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## Characterization of Polylactic Acid Films for Food Packaging as Affected by Dielectric Barrier Discharge Atmospheric Plasma

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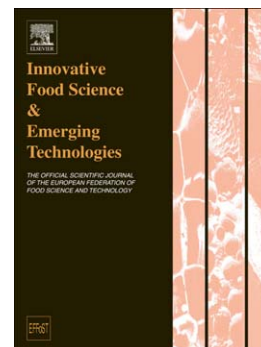
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**Characterization of polylactic acid films for food packaging as affected by dielectric barrier discharge atmospheric plasma**

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**Abstract**

Dielectric barrier discharge (DBD) air plasma is a novel technique for in-package decontamination of food, but it has not been yet applied to the packaging material. Characterization of commercial polylactic acid (PLA) films was done after in-package DBD plasma treatment at different voltage and treatment time to evaluate its suitability as food packaging material. DBD plasma increased the roughness of PLA film mainly at the site in contact with high voltage electrode at both the voltage levels of 70 and 80 kV. DBD plasma treatments did not induce any change in the glass transition temperature, but significant increase in the initial degradation temperature and maximum degradation temperature was observed. DBD plasma treatment did not adversely affect the oxygen and water vapor permeability of PLA. A very limited overall migration was observed in different food simulants and was much below the regulatory limits.

**Keywords:** Polylactic acid, DBD plasma, In-package, TGA, DSC, AFM, oxygen permeability, water vapor permeability, overall migration

## 1. Introduction

Petrochemical-based polymers like polyethylene, polypropylene, polystyrene and poly(ethylene terephthalate) are the most extensively used as food packaging materials. Recently, biodegradable polymers have attracted much attention due to the serious environmental problems with accumulation of waste disposal of conventional polymers. PLA is one of the most promising bio-based polymers which is biodegradable, recyclable and biocompatible requiring low manufacturing energy, with good processability, high transparency and water solubility resistance (Gupta, Revagade, & Hilborn, 2007; Rasal, Janorkar, & Hirt, 2010; Siracusa, Rocculi, Romani, & Rosa, 2008). Such properties coupled with a competitive market price have made it one of the first commercially available biopolymers widely used in the packaging of fresh produce. Although PLA offers a substitute for many non-biodegradable polymers, its application is limited due to its brittleness and barrier properties (Chaiwong, Rachtanapun, Wongchaiya, Auras, & Boonyawan, 2010; Rasal, et al., 2010).

Nonthermal plasma (NTP) is one of the many techniques which have been used for surface modification of polymers. The term "plasma" refers to a partially or wholly ionized gas composed essentially of photons, ions and free electrons as well as atoms in their fundamental or excited states possessing a net neutral charge (Misra, Tiwari, Raghavarao, & Cullen, 2011). On the basis of relative energy levels of electron and heavy species and the particle distribution function, plasma can be classified into thermal or nonthermal plasma. Unlike thermal plasma, NTP has temperature disequilibrium between electron and heavy species with relatively low electron density ( $<10^{19} \text{ m}^{-3}$ ) (Tendero, Tixier, Tristant, Desmaison, & Leprince, 2006). NTP at atmospheric pressures can be generated using various techniques like corona discharges which have a low current density and usually consist of an active electrode of small radius (like a point of wire) and the material to be treated. The type of corona depends on the polarity of the electrode, the voltage for spark breakdown, electrode geometry and surface condition, current and type

of gas (Harry, 2010). Corona discharges have low plasma volume and to increase the treatment area small radius electrode can be replaced with a planar electrode but it results in microarcs generation and non-homogenous treatment. To avoid this problem, a dielectric barrier discharge (DBD) was developed (Tendero, et al., 2006). DBD is one of such methods which offer versatility in its mode of operation and system configuration. In DBD, plasma is generated between two electrodes, separated by one or more dielectric barriers (Pankaj, Misra, & Cullen, 2013). The dielectric layer plays an important role by limiting the discharge current and avoiding the arc transition with randomly distributing streamers on the electrode surface, thereby ensuring a homogeneous treatment.

Recently a novel approach based on DBD plasma has been reported offering potential for decontamination of various foods products. The approach involves generation of NTP inside a sealed package which is in contact with a high voltage and a ground electrode. Upon exposure to a sufficiently high voltage, ionization of the gas within the electric field contained by the package occurs, generating significant amounts of reactive species, which in-turn will lead to decontamination of the enclosed products (Misra, et al., 2011). Based on the product's initial microbial load, gas composition and target micro-organism various processing parameters (treatment time, voltage) can be chosen. The effectiveness of the approach has been reported by Klockow and Keener (2009), Misra, Ziuzina, Cullen, and Keener (2012), Ziuzina, Patil, Cullen, Keener, and Bourke (2013) and Pankaj, et al. (2013), but consequences of processing on the packaging materials after DBD treatment have not been reported. Since it is already known that DBD plasma can modify both the surface and bulk properties of polymers (Liu, Cui, Brown, & Meenan, 2004), the aim of this work is to examine the effects of in-package plasma treatment on commercial PLA film and its suitability for the same.

## **2. Materials and Methods**

### **2.1. Materials**

Commercial co-extruded plain PLA film (average thickness =  $42.8 \pm 1.3 \mu\text{m}$ ) from NatureWorks® PLA with a crystallinity degree of 32 % was purchased from Amcor Flexibles, UK. It is a tri-layer film with PLA core covered with PLA skin layers on either side without any coating material or prior treatments. The PLA film was used as a pillow pouch packaging material for a polypropylene box of dimensions of 310mm × 230mm × 22mm. The sample in contact with the high-voltage electrode is referred as top-positioned and the sample in contact with ground electrode is referred as bottom-positioned throughout the study.

## **2.2. Plasma treatment**

A schematic of the experimental setup has been presented in Figure 1. The DBD plasma source consists of two circular aluminium plate electrodes (outer diameter = 158 mm) over perspex dielectric layers (10 mm thickness). When the potential across the gap reaches the breakdown voltage the dielectric barrier acts as a stabilizing material forming of a large number of micro-discharges. The applied voltage to the electrode was obtained from a step-up transformer (Phenix Technologies, Inc., USA) using a variac. The input of 230V, 50 Hz was given to the primary winding of high voltage step-up transformer from the mains supply. The atmospheric air condition at the time of treatment was 45 % relative humidity (RH) and 22°C. The samples were treated at 70 and 80 kV for 0.5, 1.5, 2.5 and 3.5 minutes and stored at normal room conditions before analysis.

**Figure 1: Schematic of the experimental setup for DBD plasma system.**

## **2.3. Material characterization**

### **2.3.1. Atomic force microscopy (AFM)**

AFM measurements were carried out to observe the surface topography of the samples before and after DBD plasma treatment. The AFM used was MFP-3D BIO 1126 (Asylum Research, Santa Barbara, CA, USA) operated in intermittent contact (tapping) mode. The images were collected at a fixed scan rate of 0.5

Hz. The sampling rate was 512 lines. The data was processed using MF3D software (version 111111+1219). The scanned area was  $25 \mu\text{m}^2$  and was repeated five times.

### 2.3.2. Thermogravimetric analysis (TGA)

TGA analysis were performed with a Mettler Toledo thermal analyser, model TGA/SDTA 851e (Schwarzenbach, Switzerland). Approximately 5g samples were heated at  $10^\circ\text{C min}^{-1}$  from room temperature to  $700^\circ\text{C}$  under nitrogen atmosphere (flow rate  $50 \text{ ml min}^{-1}$ ). Initial degradation temperature ( $T_5$ ) was determined as the temperature at which 5% of mass was lost and maximum degradation temperature ( $T_{\text{max}}$ ) was determined from the first derivative.

### 2.3.3. Differential scanning calorimetry (DSC)

DSC analysis was performed with a TA Instruments DSC Q2000 (New Castle, DE, USA) under a dry nitrogen gas flow rate of  $50 \text{ ml min}^{-1}$ . Approximately 3 mg samples were weighed in aluminium pans ( $40 \mu\text{L}$ ) and subjected to two heating-cooling cycles from  $-30^\circ\text{C}$  to  $200^\circ\text{C}$  at  $10^\circ\text{C min}^{-1}$ . Glass transition temperatures ( $T_g$ ) and melting endotherm were determined during the first heating cycle in order to evaluate the changes occurred after plasma treatment on the PLA surface.

### 2.3.4. X-ray diffraction (XRD)

Wide-angle X-ray scattering (WAXS) of PLA films was performed on a Bruker D8-Advance (USA) diffractometer, equipped with a Cu-K $\alpha$  radiation source ( $\lambda = 1.546\text{\AA}$ ), operating at 40 kV and 40 mA as the applied voltage and current, respectively. The incidence angle ( $2\theta$ ) was varied between  $5^\circ$  and  $90^\circ$  at a scanning rate of  $2^\circ\text{min}^{-1}$ .

### 2.3.5. Fourier transform infrared spectroscopy (FTIR)



FT-IR spectroscopy was carried out by using a Nicolet Avatar 360 FT-IR E.S.P. (UK) spectrometer from 4000 to 400  $\text{cm}^{-1}$  to measure any changes in the spectra intensities. A background spectrum was collected by keeping the resolution as 1  $\text{cm}^{-1}$ . After the background scan, treated and untreated film samples were placed in the sample holder and analyzed in triplicates.

### 2.3.6. Oxygen transmission rate (OTR)

OTR analysis was conducted with an Oxygen Permeation Analyser 8500 from Systech Instruments (Metrotec S.A, Spain). Treated films were cut into circular samples (14 cm diameter) and clamped in the diffusion chamber at 25°C. Pure oxygen (99.9%) was introduced into the upper half of the chamber while nitrogen was injected into the lower half, where an oxygen sensor was placed. The oxygen volumetric flow rate per unit area of the membrane and per time (OTR,  $\text{cm}^3\text{m}^{-2}\text{day}^{-1}$ ) was continuously monitored until a steady state was reached. All samples were analyzed in triplicates.

### 2.3.7. Water vapor transmission rate (WVTR)

WVTR was measured gravimetrically using the ASTM method E 96/E 96M-05 (ASTM, 2005). Briefly, 50 g of anhydrous calcium chloride, after drying at 100 °C for 24 h, was placed in each test jar to establish the dry conditions. Control and plasma treated film sample was placed over the jar, and its edge was sealed using paraffin wax ( $T_m = 54\text{--}56^\circ\text{C}$ ; Sigma-Aldrich, Ireland). After the films were mounted, the whole assembly was weighed and placed in a climatic chamber at a set temperature of  $24 \pm 1^\circ\text{C}$  and RH of  $50 \pm 1\%$ . The assembly was weighed with an accuracy of 0.001 g at 24 hours intervals. WVTR was calculated in  $\text{g}(\text{m}^2.\text{day})^{-1}$  using the equation 1.

$$WVTR = \frac{M_t - M_o}{\Delta t} \cdot \frac{1}{A} \quad (1)$$

where  $M_0$  is the initial mass of the test jar (g),  $M_t$  is the mass of the test jar at time  $t$  (g),  $\Delta t$  is the duration of the test period (day), and  $A$  is the effective surface area of the test film ( $m^2$ ). The experimental values of WVTR represent average of measurements from the three replicate samples.

#### **2.3.8. Overall Migration**

Overall migration study was carried out according to EN 1186 standard (European Standard EN 1186-1:2002). Control and plasma treated samples (80 kV for 3.5 minutes) were cut into strips of  $1\text{ dm}^2$  area ( $10 \times 10\text{ cm}$ ) and totally immersed in 100 ml of food simulant in a stoppered glass tube in triplicate. Four simulants viz. A (distilled water), B (3 % w/v acetic acid), C (10 %v/v ethanol) and D (95 %v/v ethanol) were used. The test condition used was  $40^\circ\text{C}$  for 10 days corresponding to the food contact during long term storage at room temperature or below, including heating up to  $70^\circ\text{C}$  for up to 2 hours, or heating up to  $100^\circ\text{C}$  for up to 15 minutes (European Standard EN 1186-1:2002). All simulants were subsequently evaporated to dryness and kept in air-dried oven until constant weight. Mass of the non-volatile residue was determined gravimetrically.

#### **2.4. Statistical Analysis**

Analysis of variance was done for all the treatments and significance of difference between treatments was assessed using Fisher and Tukey's multiple sample comparison tests. Significance levels were tested at  $p \leq 0.05$ . Analysis was carried out in SPSS statistical package (SPSS Inc., Chicago, USA) and Minitab software (Minitab Inc., USA).

### **3. Results and Discussions**

#### **3.1. Surface Properties**

Figure 2 shows the surface topography of PLA films before and after DBD plasma treatments. The surface of the untreated sample was relatively smooth structures randomly dispersed all over the

surface. The RMS roughness was 0.93 nm. Roughness parameters for all the samples are summarized in Table 1. It is clear from the results that roughness increased with increasing voltage and treatment time. The increase in the roughness was more evident in the samples which are in contact with the high voltage electrode. It should also be emphasized at this point that the roughness of bottom-placed films at 70 kV were not significantly different ( $p>0.05$ ) for all treatment times, although at 80 kV, significant differences ( $p\leq 0.05$ ) were observed after 2.5 and 3.5 minutes of treatment time. The periodic cone-like structures appearing on the samples in contact with the high voltage electrode would be most likely due to the etching effect of applied plasma. These structures presented a typical feature size and spacing at the order of few micrometers or smaller. These structures are wider and closer to each other as the voltage increases, so the area of flat surface was reduced and the grooves appeared at higher heights, as shown by data of highest peak and lowest groove in Table 1. The height of the cone-like structures increased moderately from 70 to 80 kV. However, there is a large increase in the lowest groove, from 19.2 to 42.5 nm, when the voltage increases. The bombardment of energetic particles such as electrons, ions, radicals, neutrals and excited atoms/molecules and UV-vis radiations on the surface of polymer films resulted in etching of the surface (Yang, Chen, Guo, & Zhang, 2009). Similar types of topographical features upon air-plasma treatment were also observed by Teraoka, Nakagawa, and Hara (2006).

**Figure 2: AFM images of the surface of untreated and plasma-treated PLA films (A. Untreated, B. 70 kV-3.5min-Bottom, C. 70 kV-3.5-Top, D. 80 kV-3.5-Bottom, E. 80 kV-3.5-Top)**

**Table 1: Roughness parameters (mean value  $\pm$  standard deviation) for control and DBD plasma treated samples ( $R_{RMS}$ : root mean square roughness,  $R_{Peak}$ : Highest peak,  $R_{Groove}$ : lowest groove). Means that do not share a letter are significantly different ( $p\leq 0.05$ ).**

### 3.2. Thermal Properties

Thermal properties from TGA and DSC are presented in Table 2. The thermal stability of treated and untreated samples was studied by TGA. As expected for pure PLA, the thermal degradation of all the samples followed a one-stage weight loss. The initial degradation temperature ( $T_5$ ) of the control PLA sample was 295 °C, with a maximum degradation temperature of 316 °C. An increase in thermal stability was observed after DBD plasma treatment for all samples. The increase in  $T_5$  was more evident at the top position at 70 kV treatments and for both positions at 80 kV treatments. A significant increase in  $T_{max}$  was also observed for all treatment voltages, times and positions (Figure 3). Although air plasma is likely to generate etching on the surface, the increase in thermal stability for all the samples after plasma treatment suggests that stable cross-linking at the surface level may be formed due to the oxygen enrichment of the PLA chain, which may retard thermal degradation.

**Figure 3: Effect of treatment voltage (kV), time (min) and position on maximum degradation temperature ( $T_{max}$ ).**

PLA thermogram (first heating cycle) consists of glass transition and melting endotherm events. The glass transition temperature ( $T_g$ ) of the untreated samples was found to be 62.1 °C and for the treated samples insignificant change in  $T_g$  was observed ( $p < 0.05$ ). The melting endotherm was observed between 143 and 180 °C with peak melting temperature between 164.4 and 165.9 °C for all the studied films. Melting enthalpy ( $\Delta H_m$ ) of all films after DBD plasma treatment was found in the range of 29 and 32.5 J.g<sup>-1</sup> in compare to the melting enthalpy of 29.7 J.g<sup>-1</sup> for the untreated film. In the second heating cycle cold crystallization exotherm was also observed. Cold crystallization exotherm peak were observed in all cases between 108 °C and 144 °C. Also we found that  $\Delta H_c + \Delta H_m \approx 0$  during the second heating step, which suggests the complete removal of thermal history from the PLA film by the first DSC heating-cooling cycle (Cao, Mohamed, Gordon, Willett, & Sessa, 2003). From these DSC results, it can be concluded that DBD plasma did not have strong effects on the main structure of PLA, since glass

transitions were maintained at approximately the same temperature relative to the control sample. It may be noted that  $T_g$  is an important parameter dictating the success in industrial processing of this polymer.

**Table 2: TGA and DSC parameters for PLA samples**

### 3.3. X-ray diffraction (XRD)

Wide-angle X-ray scattering tests (WAXS) were carried out for all samples. Clear peak was detected at  $16.6^\circ$  (data not shown) for all the studied films, which corresponds to the  $\alpha$ -crystallographic form of the crystalline phase of PLA, described as pseudo-orthorhombic unit cell (Burgos, Martino, & Jiménez, 2013; Kulinski & Piorkowska, 2005). There was no significant change in this peak after plasma treatment under all treatment conditions, suggesting that DBD atmospheric plasma did not cause any change in the crystallization behavior of PLA films.

### 3.4. Fourier transform infrared spectroscopy (FTIR)

Figure 4 shows the FTIR spectra of untreated and DBD plasma treated samples. An increase in the intensity of bands at  $700$  and  $756\text{ cm}^{-1}$  was observed after DBD plasma treatment. These bands were assigned to -C-C- stretching. The intensity of the band at  $2993\text{ cm}^{-1}$  which was assigned to the -C-H stretching was also increasing. The band intensity at  $3501\text{ cm}^{-1}$  was found to increase for the top position samples when compared to the bottom position samples and was assigned to -O-H stretching (free) (Chaiwong, et al., 2010). These results can be correlated with the surface oxygen enrichment after DBD plasma treatment which was also observed by Liu, et al. (2004) and also to the increase in thermal stability found for all the treated samples by TGA.

**Figure 4: FTIR spectra of treated and untreated PLA films. [AA-BB-T/B=Treatment voltage (kV)-  
Treatment time (min)-T:top/B:bottom]**

### 3.5. Barrier properties

Oxygen permeability is one of the most important properties of a packaging material for deciding its suitability for application in different food products. The most common food quality losses in packaged food are due to oxygen (Brown, 1992). Therefore, a high oxygen-barrier packaging material is critical for maintaining the initial high quality of the packaged product (Hong & Krochta, 2006). OTR\*e values ( $\text{cm}^3 \text{mm m}^{-2} \text{day}^{-1}$ ) in the steady state were measured for untreated samples and after DBD plasma treatment for both the top and bottom-positioned films. The average OTR\*e value for the untreated sample was  $23.37 \pm 0.66 \text{ cm}^3 \text{mm m}^{-2} \text{day}^{-1}$  and no significant differences ( $p > 0.05$ ) were observed after the plasma treatment for all treatment voltages, times and positions. These results are particularly important for PLA to fit in as a competitive commercial food packaging material. For commercial adoption it is necessary to ensure that the in-place food processing method does not adversely affect the oxygen permeability of the package.

WVTR results for the control and treated films are shown in Figure 5. No significant differences were observed after plasma treatments at 70 kV for all the treatment times and both positions. At 80 kV, an increase in WVTR was observed for the film placed at the top position for all treatment times. It is worth recalling at this stage that permeation of water vapor is governed by vapor pressures and concentration gradients on the two surfaces of the polymer, while gas permeability of polymers, in general, is the combined effect of diffusion and solubility via voids or gaps present in between the segments of a polymer chain (Chaiwong, et al., 2010). Results from this study indicated that although no significant change in the oxygen permeability was observed after plasma treatment, water vapor permeability slightly increased at higher voltage levels with treatment times, most significantly for the film in contact with the high voltage electrode.

**Figure 5: WVTR of treated and untreated PLA films. [AA-T/B=Treatment voltage (kV)-T:top/B:bottom]**

### 3.6. Overall Migration

Overall migration tests were undertaken to determine the total amount of non-volatile substances that might migrate into foodstuffs from PLA films, following in-package plasma treatment (Plackett, et al., 2006). Overall migration results for the control and plasma treated samples (80 kV for 3.5 minute treatment) are shown in Figure 6. These samples were selected in consideration of their treatment at the maximum applied voltage and time. These results are based on mean values of triplicates. It is clear from the results that the DBD plasma treatment significantly increased the migration of non-volatile substances in acidic and high alcohol percentage food simulants. Lactic acid being the lone monomer of PLA, migrated agents are mainly lactic acid monomers, dimers, and oligomers (Jamshidian, Tehrany, Imran, Jacquot, & Desobry, 2010). Therefore, it could be concluded that some PLA chain were broken after DBD plasma treatment resulting in the formation of low molar mass species prone to migrate through the polymer matrix under testing conditions. In addition, according to the studies conducted by Conn, et al. (1995), very limited migration should be expected from PLA into foods and that small amount of migrant will be subsequently hydrolyzed in the aqueous and acidic media commonly found in foods and in the stomach. It should be also highlighted that values obtained for the overall migration, even at the most drastic treatment conditions, are well below of the  $10 \text{ mg dm}^{-2}$  limit indicated in the current European Legislation (Commission Regulation N10/2011).

**Figure 6: Overall migration ( $\text{mg}/\text{dm}^2$ ) of control and treated PLA films. [AA-BB-T/B=Treatment voltage (kV)-Treatment time (min)-T:top/B:bottom]**

## 4. Conclusion

Pure PLA commercial films were used in this study for testing its suitability as food packaging material for in-package NTP decontamination of foods. DBD plasma treatment increased the roughness of PLA film mainly at the site in contact with the high voltage electrode, but it did not adversely affect the glass

transition temperature or oxygen and water vapor permeability. The increase in water vapor permeability at 80 kV level was also found to be within acceptable limits for food products. DBD plasma treatment resulted in significant improvement in the thermal stability of the treated polymer by increasing the initial and the maximum degradation temperatures. Lastly, the overall migration was found to be well below the regulatory limits. The combination of usage of biodegradable food packaging materials and a novel energy efficient method for food decontamination offers a promising alternative to classical methods, whereby it permits an effective decontamination of the packaged food and avoids any post-process recontamination or hazards from the package itself.

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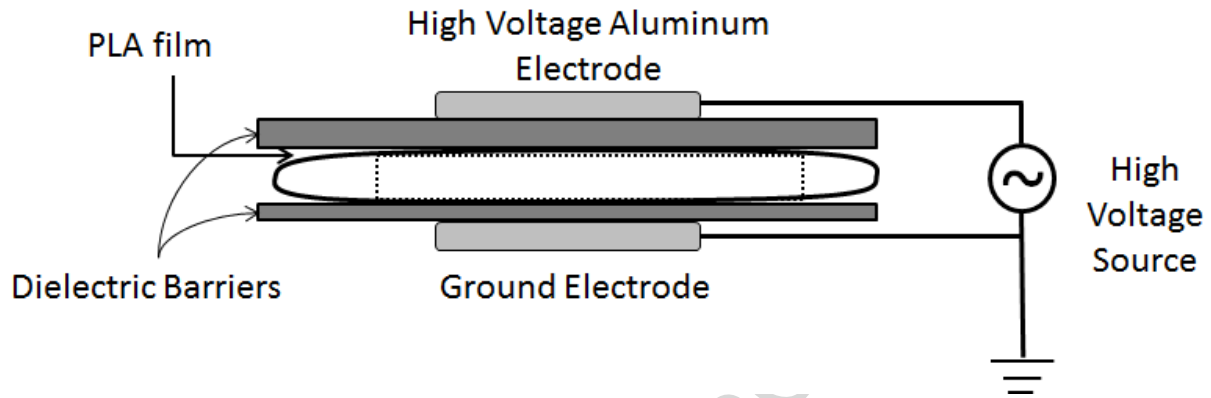


Figure 1: Schematic of the experimental setup for DBD plasma system.

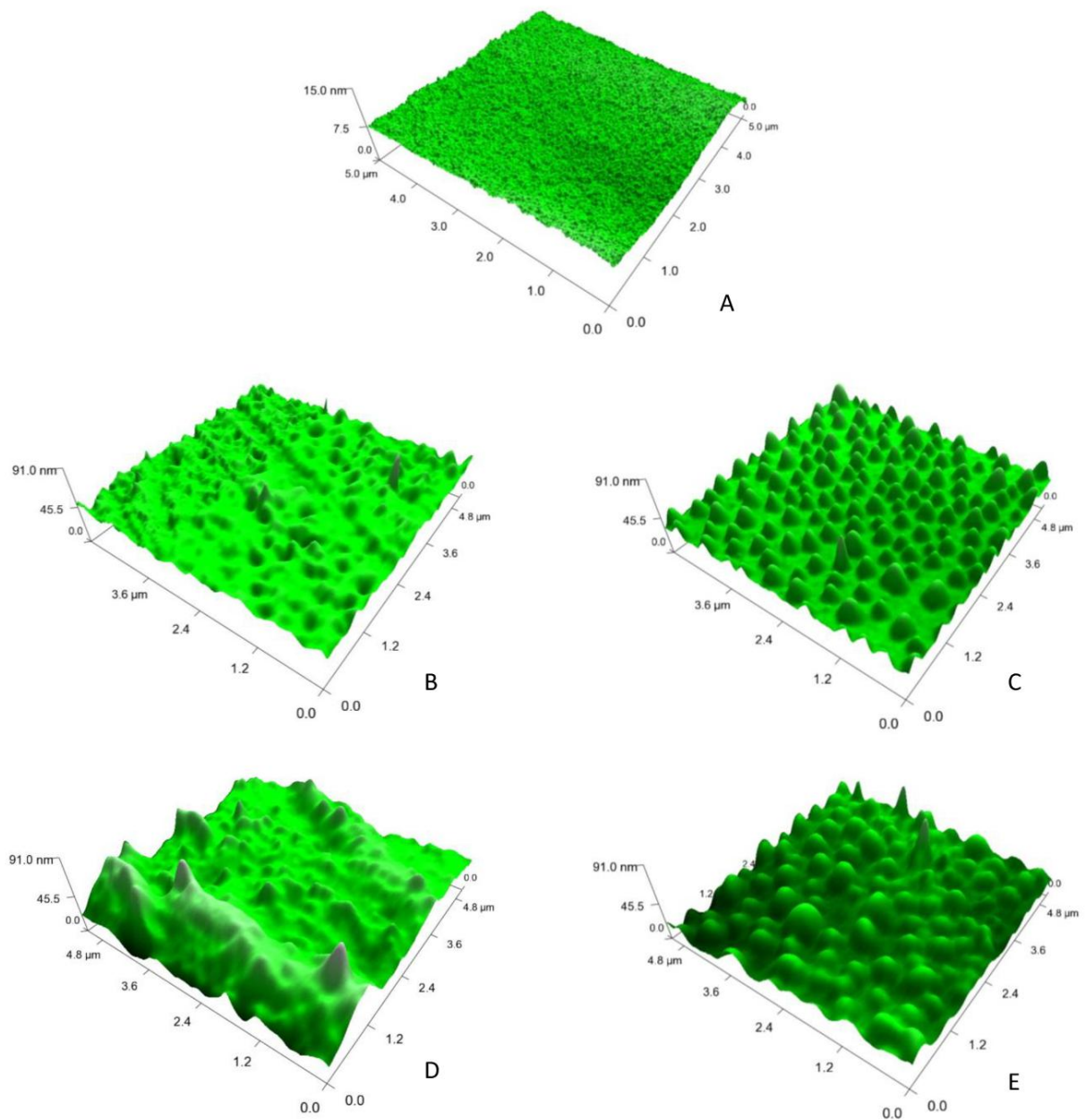


Figure 2: AFM images of the surface of untreated and plasma-treated PLA films (A. Untreated, B. 70 kV-3.5min-Bottom, C. 70 kV-3.5-Top, D. 80 kV-3.5-Bottom, E. 80 kV-3.5-Top)

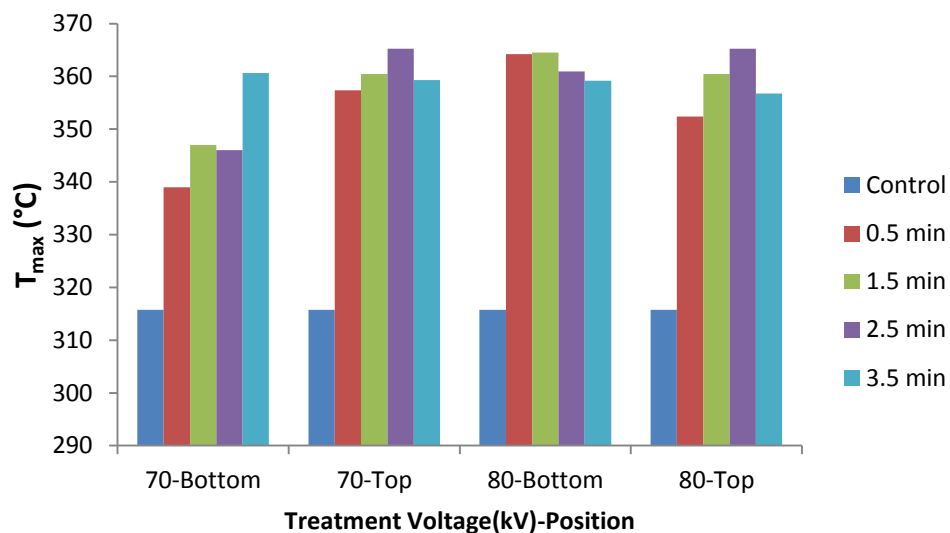


Figure 3: Effect of treatment voltage (kV), time (min) and position on maximum degradation temperature ( $T_{max}$ ).

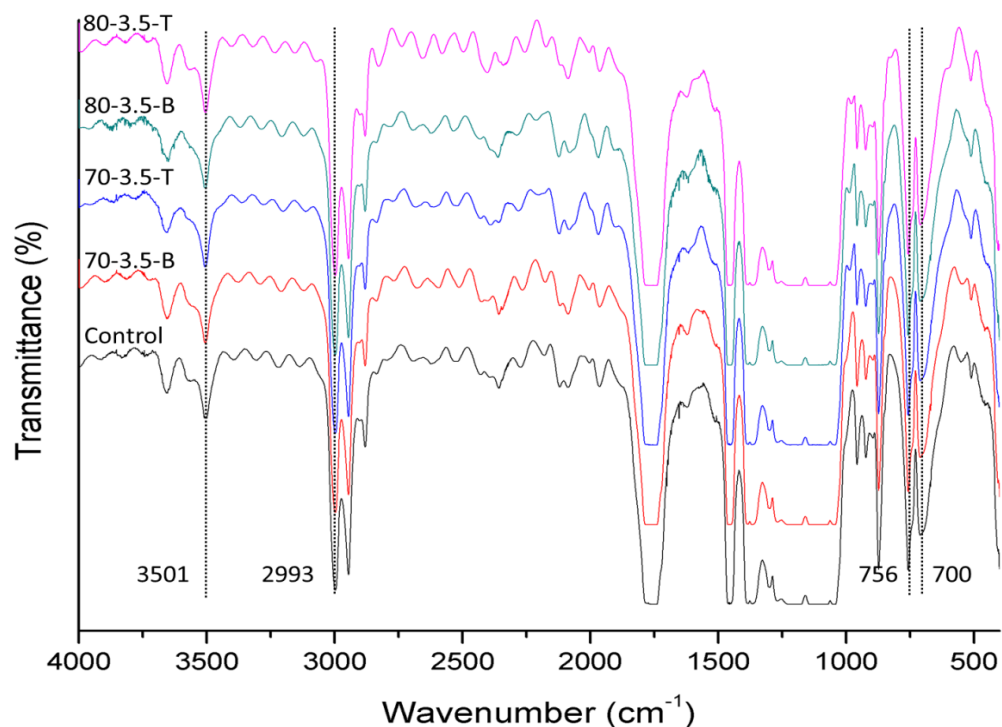


Figure 4: FTIR spectra of treated and untreated PLA films. [AA-BB-T/B=Treatment voltage (kV)-Treatment time (min)-T:top/B:bottom]

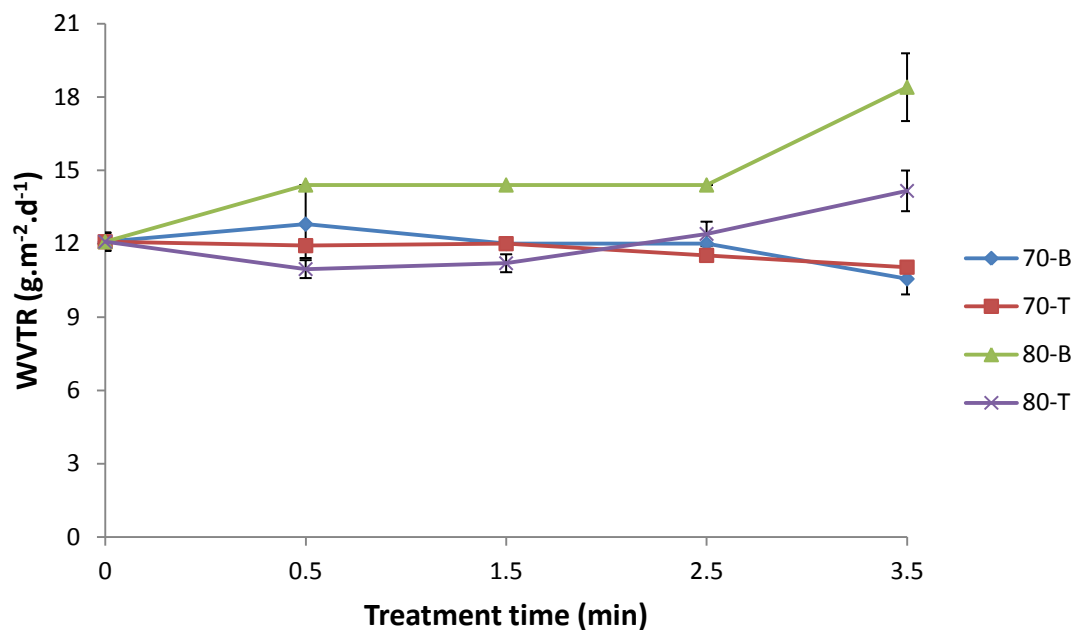


Figure 5: WVTR of treated and untreated PLA films. [AA-T/B=Treatment voltage (kV)-T:top/B:bottom]

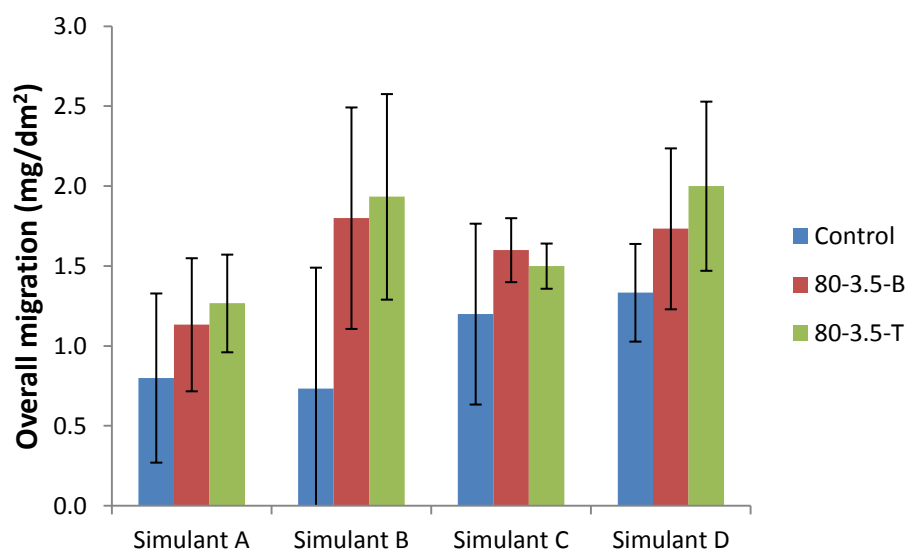


Figure 6: Overall migration (mg/dm<sup>2</sup>) of control and treated PLA films. [AA-BB-T/B=Treatment voltage (kV)-Treatment time (min)-T:top/B:bottom]

Voltage (kV)	Time (min)	Position	R <sub>RMS</sub> (nm)	R <sub>Peak</sub> (nm)	R <sub>Groove</sub> (nm)
70	Control		0.9 ± 0.7 <sup>c</sup>	3.1	4.7
	0.5	Bottom	6.1 ± 5.0 <sup>b,c</sup>	30.9	6.2
		Top	8.1 ± 8.0 <sup>a,b,c</sup>	33.4	16.7
	1.5	Bottom	8.0 ± 12.9 <sup>a,b,c</sup>	45.7	21.6
		Top	9.0 ± 10.7 <sup>a,b,c</sup>	48.2	38.2
	2.5	Bottom	7.2 ± 4.3 <sup>b,c</sup>	37.9	29.8
		Top	9.1 ± 12.4 <sup>a,b,c</sup>	52.0	39.7
	3.5	Bottom	6.1 ± 5.6 <sup>b,c</sup>	21.4	31.2
		Top	12.4 ± 6.6 <sup>a,b,c</sup>	42.5	19.2
	80	0.5	Bottom	4.9 ± 3.0 <sup>b,c</sup>	25.7
Top			8.8 ± 4.7 <sup>a,b,c</sup>	28.8	8.8
1.5		Bottom	4.4 ± 3.8 <sup>c</sup>	18.4	17.4
		Top	8.6 ± 8.4 <sup>a,b,c</sup>	39.9	15.7
2.5		Bottom	9.2 ± 7.5 <sup>a,b,c</sup>	32.2	36.2
		Top	17.6 ± 7.8 <sup>a,b</sup>	52.6	38.8
3.5		Bottom	12.8 ± 12.6 <sup>a,b,c</sup>	55.4	26.1
		Top	20.9 ± 6.6 <sup>a</sup>	51.7	42.5

Table 1: Roughness parameters (mean value ± standard deviation) for control and DBD plasma treated samples (R<sub>RMS</sub>: root mean square roughness, R<sub>Average</sub>: average surface roughness, R<sub>Peak</sub>: Highest peak, R<sub>Groove</sub>: lowest groove)

Voltage (kV)	Time (min)	Position	T <sub>5</sub> (°C)	T <sub>max</sub> (°C)	T <sub>g</sub> (°C)	T <sub>m</sub> (°C)	ΔH <sub>m</sub> (J/g)	
70	Control		295	316	62.1	165.4	29.7	
	0.5	Bottom	295	339	61.7	165.2	30.7	
		Top	333	357	62.1	165.3	29.0	
	1.5	Bottom	283	347	62.2	165.3	30.0	
		Top	334	361	61.7	165.3	32.5	
	2.5	Bottom	315	346	59.9	164.4	29.8	
		Top	339	365	59.3	165.2	29.9	
	3.5	Bottom	332	361	61.1	165.4	29.2	
		Top	326	359	59.6	165.1	31.7	
	80	0.5	Bottom	337	364	61.2	165.9	31.5
			Top	323	352	61.5	164.9	29.8
		1.5	Bottom	337	365	61.8	165.3	31.1
Top			302	361	61.1	165.3	30.6	
2.5		Bottom	337	361	61.7	165.2	31.5	
		Top	317	365	61.8	165.5	31.4	
3.5		Bottom	330	359	61.3	165.1	30.6	
		Top	314	357	61.8	165.9	31.2	

Table 2: TGA and DSC parameters for PLA samples

**Highlights**

- Evaluated the effects of in-package atmospheric DBD plasma on polylactic acid film.
- Changes in PLA were characterized to assess its suitability for food packaging.
- AFM, DSC, TGA, XRD, FTIR, OTR, WVTR and overall migration were studied.
- PLA is a suitable material for in-package DBD plasma treatment of foods.