma discharge used for surface modification can facilitate process cost reduction by removing the need for solvents and drying. The consumption of chemicals is low, and hence the requirements for their disposal and any other by-products are minimal. Moreover, these processes are easily controllable. The surface can be modified and a homogeneous surface chemical composition can be achieved, depending on the particular requirements and uses (Popelka *et al.* 2014). The application of cold

plasma for pre-treatment of polymeric surfaces is a dry, environmentally friendly method of polymer surface modification that is frequently applied to modify the surface energy and to increase its adhesion to other materials, *e.g.*, wood (Ciolacu *et al.* 2011; Acda *et al.* 2012; Novák *et al.* 2012). The activation of a polymer by plasma consists of surface actions by energetic particles, breaking polymer chains or abstracting either pendant groups or atoms such as hydrogen, forming free radicals in the barrier plasma. Active species within the barrier plasma react with the created sites to obtain thermodynamic stability, which results in changing the chemistry of the polymer surface. Activation by barrier plasma using oxygen as a processing gas leads to an increase in the surface energy of the polymer. Selection of the gas, as well as the process parameters, permits the surface of the polymer to be tailored to promote its polarity and adhesion (Kan 2015). It is well known that the modification of polymers by cold plasma leads to changes in its surface and adhesive properties (Fridman 2008; Kusano 2014).

This paper is devoted to the investigation of the surface and adhesive properties of PES films modified by atmospheric barrier plasma in air and oxygen. Measurements of the surface energy of testing liquids were carried out for characterization of the surface properties of the PES film modified by barrier plasma in air and oxygen. Changes in the chemical structure of PES films were analyzed with X-ray photoelectron spectroscopy (XPS). The strength of the adhesive joints of PES films pre-treated by barrier plasma and oak wood using water-based polyurethane (PUR) adhesive was also determined.

EXPERIMENTAL

Materials

Polyester (PES) film (Lishui Tianzhen Co., Ltd, China) with a thickness of 0.2 mm (usually applied for furniture decoration) was used in the experiments. The PES film was treated with acetone to eliminate additives influencing its surface properties. The adhesive joints of the PES with oak wood were prepared using a water-based polyurethane dispersion.

Oak wood (*Quercus petraea*) plates with semi-radial faces and 8% moisture content were planed on the tested side, cut to dimensions of 25×100 mm with a thickness of 5 mm, and used for experiments.

Modification by Barrier Plasma

The diffuse coplanar surface barrier discharge (DCSBD) plasma generator consists of 96% Al₂O₃ dielectric panels; an electro-insulated pair of comb-like metallic feed electrodes (width, 1.5 mm; gap, 1 mm) placed 0.635 mm underneath its surface was powered by a harmonic voltage of 8 kV rms, 14 kHz, and 300 W input power (Odrášková *et al.* 2008; Novák *et al.* 2012; Prysiashnyi *et al.* 2014). As a result, cold DCSBD plasma was formed on the dielectric surface, covering an area of 195×80 mm² and spreading approximately 0.3 mm outwards into free space (Figs. 1 and 2). Samples of PES film with dimensions of 25×60 mm were fixed to the holder on a rail cart and treated in the plasma by moving periodically from one side to the other and back again above the DCSBD ceramics. The distance between the sample and the ceramic plate was fixed at 0.3 mm with an exposure time within the range of 5 to 20 s.

The modification of PES film by DCSBD plasma was performed in atmospheric pressure in air and in oxygen gas of a technical purity in a dynamic regime. Air and oxygen,

when used as a plasma gas, are reactive gases. The impact of the oxygen-containing plasma on the polymer surface led to the generation of oxygen-based unstable functional groups, *i.e.*, hydroperoxides and peroxides, the latter decomposing to more stable carbonyl groups. On the other hand, because of the influence of ozone, some deterioration of mechanical properties of the polymer may occur.

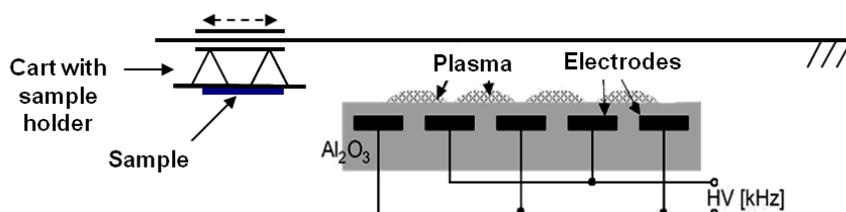


Fig. 1. Scheme of DCSBD plasma source



Fig. 2. DCSBD plasma generator and electrodes covered by plasma (in air)

Surface Energy Determination

The surface energy of the PES film surface was determined using contact angle measurements with selected testing liquids using a surface energy evaluation (SEE) system (Advex Instruments, Czech Republic). Eight drops of each testing liquid (re-distilled water, ethylene glycol, formamide, methylene iodide, and α -bromo naphthalene (Aldrich, USA); $V = 3 \mu\text{L}$) were placed on the PES film surface with a micropipette (Biohit, Finland) and the average value of the contact angle of the testing liquid was measured instantly after placing the liquid drop. The surface energy and polar ratio of selected testing liquid set are presented in Table 1.

Table 1. Surface Energy and Polar Ratio of the Testing Liquids Set at 23 °C

Testing liquid	γ_L (mJ.m ⁻²)	χ_L^P
Re-distilled water	72.8	0.7
Ethylene glycol	48.3	0.4
Formamide	58.2	0.3
Methylene iodide	50.8	0.1
1-Bromo naphthalene	45.3	0

In the table, γ_L is the surface energy of testing liquid, $\chi_L^P = \gamma_L^P/\gamma_L$ is the polar ratio.

The surface energy of the polymer as well as its polar and dispersive components were evaluated by the Owens-Wendt-Rable-Kaelble method modified by a least squares method, according to Eq. 1 (Acda *et al.* 2012; Novák *et al.* 2015),

$$\frac{(1+\cos\theta)\gamma_{LV}}{2} = (\gamma_{LV}^d \gamma_s^d)^{1/2} + (\gamma_{LV}^p \gamma_s^p)^{1/2} \quad (1)$$

where θ is the contact angle of the testing liquid (deg), γ_{LV} is the surface energy of the testing liquid ($\text{mJ}\cdot\text{m}^{-2}$), $\gamma_{LV}^d, \gamma_{LV}^p$ are the dispersive component (DC) and polar component (PC), respectively, of the testing liquid surface energy ($\text{mJ}\cdot\text{m}^{-2}$), and γ_s^d, γ_s^p are the DC and PC, respectively, of the surface energy of the polymer ($\text{mJ}\cdot\text{m}^{-2}$).

Peel Strength of the Adhesive Joint

The surface area of PES films was 25×60 mm with a thickness of 0.2 mm, and the oak wood plate was 25×100 mm with a thickness of 5 mm. The oak wood samples were covered with 1-mm layer of water-based PUR adhesive using an applicator ruler. Adhesive bonds were created after fixation of resinated oak wood samples to PES films. The prepared adhesive joints were fixed in a manual press, hardened for 48 h at room temperature, and then tested. The peel strength of the adhesive joint (P_{peel}) of plasma-modified PES to oak wood using polyurethane adhesive was determined by the peeling of adhesive joints at a 180° peel angle using a 5-kN testing equipment Instron 4301 (Instron, USA). The average values from six measurements of the peel strength of adhesive joints were calculated. The strength of adhesive joints was measured immediately after plasma pre-treatment of the PES film surface.

X-Ray Photoelectron Spectroscopy (XPS)

X-ray photoelectron spectroscopy is a surface analysis technique that can be applied to a broad range of materials and provides valuable quantitative and chemical state information from the surface of the materials being studied. The XPS spectra were recorded using a VG Scientific ESCALAB 250 (Thermo Fisher Scientific Inc., UK) device equipped with a micro-focused, monochromatic Al K_α X-ray source (1486.6 eV) and a magnetic lens that increases the electron acceptance angle and hence the sensitivity. The spectra were acquired in constant analyzer energy mode, with pass energies of 150 and 20 eV for the survey and narrow regions, respectively. Spectral calibration was performed by setting the main C1s peak at 285 eV (binding energy for the C-H 1s peak in eV). A more detailed analysis of the XPS spectra of the plasma pre-treated wood in air revealed the formation of different oxygen-containing functional groups. XPS of the carbon C1s region can indicate the existence of carboxyl, carbonyl, and alkoxy groups. The increase in oxygen (O1s peak) could also arise from oxygen sources outside the polymer. The O/C atomic ratios were determined by considering the integrated peak areas of C1s (peak for carbon) and O1s (peak for oxygen), and their respective Scofield's relative sensitivity factors corrected for the analyzer transmission function. The N1s peak (peak for nitrogen) contains various nitrogen-based functional groups. The XPS measurements were carried out immediately after modification of the film surface by plasma in three different places of the modified substrate, and the average value was taken into account.

Statistical Evaluation

Two-factor analysis of variance (ANOVA) was calculated. At the beginning of data processing the individual sample sets (the sets of measures at the given treatment time and the given processing gas) were described by the arithmetic means. Next, the methods of inductive statistics to make general inferences were used. The null hypothesis, that the mean value of surface energy with respect to peel strength is the same along the four treatment times and two processing gases, was tested. The 5% significance level was applied. Statistical software STATISTICA 12 was used.

RESULTS AND DISCUSSION

In the process of plasma pre-treatment, the polymer was exposed to plasma reactive species, such as ions, electrons, excited atoms, and molecules, which cleave existing chemical bonds and form new reactive functional groups. These reactive functional groups initiate and participate in grafting, polymerization, or cross-linking reactions on the surface. Plasma processing effectively contributes to adhesion improvement by removing surface contamination and by changing surface morphology through increased roughness resulting from etching (Pappas 2011; Petrič 2013; Kostov *et al.* 2014).

Surface Free Energy

The results of two-way ANOVA in terms of the surface free energy are presented in Table 2. The effect of treatment time, processing gas, and their interaction was significant. The null hypothesis was rejected at the given significance level. The differences of mean SFE values of the PES film modified by barrier plasma in air and O₂ were highly significant ($p < 0.000$) according to results of Duncan post-hoc tests. Values of SFE were higher in the processing gas of O₂.

Table 2. Two-factor ANOVA Test's Results Evaluating the Effect of Plasma Treatment on Surface Free Energy

Source of variation	Sum of Squares	Degree of Freedom	Mean Square	Fisher F test	p-level
Plasma Treatment Time	3798.07	3	1266.02	2241.35	0.000
Processing gas	888.17	1	888.17	1572.40	0.000
Plasma Treatment Time*Processing gas	27.02	3	9.01	15.94	0.000
Residuum (Error)	49.71	88	0.56		

Figure 3 illustrates 95% confidence intervals of mean SFE values. The surface free energy of PES film after modification by barrier plasma in O₂ (plot a) and air (plot b) significantly increased in comparison with untreated PES film. Figure 3a shows that the surface energy of PES film, modified by barrier plasma in O₂, increased from an initial value of 47.8 mJ•m⁻² (untreated PES film) up to 84.7 mJ•m⁻² after 20 s of plasma modification. This important increase of surface energy is a result of surface polarity of PES film due to creation of oxygen groups during plasma modification process in oxygen. Figure 3b shows the surface energy of PES film modified by barrier plasma in air.

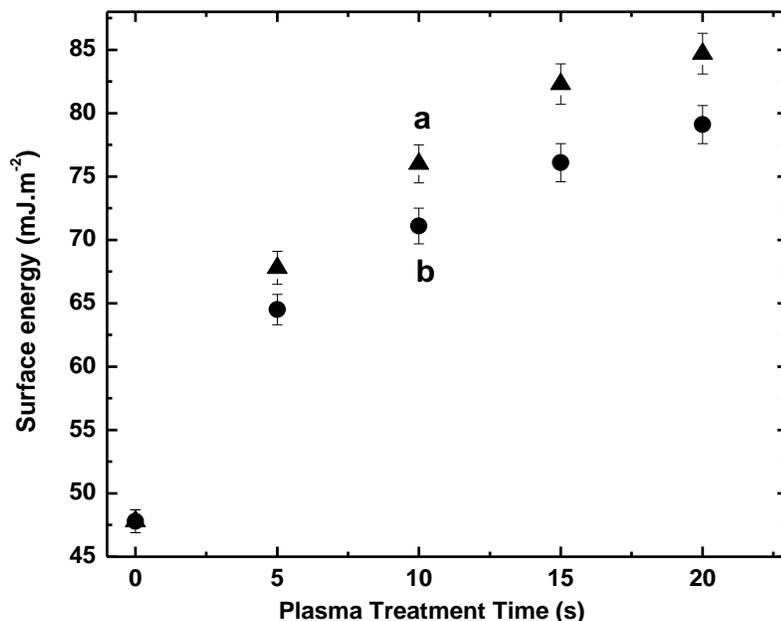


Fig. 3. Surface energy of PES film modified by barrier plasma *versus* plasma treatment time: a) O₂, b) air

The air contains not only oxygen, but a major amount of nitrogen, and due to this fact the values of surface energies in air as a processing gas were lower than those of PES film modified by the same method in O₂. For instance, after 20 s of barrier plasma pre-treatment, the PES film surface energy reached a value of 79.1 mJ·m⁻². The surface energy of unmodified oak wood was 66 mJ·m⁻², and this value was substantially higher than the surface energy of PES film, which was 47.8 mJ·m⁻². The modification of PES film using barrier discharge plasma made it possible to reach or overcome the surface energy of oak wood after a short pre-treatment time (barrier plasma, 5 s in air SFE = 64.5 mJ·m⁻² and barrier plasma, 5 s in O₂, SFE = 67.8 mJ·m⁻²). Surface energies of PES films were significantly higher after longer pre-treatment by barrier discharge plasma.

Yang *et al.* (2009) also concluded that the plasma treatment incorporated polar functional groups onto the surface of the PET film, causing rise in surface free energy. This effect is due to the presence of reactive particles (electrons, ions, radicals, *etc.*) in the discharge area. XPS analysis identified polar groups on the PET film surfaces. The above changes in PET surfaces made films more hydrophilic and surface free energy increasing suitable for industrial applications.

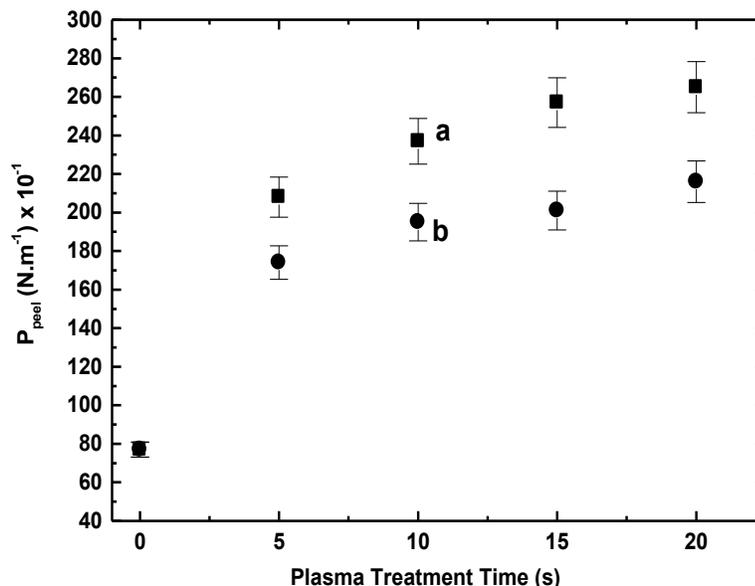
Peel Strength of Adhesive Joint

The results of two-factor ANOVA (Table 3) and Duncan post-hoc tests indicate that barrier plasma irradiation significantly ($p < 0.000$) improves the adhesive properties of PES film to oak wood using polyurethane adhesive.

Table 3. Two-factor ANOVA Test's Results Evaluating the Effect of Plasma Treatment on Peel Strength

Source of variation	Sum of Squares	Degree of Freedom	Mean Square	Fisher F test	p-level
Plasma Treatment Time	32451.71	3	10817.24	364.18	0.000
Processing gas	43605.38	1	43605.38	1468.06	0.000
Plasma Treatment Time*Processing gas	1104.04	3	368.01	12.39	0.000
Residuum (Error)	2613.83	88	29.70		

The peel strength of the adhesive joints of PES film modified by barrier plasma in oxygen and in air to polyurethane adhesive *versus* plasma treatment time is shown in Fig. 4. Due to decreasing differences between surface energy and polarity of plasma-treated PES films, the peel strength of adhesive joints of PES film – polyurethane – oak wood after modification by barrier plasma in oxygen (Fig. 4a) and in air (Fig. 4b) significantly increased, and these increases were higher for the samples modified in oxygen than for samples modified in air. The peel strength of the adhesive joint increased from $770 \text{ N}\cdot\text{m}^{-1}$ (unmodified PES) to $2160 \text{ N}\cdot\text{m}^{-1}$ (barrier plasma, 20 s, air) and $2650 \text{ N}\cdot\text{m}^{-1}$ (barrier plasma, 20 s, O_2).

**Fig. 4.** Peel strength of the adhesive joint PES film modified by barrier plasma – oak wood using PUR adhesive *versus* plasma treatment time: a) O_2 , b) air

XPS

Adhesion correlates with the fraction of the polymer surface sites that are oxidized and converted into active functional groups, as determined by X-ray photoelectron spectroscopy and infrared spectroscopy (Gonzalez and Hicks 2010; Williams *et al.* 2013).

The XPS survey scans of pristine PES film and also plasma-modified (5 s) PES film are shown in Fig. 5a, b, c. The main peaks of pristine PES film C1s and O1s were located at 284.7 and 531.9 eV, respectively. For PES film treated by DCSBD plasma in air (Fig. 5b) a sharp C1s peak at 284.7 eV can be observed in the survey scan, as well as O1s peak at 531.8 eV, due to surface oxidation of sample by DCSBD plasma. Nitrogen is also present on PES film surface (N1s at 399.7 eV). The XPS survey scans of PES film modified by DCSBD plasma in oxygen are shown in Fig. 5c. The C1s and O1s peaks of the PES film modified in oxygen plasma show higher amount of oxygen due to creation of oxygen groups.

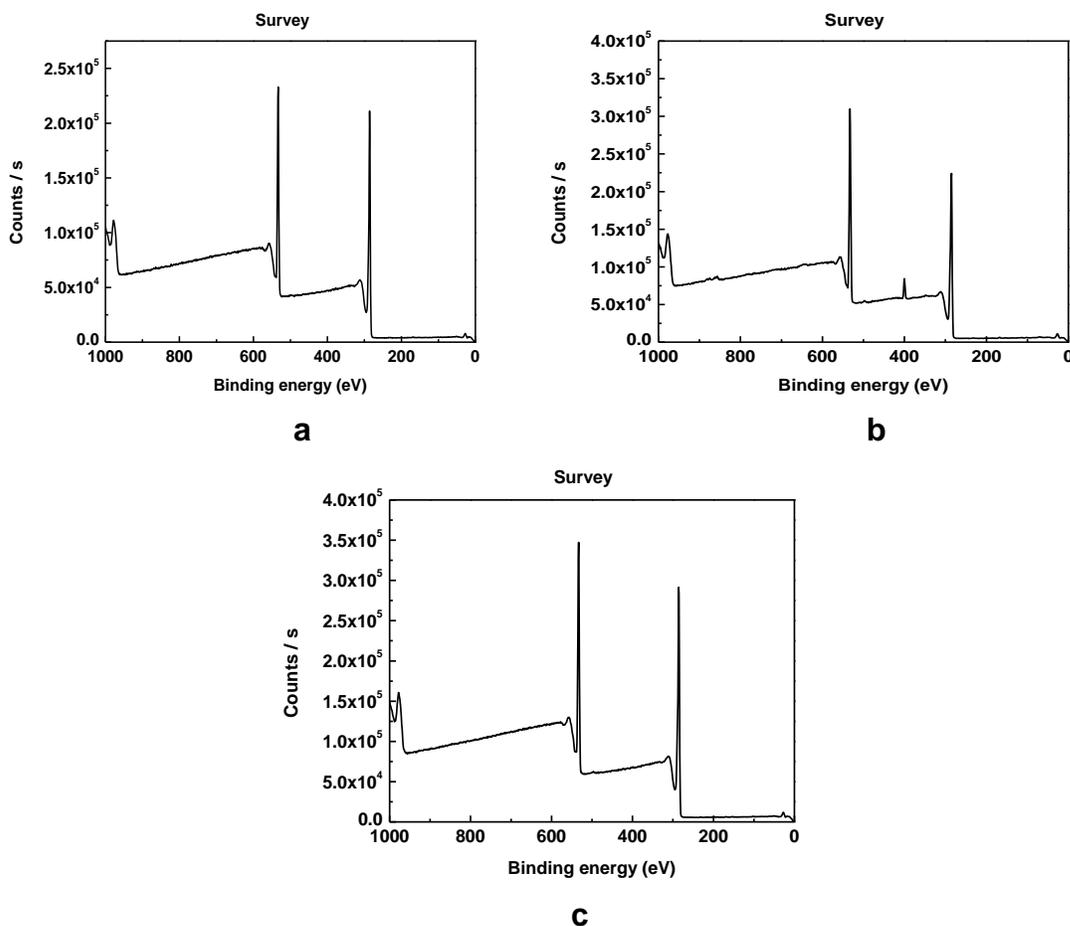


Fig. 5. XPS survey scans of PES film samples: a) pristine sample, b) sample modified by plasma in air, c) sample modified by plasma in oxygen

The XPS analysis of PES film treated by plasma in air (5 s) showed an increase in oxygen content from 24.4 at.% to 30.1 at.%. This increase was higher after pre-treatment of PES film in oxygen plasma, 5 s (from 24.4 at.% to 32.5 at.% compared with air, 5 s from 24.4 at.% to 30.5 at.%). For longer modification times in oxygen plasma, the increase in the oxygen content was lower because of the saturation of the PES surface with oxygen-containing groups. The XPS showed an increase in the nitrogen content of the PES film surface pre-treated by air plasma from 0 at.% to 1.7 at.%. After modification in oxygen plasma, no changes in the N1s peak were observed. The O/C ratio increased after modification by barrier plasma in air from 0.32 to 0.44 (air plasma, 5 s), or to 0.45 (air

plasma, 10 s); after the pre-treatment of PES film in oxygen plasma, this ratio increased to 0.48 (oxygen plasma, 5 s), or to 0.49 (oxygen plasma, 10 s). The values of the (N + O)/C ratio after the modification in air plasma and in oxygen plasma were similar (0.47 to 0.49 for PES film pre-treated in air/oxygen plasma), as presented in Table 4.

Table 4. XPS Element Amount, O/C, N/C, and (N + O)/C Ratios of PES Film Treated by Barrier Discharge Plasma

Parameter	Untreated	Plasma Air, 5 s	Plasma Air, 10 s	Plasma O ₂ , 5 s	Plasma O ₂ , 10 s
C1s	75.6	68.2	67.2	67.5	67.3
O1s	24.4	30.1	30.5	32.5	32.7
N1s	0	1.7	2.3	0	0
O/C	0.32	0.44	0.45	0.48	0.49
N/C	0	0.02	0.03	0	0
(N+O)/C	0.32	0.47	0.49	0.48	0.49

Rodriguez-Santiago *et al.* (2011) also showed an increase in the total surface energy of polymers as a function of non-thermal atmospheric-pressure plasma exposure time, with the greatest contribution coming from the polar component of the surface energy. XPS results show the incorporation of oxygen groups on all polymer surfaces, varied depending on plasma treatment time, showing a higher concentration of hydroxyl groups at short exposure times, and a higher concentration of carboxylic acid groups for longer exposure times.

CONCLUSIONS

1. The surface energy of PES film modified by barrier discharge plasma in oxygen and in air was significantly higher in comparison with the untreated surface; and this increase was higher after pre-treatment of PES film in oxygen plasma compared with air plasma.
2. The peel strength of the adhesive joints of PES film modified by barrier discharge plasma to oak wood using polyurethane water-based adhesive highly significantly increased; these values were higher after modification of PES film in oxygen plasma than in air plasma.
3. XPS showed an important increase in oxygen and nitrogen contents in the PES film surface layers after pre-treatment in air plasma. A stronger increase in oxygen content was measured in the PES film surface modified in oxygen barrier discharge plasma, but no changes in nitrogen content were observed.

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