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Physicochemical Characterization of Argon Plasma-Treated Starch Film

Zh. Sheikhi[1], L. Mirmoghtadaie[1], M. R. Khani[2], M. Farhoodi[1], S. Beikzadeh[1], Kh. Abdolmaleki[3], F. Kazemian-Bazkiaee[1], B. Shokri[4], and S. Shojaee-Aliabadi[1*]

ABSTRACT

Cold plasma is a novel non-thermal technology for the food and packaging industry. In this study, the effects of argon glow discharge plasma on the mechanical properties, surface topography, chemical composition, film hydrophilicity, film solubility, and barrier properties of the starch films were examined. Plasma treatment improved Tensile Strength (TS) of the starch film. In contrast to TS, elongation at the break of the plasma-treated films remained unchanged. The surface roughness of starch film increased after plasma treatment. An apparent increase in the surface hydrophilicity was observed due to formation of oxygen-containing polar groups. FTIR analysis confirmed the increase in the oxygen containing groups in plasma-treated starch film. However, film surface hydrophilicity caused no significant change in the solubility of films. No significant difference was found in the barrier properties of the starch films. The evaluation of films modifications by glow discharge plasma will contribute to in-package decontamination studies of food products by plasma.

Keywords: Biopolymer film, Food packaging, Mechanical properties, Tensile strength.

INTRUDUCTION

Manufacture of the objects from petroleum-derived synthetic plastics creates an unavoidable dependence on nonrenewable resources. These objects present negative impacts on the environment due to their large volume and their long decomposition time. Thus, there is great interest in developing biopolymer from natural resources (Florez *et al.*, 2019). Biodegradable films have been mostly produced from edible polymeric materials including proteins, polysaccharides and lipids. Polysaccharides such as starch, chitosan, pectin, cellulose derivatives, alginate and carrageenan are used as the main materials for their remarkable potential to form excellent films (Rachtanapun *et al.*, 2010). Starch is the most promising biopolymers for this purpose due to its complete biodegradability, worldwide availability, low cost (Bastos *et al.*, 2013) and the capability of forming a continuous matrix. Nevertheless, the technological application of starch is restricted by its poor mechanical properties and inherent hydrophilicity (Ghanbarzadeh *et al.*, 2011).

Chemical and physical methods were used to overcome these shortcomings. Chemical modification such as cross-linking gives high efficiency in this respect. However, the issues

[1] Department of Food Science and Technology, National Nutrition and Food Technology Research Institute, Faculty of Nutrition Sciences and Food Technology, Shahid Beheshti University of Medical Sciences, Tehran, Islamic Republic of Iran.
[2] Laser and Plasma Research Institute, Shahid Beheshti University, G. C. Zip Code: 19839-63113Evin, Tehran, Islamic Republic of Iran.
[3] Department of Food Science and Technology, School of Nutrition Science and Food Technology, Kermanshah University of Medical Science, Kermanshah, Islamic Republic of Iran.
4 Department of Physics, Shahid Beheshti University, Tehran, Islamic Republic of Iran.
*Corresponding author, e-mail: s_shojaee@sbmu.ac.ir

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of safety, high cost, and environmental contamination by some of the cross-linking agents have limited their potential applications (Chaiwat *et al.,* 2016; Fazeli *et al.,* 2019). On the contrary, physical methods, especially a novel approach such as cold plasma treatment, could overcome the drawbacks of chemical modifications. Plasma, a quasi-neutral ionized gas, is primarily composed of ions, photons, and the free electrons as well as atoms in its fundamental or excited states with a net neutral charge. It is simply induced when supplying suitable power and frequency of energy to gas under appropriate gas type, flow, and pressure (Chaiwat *et al.,* 2016). Glow discharge plasma is a low-pressure plasma, which is generated at room temperature. It is the most common type of plasma used in materials modification. Therefore, using the glow discharge plasma is proposed for the modification of natural polymers such as starch (Zhang *et al.,* 2014). Many investigations have been conducted about using plasma technology for producing a non-hydrophilic starch film. Plasma polymerization can protect the starch-based materials against humidity changes with a thin polymeric layer (Andrade *et al.,* 2005). Several plasmas such as Sulfur hexafluoride (SF6), 1-butene, Hexamethyldisilazane (HMDSN) and Hexamethyldisiloxane (HMDSO) have been utilized for creating a hydrophobic surface in the starch film (Andrade *et al.,* 2005; Bastos *et al.,* 2013; Bastos *et al.,* 2009; Behnisch *et al.,* 1998; Santos *et al.,* 2012).

Utilization of helium plasma has been explored by Hwang *et al.* (2005) who found that etching and cross-linking reactions improved the mechanical properties of polypropylene nonwoven fabrics. Inert gases like helium and argon can be used for these purpose, however, argon plasma has been shown to be more effective (De Geyter *et al.,* 2007). To the best of our knowledge, there is no investigation on the improvement of mechanical properties of the starch film using plasma treatment. Therefore, the objective of this study was to characterize the effects of argon glow discharge plasma treatment on the physicochemical properties of starch films.

MATERIALS AND METHODS

Starch Film Preparation

Starch film was prepared according to the method proposed by Ghanbarzadeh *et al.* (2011) with a minor modification. Briefly, starch solution (4% w/v) was prepared by dispersing 4 g of wheat starch in 100 mL distilled water and heating it at 90ºC for 30 minutes followed by stirring until it was gelatinized. Afterwards, glycerol (2 mL) was used as a plasticizer added to the starch solution. Then, 80 mL of the sample was cast onto teflon dishes (14 cm diameter), and dried in a ventilated oven at 40°C for 18 hours. Dried film with 0.18 µm thickness was peeled off.

Plasma Treatment

The plasma treatment of starch films was carried out using a double-electrode capacitively coupled radio-frequency (20 kHz) cold plasma (AC glow discharge) (Figure 1). The system was equipped with a rotary vacuum pump contained in a quartz chamber with a 20 cm diameter, which was evacuated to the base pressure of 600 mTorr. Samples were put on the holder and then plasma treatment was performed with 99.999% pure argon. The argon flow rate was 5 sccm (standard cubic centimeters per minute), constant in all experiments. Samples were treated for various durations (4, 8, and 12 minutes). Power was applied in continuous wave mode (20-30W).

Mechanical Properties

Mechanical properties of the film samples including Tensile Strength (TS) and Elongation at break (E) were determined according to ASTM standard method D882

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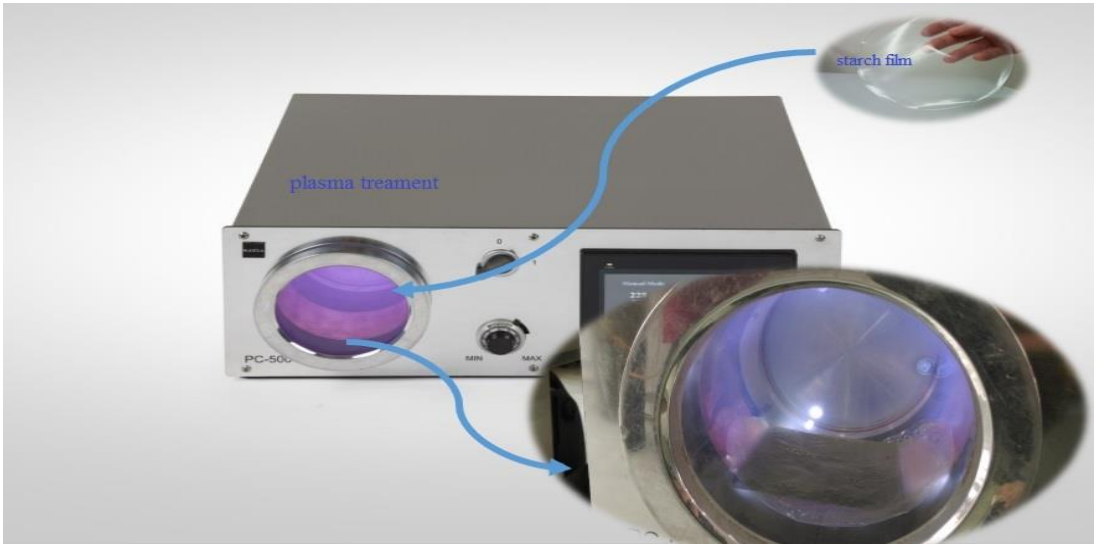

Figure 1. Experimental schematic for argon glow discharge plasma treatment of starch films.

(2009) by a Testometric Machine M350-10CT (Testometric Co., Ltd., Rochdale, Lancs., England) at 25°C. Before testing, film specimens (1.5×10 cm) were equilibrated in desiccators containing saturated solution of $Mg(NO_3)_2$ at 25°C and 53% relative humidity for 48 hours. Afterwards, equilibrated film strips were mounted between the tensile grips. Initial grips separation was 50 mm and cross-head speed was 50 mm min^{-1}. The force was applied until the starch layer was fractured. For each film, at least three samples were analyzed.

Atomic Force Microscopy (AFM)

Surface topography of the films before and after argon plasma treatment was determined by atomic force microscopy (AFM Easy Scan 2, Nanosurf Co. Liestal, Switzerland). A sharpened cantilever was positioned over the sample, and 5×5 µm images were taken. A minimum of three images of different zones were captured and analyzed to transform into a three-dimensional image and to calculate the roughness values. Two statistical parameters related with sample roughness were determined: Sa (average of the absolute value of the height deviations from a mean surface), and Sq (root-mean-square average of height deviations taken from the mean data plane) (Rahmani *et al.*, 2017).

Attenuated Total Reflection -Fourier Transform Infrared (ATR-FTIR) Spectrophotometer

FTIR-ATR spectra of starch films (untreated and plasma-treated samples) were recorded on a Bomem FTIR spectrophotometer (Model: SIG 1100G, Canada) within a range of 450 to 4,000 cm^{-1} using a resolution of 4 cm^{-1}. All measurements were carried out at the room temperature with 20 scans for each sample (Honarvar *et al.*, 2017).

Contact Angle

The static contact angle of all the starch films was measured using contact angle analyzer (OCA 15 plus; Data physics Instruments, Filderstadt, Germany) with sessile drop technique, at room temperature. A drop of distilled water (3 µl) was uniformly placed on the starch film surface. The drop image was captured by a

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computer-connected camera. At least three different measurements for each sample were obtained (Honarvar *et al.*, 2017).

Solubility

A square film sample (2×2 cm) was cut from each film, dried at 110°C for 24 hours in a ventilation drying oven. After drying, films were weighed exactly to ±0.0001 g to determine the initial dry Weight (W_o, expressed as dry matter). Film specimens were placed in 50 mL of distilled water, capped, and stored at 25ºC for 24 hours. Finally, film pieces were extracted and dried at 110ºC to constant Weight (W_f, desiccated undissolved film). The Water Solubility (WS%) of the films was calculated using the following equation in triplicate.

$$WS\ (\%) = ((W_o - W_f)/W_o) \times 100 \quad (1)$$

Water Vapor Permeability (WVP)

Water vapor permeability was determined gravimetrically using the ASTM E96 method (2005). Summarily, 50 g of anhydrous calcium chloride (0% RH, assay cup) was placed in each test jar to establish dry conditions. Untreated and plasma treated film samples (0.00287 m^2 film area) were used to seal test jars. After the films were mounted, the whole assembly was weighed and placed in desiccators at a set temperature of 24±1°C and relative humidity of 75% with a saturated solution of sodium chloride. The assembly was weighed with an accuracy of 0.0001 g. WVP was determined using the following equation.

$$WVTR = S/A \quad (2)$$

$$WVP = (WVTR \times X)/\Delta P \quad (3)$$

Where, WVR is Water Vapor transmission Rate, S is the Slope of the weight gain vs. time plot, A is the film Area, X is the thickness of the films and ΔP is the Pressure difference between the inner and outer surfaces. Each test was made in three replicates.

Oxygen Rate Transmission

Oxygen Permeation Analyzer (Brugger, Germany) was used to determine Oxygen transmission rate of the films. Untreated and 12-minutes treated films were cut into the circular sample with 14 cm diameter and clamped in the diffusion chamber at 25ºC. Nitrogen was injected into the lower half of the chamber where an oxygen sensor was placed, and pure oxygen (99.9%) was introduced into the upper half of the chamber. The oxygen volumetric flow rate per unit area of the membrane and per time (OTR, $cm^3\ m^{-2}\ d^{-1}$) was continuously monitored until a steady state was reached. All samples were analyzed in triplicates, OTR*e values (cm^3 mm $m^{-2}\ d^{-1}$) were calculated based on film thickness and were used for comparison (Pankaj *et al.*, 2017).

Optical Properties

The color of the starch films was determined in terms of Lightness difference (ΔL), redness difference (Δa), and yellowness difference (Δb) using the colorimeter (colorimeter TES–135 A, Taiwan). The measurements were performed in triplicate for each sample. Total color difference (ΔE) was calculated using the following equations:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (4)$$

Where, ΔL, Δb, and Δa are the color parameter values of the film (Rahmani *et al.*, 2017).

Statistical Analysis

SPSS statistical software (version 16, SPSS Inc) was used to perform the statistical analysis of the data. Analysis Of Variance (ANOVA) followed by Duncan's multiple range test was performed to determine significant differences among the treatments at 95% confidence level. For comparison of two treatment means, t-test was used.

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RESULTS AND DISCUSSION

Mechanical Properties

Tensile strength of the starch film increased when it was treated with argon plasma, while no significant difference was observed in elongation at the break (Table 1). This increase in the tensile strength may be due to introducing potential cross-links on the film surface after plasma treatment. Ions and UV photons induced by plasma as energetic species can destroy C–H or C–C bonds that resulted in the formation of carbon radicals. The reaction of formed radicals with polymer chains can create cross- link bridges in the polymer matrix (De Geyter *et al.,* 2007). Also, etching could improve the film´s tensile strength, because etching eliminated some of the defects on the surface that would cause premature failure. Hwang *et al.* (2005) also reported that the increase in the roughness of fiber surfaces can improve the interfacial strength (TS) of fibers after plasma treatment through etching (Qiu *et al.,* 2002). As shown in Table 1, the values of the tensile strength increased more with longer plasma exposure due to the longer duration of chemical interactions between plasma-induced radicals and starch film surface.

In contrast to TS, elongation remained unchanged at the break of the plasma treated films. It was reported that degradation of the polymer due to branch scissions created low molecular weight organic molecules, which could enhance the free volume within the polymer network, subsequently increasing the flexibility of the polymer. (Oh *et al.,* 2016). However, no significant increase in %E induced by degradation was observed in this study. Percentage of E may have been decreased to compensate for increased TS of the starch film during plasma treatment.

Surface Topography

Figure 2 shows the effects of the argon plasma treatment on the starch film morphology at maximum exposure time. The untreated starch film is relatively smooth as compared with treated film with Sq and Sa values of 17.4 and 13.7 nm, respectively. The treated film was rougher compared to unprocessed starch film. This increase in the surface roughness was due to the etching effect on the surface caused by active species such as radicals, ions, electrons and UV-vis radiations generated in the plasma (Pankaj *et al.,* 2015b). These active species can cause chemical etching by bond breakage, chain scission and chemical degradation or physical etching by removal of low molecular weight fragments; argon was reported to be more powerful in the physical etching (Gomathi and Neogi, 2009; Pankaj *et al.,* 2015b). This increased roughness is in agreement with the results obtained by Pankaj *et al.* (2015) who exposed the starch film to dielectric barrier discharge atmospheric plasma. They also reported that surface roughness increased with increasing plasma treatment time.

ATR-FTIR

ATR-FTIR spectra of the argon plasma

Table 1. Physical and mechanical properties of plasma-treated starch films.[a]

Sample	Tensile strength (MPa)	Elongation at yield (%)	Contact angle (°)	WVP [b]	Solubility (%)
Untreated	3.05±0.00[d]	17.38±0.50[a]	54.15±0.14[a]	1.81 ± 0.98[a]	43.25 ± 2.57[a]
4-Min treated	4.07±0.02[c]	17.97±0.25[a]	39.80±0.48[b]	2.38 ± 0.31[a]	42.08 ± 2.93[a]
8-Min treated	4.68±0.04[b]	17.27±0.28[a]	36.18±1.14[c]	2.16 ± 0.37[a]	42.16 ± 0.36[a]
12-Min treated	5.90±0.14[a]	17.09±0.88[a]	32.97±1.27[d]	2.34 ± 0.21[a]	43.27 ± 0/96[a]

[a] Data reported are average values±standard deviations. Values within each column with different letters are significantly different in each section ($P< 0.05$). [b] The unit of WVP was g Pa^{-1} s^{-1} $m^{-1}\times10^{-13}$.

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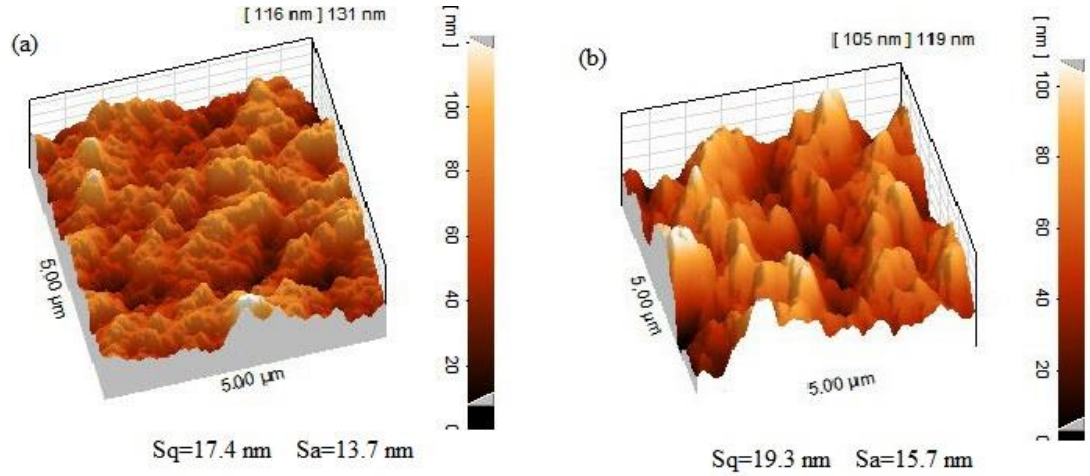

Figure 2. AFM images of the starch film surface: (a) Untreated, and (b) Argon plasma treated for 12 minutes.

treated starch film for 12 minutes and the untreated specimen are shown in Figure 3. The effect of the argon plasma can be observed by the decrease in the absorbance of the peak corresponding to C-H stretching (2,919 cm^{-1}) and also deformation of C-H bond (2,851 cm^{-1}) (Gomathi and Neogi, 2009). This leads to an increase of C-O-C and C-O, which can be observed from the spectrum at 1,043 and 995 cm^{-1}, respectively. Energetic ion bombardment breaks the C-H bond and creates an active site on the polymer surface. When the treated polymer is exposed to the atmosphere, it can bind to other atoms or molecules in the air, especially oxygen, and form oxygen-containing groups on surfaces (Lai *et al.*, 2006). The formation of these oxygen-containing groups on the surface may show the development of cross-linking in the treated film surface (Wongsagonsup *et al.*, 2014). However, a slight decrease was observed at 1,726 cm^{-1} absorption that may indicate possible involvement of carbonyl groups in hydrogen bonding. As can be seen, the intensity of the hydrogen bond (about 3,030 cm^{-1}) increased after plasma treatment.

Contact Angle

The effects of argon plasma treatment on the hydrophilicity of starch film are shown in Figure 4. A significant difference was observed in contact angle after plasma treatment for all treatment times (Table 1). This increase was due to a rise in the polar groups on the film surface (Pankaj *et al.*, 2015b). This result can be explained with FTIR analysis that shows the generation of functional groups containing oxygen on the film surface after plasma treatment. Morent *et al.* (2011) observed that argon plasma treatment increased the wettability of polypropylene due to the generation of oxygen-containing polar groups and reported that a high degree of oxidized three-dimensional cross-linked structures was introduced by argon plasma treatment. As seen, the surface of the starch film revealed more hydrophilicity with increasing exposure time due to the longer duration of plasma-substrate interaction.

Solubility

Film solubility is an essential property for deciding the packaging utilization of any polymeric film. Water solubility is a sign of hydrophilicity of the film (Kim and Ustunol, 2001). Chemical compositions and cross-linked network on the surface of the polymer affect the film's hydrophilicity. Although chemical compositions such as the presence

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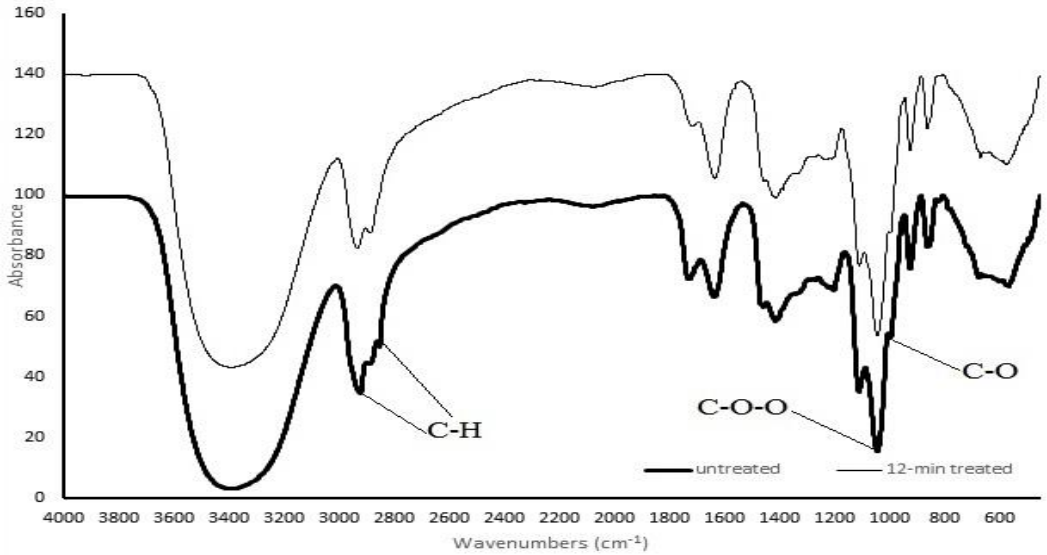

Figure 3. FTIR spectra of untreated and argon plasma treated starch films.

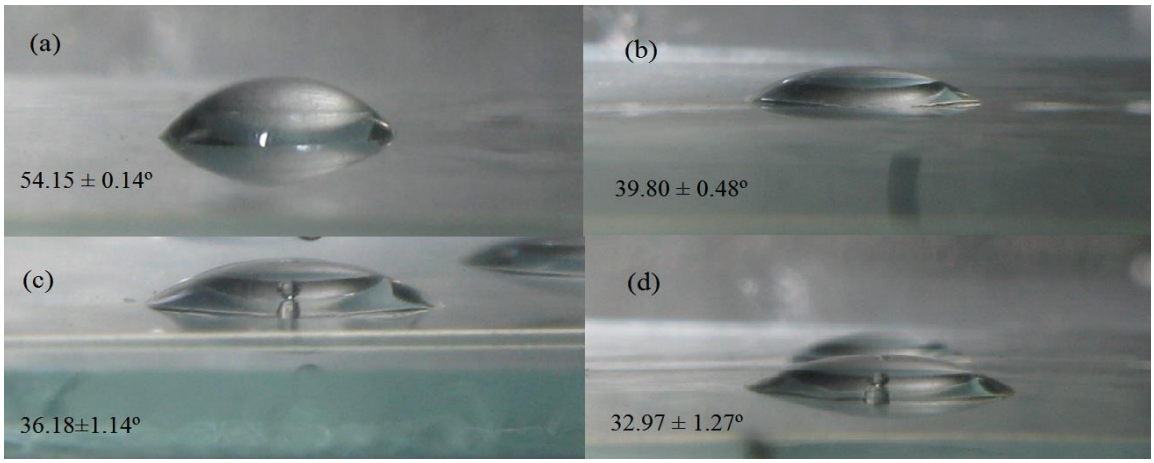

Figure 4. The contact angle: (a) Untreated; (b) 4-Minutes treated; (c) 8-Minutes treated, and (d) 12- Minutes treated starch films.

of polar groups increased by plasma treatment, no significant change was observed in the solubility of films even at different treatment times (Table 1). This can be attributed to the formation of a stable cross-linked network on the surface of the polymer by argon plasma treatment. The solubility of the film is highly influenced by the amount of cross-linking agents because solubility tends to decrease with more cross-linking on the film's structure (Mei *et al.*, 2013). These two parameters neutralize each other. Therefore, in this study film solubility remained unchanged.

Water Vapor and Oxygen Permeability

Barrier property of food packaging materials is an important parameter that determines the suitability of polymeric films for packaging applications. Water vapor permeability of untreated starch film was 1.81±0.96 g Pa^{-1} s^{-1} $m^{-1}\times10^{-13}$. WVP did not show any significant difference after plasma

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treatment for all treatment durations. The Oxygen Transmission Rate (OTR*e) of the untreated film was 0.26±0.03 cm^3 mm m^{-2} d^{-1}, while this parameter was 0.34±0.08 cm^3 mm m^{-2} d^{-1} for 12-minutes plasma treatment. Therefore, no significant difference (P> 0.05) was observed in OTR*e. It should be noted that water vapor permeability depends on bulk mechanisms such as vapor pressure and concentration gradient between the two surfaces and gas permeability relates to a combined effect of diffusion and solubility of the permeate molecules, which is transported via voids of gaps present in the segments of a polymer chain (Pankaj *et al.*, 2015a). These results show that argon glow discharge plasma treatment did not influence the bulk properties of the polymer.

Optical Properties

Hunter-Lab color was measured to assess the optical properties of untreated and plasma-treated starch films. Values ΔL, Δa, Δb, and the total color difference (ΔE) were examined (Table 2). No significant difference (P< 0.05) was observed in optical properties even after argon plasma treatment at a maximum time, which agreed with visual observations. Thus, argon glow discharge plasma treatment modified starch film without adverse effects on its optical properties.

CONCLUSIONS

Argon glow discharge plasma treatment of starch film led to an increase in the oxygen-containing groups in the surface, which is confirmed by Fourier transform Infrared spectroscopy. This increase resulted in a noticeable increase in the film hydrophilicity. However, no significant change was observed in water solubility of the starch film after plasma treatment. Barrier properties of the plasma-treated starch films remained unchanged. Tensile strength increased by plasma treatment, possibility due to cross-linking and etching effects. Also, etching processes changed surface morphology and increased surface roughness. Therefore, argon glow discharge plasma treatment has the potential to improve the properties of the starch film, hence enhancing their applicability as food packaging materials. This modified starch film could be used in packaging of food with low water activity such as bakery products.

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Table 2. Effect of argon glow plasma on optical properties on starch films.[a]

Sample	ΔL	Δa	Δb	ΔE
Untreated	89.99 ± 0.85[a]	-6.48 ± 1.31[a]	9.51 ± 0.71[a]	90.73 ± 0.82[a]
4-Min treated	90.73 ± 0.43[a]	-5.55 ± 0.43[a]	9.13 ± 0.32[a]	91.36 ± 0.43[a]
8-Min treated	90.73 ± 0.32[a]	-7.37 ± 1.34[a]	9.79 ± 0.43[a]	91.55 ± 0.16[a]
12-Min treated	90.44 ± 1.47[a]	-6.35 ± 0.38[a]	9.78 ± 0.28[a]	91.18 ± 1.44[a]

[a] Data reported are average values±standard deviations. Values within each column with different letters are significantly different in each section (P< 0.05).

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ویژگی‌های فیزیکوشیمیایی فیلم نشاسته تیمار شده با پلاسما آرگون

ژ. شیخی، ل. میرمقتدایی، م. ر. خانی، م. فرهودی، س. بیک‌زاده، خ. عبدالملکی، ف. کاظمیان-بازکیائی، ب. شکری، س. شجاعی-علی‌آبادی

چکیده

پلاسمای سرد یک تکنولوژی غیرگرمایی جدید برای صنعت غذا و بسته‌بندی می‌باشد. در این مطالعه اثرات پلاسما تخلیه تابشی آرگون بر روی ویژگی‌های مکانیکی، توپوگرافی سطحی، ترکیبات شیمیایی، آبدوستی و حلالیت فیلم و ویژگی‌های ممانعتی فیلم نشاسته مورد بررسی قرار گرفت. تیمار پلاسما سبب بهبود مقاومت کششی فیلم نشاسته شد. برخلاف TS، افزایش طول تا نقطه شکست فیلم‌های تیمار شده با پلاسما بدون تغییر باقی ماند. زبری سطح فیلم نشاسته بعد از تیمار با پلاسما افزایش یافت. افزایش چشم گیری در آبدوستی فیلم ها به علت شکل گیری گروه های قطبی حاوی اکسیژن مشاهده گردید. آنالیز FTIR افزایش گروه‌های حاوی اکسیژن در فیلم نشاسته تیمار شده با پلاسما را تائید کرد. با وجود افزایش آبدوستی سطحی تغییر معنی داری در حلالیت فیلم ها ایجاد نشد. تغییر معنی داری در ویژگی های ممانعتی فیلم ها ایجاد نشد. ارزیابی اصلاحات سطحی با پلاسما تخلیه تابشی نقش قابل توجهی در مطالعات آلودگی‌زدایی از بسته‌بندی مورد استفاده در محصولات غذایی با استفاده از پلاسما خواهد داشت.

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