Edible films and coatings based on mango (var. Ataulfo) by-products to improve gas transfer rate of peach

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ABSTRACT

Fruit waste and by-products are economical materials for the development of biodegradable and active packaging. The aims of this study were to develop, characterize and evaluate biodegradable coatings and films by using mango peel and antioxidant extracts of seed kernel. The proximate composition of peel was also determined. Structural, barrier, optical and antioxidant properties were analyzed in the films. Gas transfer rates and the ethylene production in peach were evaluated. Edible films formulated with mango peel showed good barrier properties, with the water vapor permeability varying from 0.88×10^{-10} - 1.00×10^{-10} g m⁻¹ s⁻¹ Pa⁻¹. The addition of antioxidant extract does not show a significant effect ($p > 0.05$) on optical properties. Furthermore, antioxidant activity and polyphenol content increased by 18% and 60% respectively. Peach coated with a solution of mango peel (1.09%), antioxidant extract of mango seed kernel (0.078 g L⁻¹) and glycerol (0.33%) showed 64% and 29% less ethylene and CO₂ production, respectively, and 39% less O₂ consumption when compared with peaches without coating. The reduction in gas transfer ensures the greater extension of the shelf life of fruit treated. By-products of mango may thus be suitable for the production of low-cost biodegradable and active packaging.

1. Introduction

Nowadays, short shelf life of fruit and vegetables is one of the biggest trading problems. Although packaging plays a decisive role in the improvement of the shelf life, the high accumulation of plastic packing materials has generated a growing concern in the world, as only 5% of the production of plastics are recycled (Espitia et al., 2014). In this context, the biodegradable edible coatings or films are an alternative to the replacement of synthetic packaging (Azeredo, Rosa, Souza, & Waldron, 2014). An edible coating or film is defined as primary packaging made from edible components; in which the layer of edible material may be used in liquid form to directly coat the food or formed as a solid sheet in the form of a film (Galus & Kadzińska, 2015). Some of their main functions are to protect the produce from mechanical, physical and chemical damages, and also of microbial contamination and the reduction in the transfer of gases (Falguera, Quintero, Jiménez,

Muñoz, & Ibarz, 2011). Also, bioactive ingredients as antioxidants can be incorporated into the matrix, thus enhancing functional attributes of the formulation (de Moraes, Haas, de Oliveira, & Hickmann, 2016).

A variety of polymers such as starch (Chiumarelli & Hubinger, 2014), pectin (Taqui & Stamatina, 2014), chitosan (Elsabee & Abdou, 2013), galactomannans (Cerqueira et al., 2011), alginate (Guerreiro, Gago, Faleiro, Miguel, & Antunes, 2015), proteins (Belyamani, Prochazka, Asseztat, & Debeaufort, 2014), and wax (Ochoa et al., 2011) have been used in the formulation of edible coatings or films. Therefore, the research and characterization of new biopackings based on unconventional sources is a trend (Falguera et al., 2011), highlighting renewable materials that are abundant in Nature (Cazon, Velazquez, Ramírez, & Vazquez, 2017). In this context, fruit by-products present potential benefits such as the reduction of manufacturing costs, the generation of added value and the enhancement of functional properties (Ayala et al., 2011; Schieber, Stintzing, & Carle, 2002; Varzakas,

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Zakynthinos, & Verpoort, 2016). By-products of mango processing industry are an example of by-products that can be used for the formulation of biodegradable packaging. The peel has a high content of polysaccharides (Serna, García, & Torres, 2016), while the seeds of mango have high content of bioactive compounds (Abdalla, Darwish, Ayad, & El-Hamamy, 2007; Abdullah, Mohammed, & Abdullah, 2014; Dorta, González, Lobo, Sánchez-Moreno, & de Ancos, 2014; Ribeiro & Schieber, 2010), with important antioxidant properties (Torres et al., 2016). One of the most important parameters to consider when evaluating the potential of a biomaterial in the formulation of edible films is their barrier properties (Cazon et al., 2017). Furthermore, the wettability and respiratory rate on a model fruit are of vital importance to determine the feasibility in the application of edible coatings (Cerqueira, Lima, Teixeira, Moreira, & Vicente, 2009). A fruit model with suitable characteristics to evaluate edible coatings is the peach (*Prunus persica*), as it has a fast maturation rate once harvested (Guillén et al., 2013). Therefore, the objectives of this work were: (a) to characterize edible films of mango peel added or not with seed extracts in terms of their barrier, optical and antioxidant properties, and (b) to evaluate the application of edible coatings formulated with peel and seed extracts of mango in the respiratory rate, ethylene production and surface properties of fresh peaches.

2. Materials and methods

2.1. Chemicals

Glycerol, 2,2-diphenyl-1-picrylhydrazyl (DPPH), Folin-Ciocalteu reagent, gallic acid (GA) were purchased from Sigma Chemical Co. (St. Louis, MO, USA.).

2.2. Mango by-products

Mangos (*Mangifera caesia*, cv. 'Ataulfo') were obtained from a local market in Saltillo, Coahuila, Mexico in March 2015. These were selected in commercial ripening stage and caliber number 16 (NOM-188-SCFI, 2012). The mangoes were used soon after the purchase, the pulp was manually separated from the peel and seed.

2.2.1. Obtaining mango peel flour

In order to obtain the mango peel flour (MPF), mango peel was dried at 60 °C in an oven (Napco Model 322) for 48 h and subsequently ground in a mill (Pulver 100MINI, México).

2.2.1.1. Chemical composition of MPF. The flour was analyzed for its content in ash (A) at 550 °C for 8 h crude protein content (CP) was conducted by the Kjeldahl method with a conversion factor of 6.25, and lipids content (L) (ether extract by the Soxhlet system) as described by the AOAC (AOAC 920.39, 1990). The content of soluble and insoluble fiber was evaluated by the gravimetric enzymatic methods (AOAC 991.43, 1993). Cellulose, hemicellulose and lignin contents were calculated by determining neutral detergent fiber and acid detergent fiber by the method of Van Soest, Robertson, and Lewis (1991). Total carbohydrates (Tc) were calculated using the following equation (1):

$$Tc = 100 - \%A + \%CP + \%L \quad (1)$$

where Tc represents the total carbohydrates, %A represents ash content, %CP represents crude protein content and %L lipids content.

2.2.2. Obtaining extracts of the mango seed kernel

Antioxidants were extracted from the seed kernel according to the methodology reported by Torres, Rojas, Serna, Belmares, and Aguilar (2017a). Briefly, 1 g of kernel was homogenized on a vortex tube mixer with 90% ethanol; the extraction conditions were: kernel weight-to-solvent volume ratio 1:50 (w/v), temperature of 75 °C and 2 extraction cycles using microwave (MARS 6-USA). The extract was used for its

high antioxidant potential (Torres et al., 2017a).

2.3. Films and coatings preparation

The filmogenic solution was prepared following the method of Fai et al. (2016), with some modifications. Briefly, MPF was used at a concentration of 1.09% (w v⁻¹) and glycerol 0.33% (w v⁻¹), which were chosen based on preliminary tests (data not shown). In order to form the films/coatings, the MPF (particle size < 125 µm) was dissolved in a citrate buffer solution (pH 4.5) with constant agitation (Corning pc-220, USA) at 250 rpm for 45 min at 70 °C and filtered through polyester cloth meshes. Subsequently, glycerol was added to the solution and kept under constant agitation at 70 °C and according to the treatment was added or not the antioxidant extract (E). Finally, the temperature was lowered abruptly at 5 °C using an ice bath. In summary, two emulsion-based edible films and coatings were obtained, F: containing MPF (1.09%) and glycerol (0.33%), and FE: containing MPF (1.09%), glycerol (0.33%) and extract of mango seed (0.078 g L⁻¹). The concentration of antioxidant extract was selected according to a previous study of our research group (Torres et al., 2017a), and corresponds to the concentration of extract required to reduce the initial concentration of DPPH radicals by 50% (IC₅₀: 0.078 g L⁻¹).

The film was produced from the formulation F and FE by casting technique (30 mL), which were dispersed in polystyrene plates (9 cm) and dried at 50 °C for 16 h. The films were removed manually from the plates (Fig. 1) and stored at 20 °C in a desiccator at 50% relative humidity (RH) for at least 24 h. The analysis was performed immediately after.

2.4. Evaluation of edible films

2.4.1. Film thickness

The film thickness was measured with a digital micrometer (No. 293-561, Mitutoyo, Japan). Five thickness measurements were taken on each testing sample in different points (Razavi & Zahedi, 2015), and the mean values (n = 6) were used to calculate permeability.

2.4.2. Water vapor permeability (WVP)

The WVP was performed gravimetrically according to the method ASTM E96-92 (ASTM E96-92; 1990) with some modifications. Films were sealed on the top of a permeation cell containing silica (0% RH; 0 Pa vapor pressure at 20 °C), permeation cells were placed in a desiccator at 20 °C and 100% RH (2337 Pa vapor pressure). The cells were weighted at 1 h intervals for 6 h. Steady-state and uniform water pressure conditions were assumed by keeping the air circulation constant. The WVP (g m⁻¹ s⁻¹ Pa⁻¹) of the films was determined by the following Equation (2).

$$WVP = \frac{WVTR \times L}{\Delta P} \quad (2)$$

where WVTR (g m⁻² s⁻¹) is the water vapor transmission rate through the film calculated from the slope of the curve divided by the film area;

Fig. 1. Edible films based on mango by-products: (A) peel-based film and (B) peel-based film with antioxidants of mango seed kernel.

L (m) is the mean film thickness; ΔP (Pa) is the partial vapor pressure difference across the two sides of the film.

2.4.3. Moisture content and water solubility

Film samples (1 cm × 7 cm) were first weighed (M_0) and then placed in an oven at 105 °C, during 24 h until constant weight. After, the films were weighed (M_1) again. Moisture content was determined as the percentage of initial film weight lost during drying and reported on a wet basis with the following equation (Li, Miao, Wu, Chen, & Zhang, 2014):

$$\text{Moisture content} = \frac{(M_0 - M_1)}{M_0} \times 100 \quad (3)$$

The films previously dried at 105 °C and with reported weight (M_1) were immersed in 50 mL of distilled water and placed in a shaker water bath at 25 °C. After 10 min, the samples were filtered through polyester cloth meshes, insoluble film was dried to constant weight (M_2) using an oven at 105 °C to calculate water solubility of the film using the following equation (Cuq, Gontard, Cuq, & Guilber, 1996):

$$\text{Water solubility} = \frac{(M_1 - M_2)}{M_1} \times 100 \quad (4)$$

where M_0 , M_1 and M_2 are the weights of initial, dry matter and insoluble dry matter, respectively. All tests were carried out in triplicate.

2.4.4. Color, light transmission rate, and film transparency

The CIE $L^*a^*b^*$ scale was used to determined L^* (lightness), a^* (redness, greenness) and b^* (yellowness, blueness) color parameters, using a 3nh Colorimeter (NR20XE, China). Light transmission rate and film transparency were measured at 600 nm using UV/visible spectrophotometers (Genesys 20, USA) according to the method reported by Zhang and Han (2006).

$$\text{Transparency} = \frac{A_{600}}{\text{Thickness}} \text{ or } -\frac{\log T_{600}}{\text{Thickness}} \quad (5)$$

where A_{600} and T_{600} are absorbance and transmittance at 600 nm, respectively.

2.4.5. DPPH radical scavenging activity

The reduction of DPPH radical was made following the methodology reported by Molyneux (2004) with some modifications. A solution of DPPH at 60 μM was prepared, 193 μL of DPPH solution was mixed with 7 μL of film solution in each well of a microplate. After 30 min of reaction under dark conditions, absorbance was measured at 517 nm, using a spectrophotometer microplate reader (Epoch, BioTek Instruments, Inc.; Winooski, VT, USA) controlled with the Gen5 Data Analysis software interface. The reduction of the DPPH radical was calculated as a percentage of inhibition by equation (6):

$$\text{DPPH inhibition (\%)} = \frac{(A_c - A_s)}{A_c} \times (100) \quad (6)$$

where A_c is the control absorbance and A_s is the absorbance of the sample.

2.4.6. Total phenolic content

The total phenolic content was estimated by colorimetric assay according to the methodology reported by Wong, Muñiz, Aguilar, Rodríguez, and Aguilar (2014). First, 20 μL of film solution was mixed with 20 μL of Folin-Ciocalteu reagent in a well. After, 5 min, 20 μL of sodium carbonate (0.01 mol L⁻¹) was added to each sample and allowed to stand for 5 min. Then, 125 μL of distilled water was added. Absorbance was read at 790 nm (Epoch, Biotek industries, Highland park, USA). Results were expressed as mg of gallic acid equivalents per g of film solution (mg g⁻¹ GAE), according to a GA standard curve.

2.4.7. Contact angle measurements

The influence of addition of antioxidant extract on the surface polarity of films was studied by means of contact angle measurements. Briefly, one drop of 2 μL of water was placed on the surface of the films and a Contact Angle System OCA15 Plus with CCD video camera (resolution of 752 × 582 pixels) and C20 software was used (Fabra et al., 2016).

2.5. Evaluation of edible coating

2.5.1. Fruit conditioning and coating application

Peaches (*Prunus persica* L. Batch cv. 'Red Heaven') were purchased at the commercial ripening stage from a local market in Braga, Portugal in April 2016. The fruits were selected in maturity ripe stage, with uniform weight (88.53 ± 5.14 g) and with no physical damage. Fruits was immediately transported to the industry and processes laboratory. Subsequently, the fruit were washed and disinfected (500 g L⁻¹ sodium hypochlorite) for 15 min followed by washing with distilled water and then surface dried at room temperature.

The coating application was conducted by immersing peaches in the following treatments: F, FE and distilled water (control) for 10 min, then immediately dried under air flow (25 °C) until solidification (Approximately 15 min) of edible coating (Guillén et al., 2013). Small portions of the outer surface (1 × 8 cm) of the fruit were cut with a knife and placed on a glass plate in order to measure the contact angle.

2.5.2. The wettability

The wettability was studied by determining the values of the spreading coefficient (W_s) and the works of adhesion (W_a) and cohesion (W_c). The contact angle (θ) of a liquid drop on a solid surface is defined by the mechanical equilibrium of the drop under the action of three interfacial tensions: solid-vapor (γ_{SV}), solid-liquid (γ_{SL}), and liquid-vapor (γ_{LV}). The equilibrium spreading coefficient (W_s) is defined by equation (7) (Rulon & Robert, 1993), and can only be negative or zero.

$$W_s = W_a - W_c = \gamma_{SV} - \gamma_{LV} - \gamma_{SL} \quad (7)$$

$$W_a = \gamma_{LV} (1 + \cos(\theta)) \quad (8)$$

$$W_c = 2 \gamma_{LV} \quad (9)$$

W_s was obtained by measuring the contact angle (θ) between the coating solutions and the peach surface and the surface tension (γ_L) of the coating solutions. The contact angle at the peach surface was measured by the sessile drop method (Carneiro-da-Cunha et al., 2009). Briefly, one drop of 2 μL was placed on the fruit surface and the contact angle was measured with Contact Angle System OCA15 Plus with CCD video camera (resolution of 752 × 582 pixels) and C20 software. Fifteen replicates of contact angle measurements and four replicates of surface tension measurements were obtained at 22 °C.

Surface tension values (γ_L) of each coating solution were determined according to the Ring method described by Gudiña et al. A Krüss K6 tensiometer (Krüss GmbH, Germany) equipped with a 1.9 cm De Noüy platinum ring was used. All the measurements were performed in triplicate at room temperature (20 °C).

2.5.3. Zeta potential

Zeta potential (Z_p) was determined by dynamic light scattering (DLS) with a Malvern Zetasizer, NANO ZS (Malvern Instruments Limited, UK), using a He-Ne laser (wavelength of 633 nm) and a detector angle of 173°. The Z_p values were calculated using the Smoluchowski equation (Hunter, 1988).

2.5.4. Gas transfer rate and ethylene production

Two peaches were placed inside a hermetic jar by treatment. Temperature and relative humidity (RH) were monitored with an iButton Hygrochron Temperature/Humidity Logger (DS1923, USA).

The jar was stored in a controlled chamber (Binder KBF, Germany) at $20 \pm 1^\circ\text{C}$ and RH of $80 \pm 1\%$. Respiration rate and ethylene production were measured every day during eight days, by extracting 500 mL of air inside of the jars with a syringe through a septum fitted in the jar lid (Lima et al., 2010).

2.5.4.1. O_2 and CO_2 transfer rates. The O_2 and CO_2 production/consumption rates were determined using a gas chromatograph (GC Bruker Scion 456, USA) at 100°C with a column Packed SS MolSieve 13x (80/100), $2\text{ m} \times 2\text{ mm} \times 1/8''$ to separate the O_2 and a column megabore BR Q PLOT, $30\text{ m} \times 0.53\text{ mm}$, $0.20\text{ }\mu\text{m}$ (film thickness) to separate the CO_2 and Thermal Conductivity Detector (TCD) at 130°C . Argon at 30 mL min^{-1} and Helium at 15 mL min^{-1} were used as carrier gas. A mixture containing 10% CO_2 , 20% O_2 and 70% N_2 was used as a standard for calibration. All determinations were performed at $20 \pm 1^\circ\text{C}$ for eight days. The O_2 and CO_2 rates were determined as established by Cerqueira et al. (2009), applying Equations (10) and (11), developed for a closed system impermeable to gases.

$$R_{\text{O}_2} = -\left(\frac{dy_{\text{O}_2}}{dt}\right) \cdot \left(\frac{V_f}{w}\right) \quad (10)$$

$$R_{\text{CO}_2} = \left(\frac{dy_{\text{CO}_2}}{dt}\right) \cdot \left(\frac{V_f}{w}\right) \quad (11)$$

where, R_{O_2} is the O_2 consumption rate ($\mu\text{g kg}^{-1}\text{ s}^{-1}$), R_{CO_2} is the CO_2 production rate, ($\mu\text{g kg}^{-1}\text{ s}^{-1}$), w (g) is the weight of the peach, and V_f (L) is the free volume of the container. The free volume V_f of the package was calculated by equation (12):

$$V_f = V_p - \frac{w}{\rho_{\text{peach}}} \quad (12)$$

where, V_p (L) is the total volume of the container, w (g) is the weight of the peach, and ρ_{peach} is the true density of the peach fruit, in this case, 985 g L^{-1} , obtained experimentally following the method described by Zohrabi, Seiedlou, and Alipasandi (2013). The graph of O_2 consumed versus time or CO_2 produced versus time was used to calculate the slopes corresponding to the derivatives, dy_{O_2}/dt (or dy_{CO_2}/dt).

2.5.4.2. Ethylene production. Ethylene production rates were monitored using a gas chromatograph (Varian 3800, USA) with a column megabore Zebtron ZB Wa $\times$ Plus $30\text{ m} \times 0.53\text{ mm}$, $1.00\text{ }\mu\text{m}$ (Injector Split/splitless at 250°C), and flame ionization detector (FID) at 250°C , air at 250 mL min^{-1} , H_2 at 30 mL min^{-1} and makeup gas (N_2) at 30 mL min^{-1} . Helium was used as a carrier gas at a flow rate of 4 mL min^{-1} (60°C). Ethylene (Calgaz, UK) was used as a standard. Ethylene rate was calculated similarly to CO_2 , applying Equation (13).

$$R_{\text{Ethylene}} = \left(\frac{dy_{\text{Ethylene}}}{dt}\right) \cdot \left(\frac{V_f}{w}\right) \quad (13)$$

where, R_{Ethylene} is the ethylene production rate ($\text{ng kg}^{-1}\text{ s}^{-1}$), w (g) is the weight of the peach, and V_f (L) is the free volume of the container. The graph of ethylene produced vs. time was used to calculate the slopes, which correspond to the derivative, dy_{Ethylene}/dt .

2.6. Statistical analyses

The experimental design was completely randomized. All data were expressed as mean values $\pm$ SD. The Student's t -test was run to determine significant differences ($p < 0.05$) in the partial characterization of edible films. The other data were subjected to analysis of variance (ANOVA) ($p < 0.05$) and the mean comparisons were performed using the Tukey's test to examine if differences between treatments were significant ($\alpha = 0.05$). All statistical determinations were performed using Statistica 7.0 software (StatSoft, Tulsa OK, USA).

Table 1

Proximal chemical composition of mango peels variety Ataulfo.

Component	Concentration (%)
Moisture (fresh)	64.06 ± 0.023
Moisture (dry)	5.57 ± 0.153
Ash	4.33 ± 0.138
Protein	5.05 ± 0.014
Lipid	1.78 ± 0.212
Carbohydrates	88.87 ± 0.381
Total dietary fiber	24.35 ± 0.664
Soluble Dietary Fiber	4.17 ± 0.558
Insoluble fiber Dietetics	20.18 ± 0.770
Hemicellulose	5.62 ± 0.565
Cellulose	8.16 ± 0.176
Lignin	6.41 ± 0.417

Values reported are the means $\pm$ standard deviations ($n = 3$).

Values are expressed on dry weight basis.

3. Results and discussion

3.1. Chemical composition of MPF

The total ash, protein, lipid and dietary fiber content in MPF are shown in Table 1. The contents of ash and protein were higher than those previously reported by Rojas et al. (2015) (2.12% and 3.04%, respectively) in Ataulfo mango peel; contrarily, the lipid content was lower (2.35%). On the other hand, the dietary fiber content was similar to that reported in Ataulfo mango peel (23.6%) by García, García, Bello, Sáyo, and Oca (2013). The results of this study were similar to those presented by other authors in mango peels of other varieties (Ajila, Naidu, Bhat, & Rao, 2007b, 2007a). The results evidenced the high content of biopolymers, mainly represented in dietary fiber (24.3%). Previously Serna et al. (2016), reported that pectin is the major component of soluble dietary fiber in the mango peel, this polysaccharide is widely used as a food additive for its thickening, gelling and emulsifying properties (Rojas et al., 2015); therefore, naturally present pectin can favor the use of peel in the formulation of biodegradable films and coatings. As previously reported Andrade, Ferreira, and Gonzales (2014), an available content of carbohydrate higher than 84%, including insoluble dietary fiber content of 80%, is suitable to form biodegradable films. In addition to the reduction in costs, the complete use of the mango peel ensures the use of all the valuable components present in the polymer matrix, e.g. the proteins have beneficial effects in the increase of the mechanical properties, as the amino acids allow forming numerous linkages via disulfide (S–S) covalent bonding, electrostatic forces, hydrogen bonding and hydrophobic interactions (Flores-Lopez, Cerqueira, Jasso, & Vicente, 2016). Meanwhile, lipids contribute significantly to reduce the WVP, and their incorporation in polysaccharide and protein films has the potential to improve the moisture barrier (Pérez-Gago & Rhim, 2014).

3.2. Evaluation of edible films

WVP measures the diffusion of water molecules through the cross-section of the film and can give an estimation of its barrier properties (Acevedo-fani, Salvia-trujillo, Rojas-graü, & Martín-belloso, 2015). In a food product, the transfer of moisture must be reduced to control the loss of moisture to the environment; for this reason, low values of WVP are ideal. The results for WVP of the films tested here are shown in Table 2. FE film presents WVP values significantly higher than those measured for F film. Similar behavior was observed by Bierhalz, da Silva, and Kieckbusch (2012), in films formulated with pectin and added with an antimicrobial compound. The authors associated this behavior to a looser packing of the film macromolecules increasing the free volume of the polymeric structure, which enhances permeability. This behavior may be reflected in a significant increase in the values of

Table 2
Partial characterization of edible films.

	F	FE
WVP ($\times 10^{-10} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$)	0.88 ± 0.02^a	1.00 ± 0.00^b
Thickness (mm)	0.10 ± 0.00^a	0.11 ± 0.01^b
Moisture (%)	3.11 ± 0.84^a	4.14 ± 1.30^a
Solubility (%)	60.24 ± 4.38^a	52.56 ± 9.66^a
Light transmission rate (% T_{600})	26.11 ± 2.36^a	25.20 ± 2.32^a
Transparency ($A_{600} \text{ mm}^{-1}$)	5.83 ± 0.61^a	5.54 ± 0.47^a
L^*	73.11 ± 0.75^a	72.29 ± 0.77^a
a^*	10.39 ± 0.38^a	10.32 ± 0.70^a
b^*	53.13 ± 1.72^a	48.34 ± 0.36^b
DPPH radical scavenging activity (%)	51.90 ± 3.57^b	63.55 ± 3.62^a
Polyphenols ($\text{mg g}^{-1} \text{ GAE}$)	14.67 ± 3.57^b	44.42 ± 4.82^a
Contact angle ($^\circ$)	72.20 ± 1.68^a	68.20 ± 0.90^b

Values reported are the means $\pm$ standard deviations. Statistics were analyzed using the program Student's t-test. Means with different lowercase letters are significantly different ($p < 0.05$). The same letters within the row indicate no significant difference among different samples.

thickness of the formulations added with antioxidant extract (Table 2). However, the WVP of edible films is affected by various factors including the nature of film forming material, type of plasticizer, additive, and film preparation process (Cheng, Wang, & Weng, 2015). The WVP values obtained (0.88×10^{-10} – $1.00 \times 10^{-10} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$) are lower and, consequently, better than those reported by de Moraes et al. (2016), for films obtained with agro-industrial waste (1.5×10^{-10} – $2.8 \times 10^{-10} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$), Pitak and Rakshit (2011) in films made with chitosan, glycerol and banana flour (4.5×10^{-10} – $4.8 \times 10^{-10} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$) and Belyamani et al. (2014) in films of caseinate (1.4×10^{-10} – $1.8 \times 10^{-10} \text{ g m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$).

Moisture content and water solubility of F and FE films are also shown in Table 2. Both parameters were not affected significantly by the addition of antioxidants. Similar behavior was also observed by Bittencourt, Fávoro, Sobral, and Carvalho (2014) in gelatin films formulated with turmeric ethanolic extract, and by Li et al. (2014) in films made with gelatin and added with antioxidant extracts. According to Wu et al. (2013), these solubility values are justified by the increase in the structural strength due to greater interaction between polyphenols and polysaccharides present in the matrix.

The optical properties are very important in the analysis of edible films and coatings, particularly if the film is to be used in a food sensitive to degradation by light (Falguera et al., 2011). As shown in Table 2, the light transmission (measured at 600 nm) of the film with the addition of extract (FE) did not present a significant difference ($p > 0.05$) with respect to F film. Zhang and Han (2006) reported higher results of light transmission and transparency in starch films. This result indicates that the films are a good barrier to radiation in the light spectrum measured. Li et al. (2014) reported that the addition of phenolic compounds decreases the transmission of light (–OH contained in polyphenols compounds). The values obtained in this work can be explained by the content of carotenoids and anthocyanins present in the mango peel, which absorb light at these wavelengths. In the color analysis, it is evidenced that the addition of antioxidant does not affect significantly ($p > 0.05$) luminosity nor a^* values. The high values obtained for b^* reflect the presence of typical yellow coloration of the pigments present in the mango peel.

Active packaging is an attractive technique for extending produce shelf life (Ribeiro-Santos, Andrade, Melo, & deSanches-Silva, 2017). Table 2 showed a significant increase ($p < 0.05$) in the antioxidant activity and the polyphenol content in FE films. Results suggested that the addition of 0.07 g L^{-1} extract potentiated a strongest antioxidant activity. Such effect is due to the phenolic compounds present in the solution; as Dorta, Lobo, and González (2012) demonstrated, polyphenol content is responsible for antioxidant activity in mango by-products.

Contact angle with water measurements allows the prediction of how hydrophobic a surface is, being a higher contact angle ($\theta > 70^\circ$) an indicative of a hydrophobic surface (Belyamani et al., 2014). Results in Table 2 show that films with antioxidant extract had a higher hydrophobicity ($p < 0.05$). The values of contact angle are consistent with the previously reported by Abreu et al. (2015) in films based on native corn starch (45.3 – 82.2°), and were higher than the values reported by Belyamani et al. (2014) in films of sodium caseinate (55°). It is worth highlighting that biopolymers commonly used to produce films usually require expensive pre-treatments (extraction, purification, and separation) blending with other materials, genetic or chemical modification or combinations of the above approaches to improve their barrier, optical and rheological properties (Fai et al., 2016). The results of this study demonstrate the potential of MPF extraction with buffer solutions to produce films with features suitable for use in foods.

3.3. Evaluation of edible coatings

Peach (*Prunus persica*) is a highly perishable produce item that is classified as a climacteric fruit and has a short storability because it undergoes rapid ripening (Guillén et al., 2013). Thus, it was used as a climacteric fruit model to study the effect of mango peel-based edible coatings.

3.3.1. Wettability

W_s is one of the most important properties when evaluating the capacity of a solution to coat a surface of interest; values close to zero are considered most suitable for coating of a surface (Carneiro-da-Cunha et al., 2009). The values of W_s are shown in Fig. 2, from 0 to 120 s after the coating was applied on the surface of peaches. It is clear that after 60 s of adding the coating, W_s values remained constant. The addition of antioxidant extracts increased coatings' wettability, without having a significant effect with respect to F film. The addition of antioxidant extract to the coating forming solution reduced significantly the surface tension of the liquid: the solution without extract (F) showed a surface tension of $57.82 \pm 0.36 \text{ mN m}^{-1}$, whilst with antioxidant (FE) surface tension was $52.06 \pm 0.47 \text{ (mN m}^{-1})$. These values were lower than those calculated for water ($69.80 \pm 0.00 \text{ mN m}^{-1}$). The positive effect of reducing the surface tension contributes to reducing significantly the cohesive forces ($F = 115.64 \pm 0.73 \text{ mN m}^{-1}$ and $FE = 104.12 \pm 0.93 \text{ mN m}^{-1}$), thus improving the compatibility between the solution and the fruits' skin surface. This phenomenon may be attributed to the reorientation of polar groups in the surface layer, which increases the hydrophobicity of the solution (Wiacek, 2015). In this regard, Foschia, Peressini, Sensidoni, and Brennan (2013) explained that the hydroxyl groups of antioxidant compounds, such as gallic acid, improve the wettability of coatings. Previously, Torres et al. (2017a) demonstrated that phenolic compounds such as 1,2,3,4,6 penta-O-galloyl- β -D-glucose (PGG) and its derivatives are the

Fig. 2. Values of wettability determined for mango peel-based coatings (F) and mango peel-based coatings with antioxidants of mango seed kernel (FE) vs time. Bars represent mean $\pm$ standard error ($n = 10$). Identical letters indicate that the samples are not significantly different ($p > 0.05$).

predominant compounds in the extract of mango seed kernel. PGG is composed of five galloyl groups (carboxylic groups of gallic acids) with a glucose this structure-activity relationship especially the contributions of specific OH-groups gives its a high bioactivity (Torres et al., 2017b).

The charge of polysaccharides has a direct relationship with the zeta potential (Zp) of the solution (Carneiro, Cerqueira, Souza, Teixeira, & Vicente, 2011). Zp expresses the surface charge of particles in a dispersion and for studying their stability from an electrostatic point of view, when Zp values are further away from zero the corresponding solutions are more stable (Zambrano et al., 2013). Zp results reveal that F (-14.57 ± 0.69 mV) and FE (-13.77 ± 0.53 mV) solutions have a negative charge. This result shows that the solution formed with the mango peel has an anionic nature (Salvia, Rojas, Soliva, & Martín, 2015). Zp values of FE solution were higher than those reported in other edible coatings formulations (-13.7 to -2.1 mV) (Carneiro et al., 2011), and ensure the adequate stability of the solution. The good results obtained in this study open the possibility in future studies to optimize the parameters that influence the properties of biopackaging made with mango by-products.

3.3.2. Ethylene production and respiration rate

The coated peach allowed a lower gas exchange, and the coatings with antioxidant extract reduced significantly the production of CO_2 (in 29%) and O_2 consumption (by 39%) when compared with uncoated peaches (control) (Fig. 3). Although ethylene production is increased during storage, the fruits coated with mango peel and antioxidant extract showed a reduction greater than 64%; a delay in ethylene production is related to the reduction of senescence of fruit (Shah, Vishwasrao, Singhal, & Ananthanarayan, 2016). Reduction of ethylene production due to edible coatings application has been reported for peach (Guillén et al., 2013), fresh-cut apples coated with alginate (Guerreiro, Gago, Faleiro, Miguel, & Antunes, 2017), tomato coated with gum arabic (Ali, Maqbool, Ramachandran, & Alderson, 2010), and ciruelas recubiertas con alginate (Valero et al., 2013). This may be due to the creation of a modified atmosphere within the fruit created by the coatings (Ali et al., 2010; Elsabee & Abdou, 2013; Falguera et al., 2011).

It is worth highlighting that the rate of CO_2 production was higher than the rate of O_2 consumption, both in coated and uncoated fruit. This guaranteed that the coating layer did not affect significantly the gas balance in the fruit and that only the transfer rates were decreased (Lima et al., 2010). Therefore, reducing maturation physiologically expressed in ethylene synthesis is associated with the effect of the coatings and not to the accumulation of CO_2 in the package.

The synthesis of CO_2 and O_2 consumption in fruit presented a

behavior similar to that observed in the production of ethylene. The addition of the antioxidant extracts significantly ($p < 0.05$) influenced the reduction of the respiratory rate and the production of ethylene compared to the control and the coating without extract. This behavior has already been reported by Guillén et al. (2013) in peaches coated with *Aloe vera*, material that has been reported to have a high antioxidant activity (Kang et al., 2014).

The difference between F and FE is the incorporation of a mango seed extract rich in phenolic compounds such as 1,2,3,4,6 penta-O-galloyl- β -D-glucose (PGG) and its derivatives (Torres et al., 2017a). The beneficial effects of a coating with antioxidant extract in controlling respiratory rate can be explained by the increase of the wettability of the solution (Fig. 2). This factor ensures better coating of the skin of peaches and greater control in the transfer of gases. CO_2 and O_2 are critical factors for controlling postharvest behavior and quality of the fruit (Domínguez, Lafuente, Hernández, & Gavara, 2016), thus, the coating with antioxidant extract has a great potential to increase the shelf life of peaches. Visual evaluation of peaches at eight days of storage ($20^\circ C$ and 80% RH) also confirmed the beneficial effect of the edible coating (Fig. 4) on the prolongation of shelf life, as the coated peaches did not present damage by microbial growth or by enzymatic browning.

4. Conclusions

This work shows the feasibility of using mango (Variety Ataulfo) by-products in the formulation of edible films and coatings. Films of mango peel showed good properties of permeability, color, antioxidants and greater hydrophobicity. The addition of antioxidant extract of mango seed to the coatings formulation contributed to improve surface properties. A 39% less O_2 consumption, 64% and 29% less ethylene and CO_2 production respectively were observed in coated peach when compared with peaches without coating. This study demonstrate the great potential of mango by-products for producing coatings and packaging materials. It is important to continue research to optimize variables that influence the formulation of bio-packaging.

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Fig. 3. CO_2 and O_2 transfer rates (a) and ethylene production rate (b) of peaches coated with mango peel (grey) or mango peel and antioxidant extract of mango seed (black) or distilled water as control (white) during storage (8 days) at $20^\circ C$. Bars represent mean $\pm$ standard error ($n = 2$). Different letters denote significant difference according to one-way ANOVA and Tukey's test ($p < 0.05$).

Fig. 4. Visual aspect of peaches on the eighth day of storage (20 °C, 80% RH). (A) Uncoated peach samples (Control), (B) peel-based coated samples with antioxidants of seed kernel, (C) peel-based coated samples.

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