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Active food packaging prepared with chitosan and olive pomace

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ABSTRACT

There is currently a strong trend towards the use of biodegradable packaging to replace synthetic. Therefore the objective of this work was to evaluate the effect of the addition of different concentrations of flour and microparticles of olive pomace flour in chitosan based films. The films were evaluated for their barrier, mechanical, optical and antioxidant properties. The protective effect of films against nut oxidation was also evaluated. The incorporation of the olive residue flour into the chitosan matrix caused changes in the morphology, making the film more heterogeneous and rough. The addition of 10% of olive microparticles significantly improved the tensile strength (22.40 ± 0.22 MPa) of films without altering their original properties. The flour and the microparticles of olive increased the antioxidant capacity of the films that was proportional to the concentration of flour or microparticles added to the film. The films with 30% of flour or microparticles were effective as protective packaging against the oxidation of nuts during 31 days. The packages developed in this study are viable considering the material used in its production, its biodegradability and the added antioxidants naturally obtained from waste.

1. Introduction

The growing requirement for sustainability has increased the interest of researchers in the development of biodegradable packages from biopolymers incorporating bioactive compounds obtained from material considered as waste, thus giving added value to these products. The advantages of biopolymers in relation to polyethylenes are numerous such as rapid biodegradability, non-toxicity, biocompatibility with other biopolymers, easy interaction with food, ability to act as a vehicle for antimicrobial compounds and antioxidants (Dutta, Tripathi, Mehrotra, & Dutta, 2009; Hosseini, Rezaei, Zandi, & Ghavi, 2013). Among the natural polymers used for biodegradable packaging development, chitosan stands out due to its excellent film forming ability, high mechanical strength and good barrier capacity (Martins, Cerqueira, & Vicente, 2012). Chitosan is obtaining by the deacetylation of chitin, a compound found mainly in crustacean shells (No & Meyers, 1995).

Aiming to prolong shelf life, maintain food quality and reduce the use of synthetic food additives many studies have evaluated the incorporation of natural antioxidants into chitosan films such as green tea extract (Siripatrawan & Noipha, 2012) and propolis (Siripatrawan & Vitchayakitti, 2016). Compounds extracted from plants or obtained from food residues such as grape pomace extract (Ferreira, Nunes, Castro, Ferreira, & Coimbra, 2014) and grapefruit seed extract (Bof, Jiménez, Locaso, García, & Chiralt, 2016) were used in active films. The integral use of residues from the food industry, mainly fruits, and vegetables, is attractive due to its high content of bioactive compounds. The antioxidant potential of these residues has been proven in gelatine films added with blueberry bagasse (Crizel, Costa, Rios, & Flôres, 2016a) and minimally processed beet residues (Iahnke, Costa, Rios, & Flôres, 2016). Therefore the use of flours of blueberry and beet residues can modify the structure and consequently the mechanical properties of films.

The olive oil industry generates tons of olive pomace worldwide. This residue is rich in dietary fiber, phenolic compounds and other antioxidants such as carotenoids (Crizel, Hermes, Rios, & Flôres,

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2016b), and it is usually burned by industries (Rubio-Senent, Rodríguez-Gutiérrez, Lama-Muñoz, & Fernández-Bolaños, 2015). Furthermore, from this residue can be extracted from pectin and, according to Cardoso, Coimbra, & Lopes-da-Silva (2003), the gelling ability of this extract is similar to the citrus pectin sold commercially.

According to Crizel et al. (2016b), the bagasse from olive processing has a fraction of 92% of insoluble fiber, which could hinder the incorporation of the flour in the chitosan matrix of the films and consequently impair the physical properties of the film. For that reason, it becomes interesting the development of microparticles from this residue since the encapsulation consists in making the ingredients soluble in a solution and in addition it is a technique that helps in the stability of the antioxidant compounds (Rosa et al., 2014).

Considering the high antioxidant capacity of olive oil pomace, the aim of this study was to evaluate the effect of the incorporation of different concentrations of flour and microparticles on biodegradable chitosan films for food packaging.

2. Materials and methods

2.1. Materials

The chitosan used in the preparation of films was acquired from Golden-Shell Biochemical Co., Ltd., located in China, and had a degree of deacetylation (DD) greater than 85%.

Regarding the olive pomace used, it was composed of fruit bagasse and lumps collected after the first pressing for oil extraction, and was provided by Olivas do Sul Company (Cachoeira do Sul city- RS/Brazil).

Fresh walnut (*Juglans regia* L., cultivar Chandler) kernels were supplied by H. Reynolds de Souza, Estremoz, Portugal, packed in the commercial film (PA/PE 90). Walnuts had been harvested during the period September–October 2015 and experiments were carried out during the period October-November.

The commercial film (polyamide cast flexible–polyethylene – PA/PE 90) was purchased from AlemPack, Portugal, and presented an oxygen permeability value below 4.6×10^{-17} mol m/m^{2}sPa.

2.2. Production of olive pomace flour

Olive pomace was frozen in an ultra-freezer at −40 °C for 48 h and subsequently lyophilized (Freeze Dryer Liotop, L101, Brazil). The lyophilized material was ground in a mill (Bertel Brand, Model MCF55, Brazil). The lumps present in the waste and particles smaller than 500 nm were separated using sieves for particle size analysis (Bertel, Brazil); The flour was packed in a vacuum sealer (Sealer ECOVAC, Model ECOVAC 40, Italy) and stored in the dark at room temperature (~25 °C).

2.3. Preparation of microparticles by spray drying

For the preparation of microparticles, 5 g of olive pomace flour was mixed with water (150 mL) and stirred with a magnetic stirrer (Velp Scientifica, Model Arex, Italy) for 40 min. Tween 80 (0.1 g) was added to this mixture and, subsequently, the solution was stirred using an Ultra Turrax® homogenizer (IKA Ultra Turrax digital, Model T25 basics, Germany) for 60 s at a stirring rate of 13500 rpm. The resulting suspension was pumped at 8 mL/min with a peristaltic pump to the spray dryer (LabPlant, Model SD-05, United Kingdom), operating with an inlet drying air temperature of 160 °C.

2.4. Films preparation

The chitosan (CH) solutions with a concentration of 2% (w/w) were prepared by dissolving the chitosan flour in a 1% (w/w) aqueous acetic acid under constant stirring until complete dissolution, after which glycerol (1.0% w/w) was added.

Depending on the type of film to be prepared, olive pomace flour or microparticles were added to the filmogenic solution under magnetic stirring for 30 min. The resulting filmogenic solutions were treated in an ultrasound bath for 30 min to remove the dissolved air bubbles before being poured in polystyrene Petri dishes (0.50 g/cm^2) and dried in an oven (Binder, Model D –78532, Germany) at 40 °C for 48 h. After drying, the films were peeled off from the casting surface and maintained at 48% relative humidity and 25 °C for 48 h before their characterization.

Seven different formulations of chitosan films with olive pomace were prepared: three formulations using the flour, described in item 2.2, (concentrations of 10%, 20% 30% in relation to the mass of chitosan used in the preparation of the film); three formulations with the microparticles obtained by spray drying, (added at concentrations of 10%, 20% and 30%), and a control formulation only with chitosan.

2.5. Film thickness

The thickness of films was measured using a micrometer (Model MDC-25, MitutoyoCorp. Tokyo, Japan) with a precision of 0.001 mm and resolution: 0 mm–25 mm. The analysis was performed in triplicate, and each film sample was measured in 10 different random locations.

2.6. Scanning electron microscopy

For scanning electron microscopy, the samples were placed on mutual conductive adhesive tape on aluminum stubs and covered with a film of Au/Pd, about 30 nm thick in a sputter coater (Quorum Technologies, Model Q150T ES, United Kingdom). Olive pomace flour, microparticles, and all the film samples were analyzed by field emission scanning electron microscopy (JEOL, Model JSM7001F, Japan).

2.7. Water vapor permeability (WVP)

The films samples were placed in the superior part of a glass permeation cell (diameter of 5 cm) filled with granular anhydrous calcium chloride. The cells were sealed with silicone, weighed and placed in a desiccator that contained a saturated solution of sodium chloride, resulting in an environment of 75% relative humidity (RH) at 25 °C. The mass gain was determined by the difference between mass at time zero and 24 h. The WVP was performed in using equation (1):

$$WVP = \frac{w.L}{A.t.\Delta p} \quad (1)$$

In which w is the mass gain rate (water) (g) by the permeation cell, L is the thickness of the film (mm), A is the permeation area (m^2), t is the time of permeation (h), and Δp is the water vapor pressure difference between the two sides of the film (3.2 kPa at 25 °C).

2.8. Moisture content (MC) and water solubility (WS)

For moisture analysis, squares of each film sample (2 cm × 2 cm) were weighed and placed in a drying oven (Model D –78532, Mark