



Preparation and characterization of montmorillonite/brea gum nanocomposites films



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ABSTRACT

The aim of this study was the formulation and characterization of films, based on gum exudates from Brea tree and nanoclay particles, for food applications. Brea gum (BG) is a renewable resource available in semi-desert areas. Functional properties of brea gum based films were improved through the incorporation of montmorillonite (MMT). Studies on film forming solution of BG/MMT were conducted. Results indicated a reduction of foam forming with MMT incorporation due to the increase in surface energy. Nanoclay was incorporated to the polymer matrix and films were formed by casting. Effect of nanoclay concentration on physicochemical properties of films was studied. FTIR spectra showed a strong interaction between MMT and BG molecules and X-Ray diffractograms indicated an exfoliated MMT dispersion into film matrix. Optical properties of films were dependent on nanoclay concentration. Solubility and water and gas permeabilities decreased with increasing MMT content. Moisture sorption isotherms of BG and BG/MMT films were obtained. Results indicated that nanoclay incorporation produces a decrease in water uptake of BG. Nanoclay addition produced an increase in Young's module and tensile strength and a decrease in film elongation. Results showed that incorporation of 5% of MMT improved water resistance and water and gas barrier properties of BG based films and enhanced mechanical resistance of these films. Gas permeability measurements indicated that MMT addition reduced permselectivity (CO₂/O₂) of BG film.

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1. Introduction

For centuries man has searched effective substitutes for natural coating of food to keep them fresh isolated from physical, chemical and/or microbiological contaminants. For many years, the industry of packaging has used materials produced from various petroleum-derived monomers to elaborate varieties of plastics, which have very good functional properties but also produce serious pollution problems. Thanks to advances in science and the growing interest in the environmental impact of discarded plastics, it was developed a new series of materials of biological origin (plant, animal and microbial), whose main advantage is that they are renewable, fully

degradable and even edible in some cases. Edible films and coatings generate a modified atmosphere by creating a semi-permeable barrier against O₂, CO₂, moisture and solute movement, thus reducing respiration, water loss and oxidation reaction rates (Martínez-Romero et al., 2006). Various compounds have been used as edible films and coatings, like lipids, proteins and carbohydrates. Proteins and carbohydrates have good mechanical properties and barrier properties against O₂ and CO₂. However, the main functional properties (mechanical and barrier properties) of these hydrophilic materials depend on their water content. This is due to water vapour strongly interacts with polymer matrix affecting the structure (Bertuzzi, Castro Vidaurre, Armada & Gottifredi, 2007; Perdomo et al., 2009). In order to overpass this problem, many attempts have been done like introducing lipids as microdroplets inside the film matrix (Debeaufort, Quezada Gallo, Delporte, & Voilley, 2000; Zahedi, Ghanbarzadeh, & Sedaghat, 2010), incorporation of nanofiller of organic or inorganic origin (Bodirlau, Teaca, &

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Spiridon, 2013; Kampeerappun, Aht-ong, Pentrakoon, & Sri-kulkit, 2007) and producing laminated films that consist in a thin lipid layer on the polysaccharide film (Debeaufort et al., 2000; Phan The, Debeaufort, Luu, & Voilley, 2008).

Polysaccharide gums represent one of the most abundant raw materials. Researchers have mainly studied polysaccharide gums due to their sustainable, biodegradable and bio-safe characteristics. The term “gum” is used to describe a group of naturally occurring polysaccharides that come across widespread industrial applications due to their ability either to form gel or make viscous solution or stabilize emulsion systems. The considerably growing interest in plant gum exudates is due to their diverse structural properties and metabolic functions in food, pharmaceutical, cosmetic, textile and biomedical products (Mirhosseini & Amid, 2012).

Brea gum (BG) is the exudate obtained from the Brea tree (*Cercidium praecox*). The gum is harvested from wild trees throughout the northwest region of Argentina by native population (Chane, Wichis and Churupies). BG is a complex hydrocolloids formed by L-arabinose, D-xylose, D-glucuronic acid, and 4-O-methyl-D-glucuronic acid and approximate 8% of protein. The major structural features of BG appear to be a $\beta(1,4)$ -linked D-xylan backbone (possibly containing some 1,2-linkages) that is heavily 2-substituted by short branched-chains containing residues of D-xylose (and L-arabinose) and D-glucuronic acid, in which both types of residue may be terminal (Cerezo, Stacey, & Webber, 1969). Toxicological studies indicated that BG is not toxic for human ingest (Von Müller, López, Eynard, Aldo & Guzmán, 2009) and it is approved as food additive in Argentina. Physicochemical characteristics of BG are similar to Arabic gum (Bertuzzi, Slavutsky, & Armada, 2012), and then BG can be used as a replacement for Arabic gum in many applications. The easy access to this inexpensive, non-toxic, hydrophilic, biocompatible and biodegradable polysaccharide makes viable its potential use as film matrix for different applications. The development of applications from this natural product and their use at the production zone, for example, to increase the fruit shelf life, might be an important contribution for the local economy.

Many authors formulated edible film and coating using different gums with or without addition of substances like lipids, waxes or polysaccharides to improve their mechanical and water barrier properties (Ali, Maqbool, Ramachandran, & Alderson, 2010; Bosquez-Molina, Guerrero-Legarreta, & Vernon-Carter, 2003; Rojas-Argudo, del Río, & Pérez-Gago, 2009; Ruiz-Ramos et al., 2006). Nanocomposites are hybrid nanostructured materials. A widely studied type of nanocomposite is a class of hybrid materials composed of organic polymer matrices and nanoclay fillers. The essential nanoclay raw material is montmorillonite (MMT), a 2:1 layered smectite clay mineral with a platelet structure. MMT consists in 1 nm thick aluminosilicate layers surface-substituted with metal cations and stacked in 10 μm -sized multilayer stacks. Naturally occurring MMT is hydrophilic (Alexandre & Dubois, 2000).

The introduction of organic or inorganic fillers into a polymeric matrix increases its strength and stiffness and sometimes creates special properties, originating from the synergetic effect between the component materials (Tjong, 2006). Different authors have researched about the use of MMT to improve the characteristics of edible films (Almasi, Ghanbarzadeh, & Entezami, 2010; Chivrac, Pollet, Dole, & Avérous 2010; Tang, Alavi, & Herald, 2008). Tunç and Duman (2010) reported that water adsorption and water solubility decreased as nanoclay concentration increased within the film matrix. Water vapour and gas permeability are also reduced due to the interruption in the diffusion path created by filler particles distributed in the polymeric matrix (Tang et al., 2008). To the best of our knowledge, the use of nanocomposites with plant gums exudates has not been reported in the literature.

The scope of this contribution was to improve water resistance and barrier properties as well as mechanical properties of BG films through the incorporation of nanoclay using an appropriate methodology that ensures adequate exfoliation of the MMT layers within the film matrix.

2. Materials and methods

2.1. Materials

BG was donated from a native community group who live in the production zone of Brea tree (Tartagal, Salta, Argentine). The BG exudate from the plant is collected in the form of small drops or tears. The purification process included the steps of grinding, dissolution, decantation, filtration and drying in oven at temperatures below 50 °C, then grinding to fine powder (mesh 80-ASTM). Humidity of BG powder was $13.5 \pm 0.5\%$, ash $3.8 \pm 0.3\%$, calcium $1.1 \pm 0.1\%$, magnesium $0.3 \pm 0.1\%$ and the nitrogen content was $0.95 \pm 0.37\%$. The pH of BG aqueous solutions was 4.7. Natural sodium montmorillonite without purification and with quartz and albite present as accessory minerals, with a cation exchange capacity (CEC) of 89.8 meq/100 g clay, was supplied by Minarco S.A. (Buenos Aires, Argentine). Samples were homogenized by sieving in 200-mesh (ASTM). P₂O₅ (Mallinckrodt, USA) was used as a desiccant. Glycerol (Mallinckrodt, USA) was added as film plasticizer. All salts used to obtain different relative humidity ambient (% RH) were provided by Aldrich (USA).

2.2. Preparation of MMT solution

MMT solution was prepared stirring nanoclay and water (1.5% w/v) during 3 h at 80 °C. The suspension was centrifuged at 2500 rpm. Insoluble matter was rejected. The larger particles or aggregates of nanoclay constituted the precipitate. Smaller particles of MMT remained in colloidal solution and they were separated in the liquid phase from particles of greater size. Centrifugation was used as a selective method of separation. MMT concentration in MMT solution was determined by drying to constant weight, 20 mL aliquot in an oven at 105 °C.

2.3. Film preparation

Film-forming solution was prepared by mixing BG (10% w/v), glycerol in a concentration of 25% w/w of BG, water and the addition of an appropriate amount of MMT solution (prepared as was described in 2.2) to obtain a MMT concentrations ranged from 1 to 5% w/w of BG. The resulting dispersion was kept 60 min in an ultrasonic bath. The pH of film forming solution was not affected by the incorporation of MMT solution. Film forming solution was poured onto polystyrene plates. Then, they were placed in an air-circulating oven at 35 °C and 53% RH for 15 h. After that, plates were removed from the oven and films peeled off. Films were storage at 25 °C and 53% RH before characterization. MMT concentration in films was confirmed through ash content (AOAC, 2003) of BG and BG/MMT films. Analyses were carried out at 800 °C for 4 h and test was done in triplicate for each sample.

2.4. Surface tension and foam capacity of BG/MMT solutions

Surface tension of BG/MMT solutions was measured using Du Noüy ring tensiometer (Krüss Hamburg, Germany) with platinum ring at fixed temperature (25 °C). The method involves slowly lifting a platinum ring from the solution surface. The force required to raise the ring from the liquid surface is measured and related to

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