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Comparative study on properties of edible films based on pinhão (*Araucaria angustifolia*) starch and flour

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ABSTRACT

The aim of this study was to develop and compare the properties of edible films based on pinhão starch and pinhão flour. Seven formulations were developed by casting methodology: 5% pinhão starch with 0, 1, 1.5, and 2% glycerol, and 5% pinhão flour with 1, 1.5, and 2% glycerol. The films were evaluated and compared to each other for thickness, morphological analysis by Scanning Electronic Microscopy (SEM), glass transition temperature (T_g), apparent porosity (Ø), water vapor permeability (WVP), tensile properties, color, opacity, and Fourier transform infrared analysis (FTIR). In addition, the raw materials were analyzed for moisture content, water activity (Aw), apparent porosity and pasting profile. Pinhão starch and flour did not show differences in moisture content and pinhão starch presented lower Aw, Ø, but higher peak viscosity than pinhão flour. Films made with pinhão starch exhibited lower thickness, Ø, WVP and opacity than pinhão flour films, but higher Young's modulus, tensile strength and elongation at break. In addition, films with higher amounts of glycerol exhibited higher WVP and elongation at break, with lower tensile strength and Young's modulus. The pinhão flour films structure presented irregularities and were rougher than pinhão starch films. Differences in glycerol content did not affect T_g values of these films. Flour films were yellowish and also had a higher opacity value. The FTIR spectra of pinhão starch and pinhão flour films showed similar IR absorbance patterns, with no structural change in the presence or absence of glycerol.

1. Introduction

Plastics derived from petroleum are the most common film packaging materials in the food industry. Plastic degradation takes a long time and can cause many environmental problems by not being biodegradable (Sorrentino, Gorrasi, & Vittoria, 2007; Xu, Kim, Hanna, & Nag, 2005). The interest in developing edible films and coatings as an alternative to plastic materials has increased because of their capability to improve global food quality and extend food shelf-life. Many studies show evidence of the beneficial effects of using such films on fresh and processed foods (Chillo et al., 2008; Flores, Famá, Rojas, Goyanes, & Gerschenson, 2007). Biodegradable packaging made from entirely renewable natural polymers can decrease environmental pollution, as they degrade after their disposal, creating new markets for agricultural products (Gontard & Guilbert, 1994; Xu et al., 2005). In addition, some biodegradable films may also be edible and be consumed with the food they are wrapping.

Biodegradable and edible films can be used to limit moisture, aroma, and lipid migration between food components, to restrict absorption of oxygen, to improve mechanical handling properties, or to offer an alternative to commercial packaging materials (Bourtoom & Chinnan, 2008). Starch-based films from different sources and with different properties have been widely studied (Al-Hassan & Norziah, 2012; Chen & Lai, 2008; Chillo et al., 2008; Famá,

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Flores, Gerschenson, & Goyanes, 2006; Fakhoury et al., 2012; Garcia, Pinotti, & Zaritzky, 2006; Kechichian, Ditchfield, Veiga-Santos, & Tadini, 2010; Mali, Grossmann, García, Martino, & Zaritzky, 2004, 2005; Mali, Grossmann, García, Martino, & Zaritzky, 2006; Maran, Sivakumar, Sridhar, & Prince Immanuel, 2013; Souza et al., 2012; Vásconez, Flores, Campos, Alvarado, & Gerschenson, 2009) and have indicated that these carbohydrates are promising materials in this regard. Starches show physical properties similar to plastic materials, are readily available and have good ability to form odorless, colorless, transparent, non-toxic and biologically degradable films (Cano, Jiménez, Cháfer, Gónzalez, & Chiralt, 2014; Famá, Rojas, Goyanes, & Gerschenson, 2005; Flores et al., 2007; Soliva-Fortuny, Rojas-Graü, & Martín-Belloso, 2012). Starch-based and composite edible films can enhance food quality, safety and stability as they can control mass transfer between components within a product, as well as between product and the environment (García, Pinotti, Martino, & Zaritzky, 2009).

Edible films can also be developed from a mixture of components, as proteins, lipids and carbohydrates. Sometimes, these components present incompatibilities between one another. One approach to avoid incompatibility between the polymers from different sources is to use unrefined raw materials, such as flours. Then, the films will have the natural mixtures of carbohydrates, proteins, lipids and fibers in a totally compatible way (Maniglia, Domingos, de Paula, & Tapia-Blácido, 2014). In recent years, some studies have reported the use of flour as a raw material suitable for preparing films (Andrade-Mahecha, Tapia-Blácido, & Menegalli, 2012; Colla, do Amaral Sobral, & Menegalli, 2006; Dias, Müller, Larotonda, & Laurindo, 2010; Mariniello et al., 2003; Rayas, Hernandez, & Ng, 1997; Salas-Valero, Tapia-Blácido, & Menegalli, 2015; Tapia-Blácido, Sobral, & Menegalli, 2005). The use of natural blends of proteins, polysaccharides, and lipids directly obtained from agricultural sources takes advantage of each component in the original system and appears to be a new opportunity for composite materials in the area of edible films (Tapia-Blácido et al., 2005).

Since the earliest times, cultivation of plants containing high quantities of starch has been of great importance to humanity. Cereal grains, legume seeds, tubers, and certain fruits contain from 30% to 85% starch on a dry basis. In the native state, starch exists as insoluble granules with characteristic shape and some crystallinity. Starches contain between 18% and 30% amylose, except for high amylose starches and waxy corn types that are practically all amylopectin. Each raw starch has its own characteristic viscosity/temperature profile, as a result of its particular granular composition and structure (Kramer, 2009) and these difference influence the final characteristics of starch-based films.

Pinhão is a regional seed of South America widely consumed in the local cuisine. The seeds have starch as the main component, at approximately 36% on wet basis (w.b.) (nearly 72% on dry basis) (Cordenunsi et al., 2004). Raw pinhão flour has a greater amount of starch, at about 64%, and it contains between 3 and 5% proteins, 2 and 6% lipids, around 6% dietary fiber and 2.5% ash (Capella, Penteado, & Balbi, 2009; Cladera-Olivera, Marczak, Noreña, & Pettermann, 2011). These characteristics show that pinhão flour and starch are good sources of complex carbohydrates and potential film forming ingredients for edible films. In addition, this regional seed in new sustainable products can support Brazil's conservation efforts that encourage consumption of this crop. New uses for pinhão can encourage agricultural extension for small farmers and extend the use of pinhão year round, rather than only during the harvesting season, increasing its market (Figueiredo-Filho, Orellana, Nascimento, Dias, & Inoue, 2011; Santos, Corso, Martins, & Bittencourt, 2002).

Based on the constant necessity of food industry for new and innovative products, pinhão (*Araucaria angustifolia*) seeds were proposed for the first time in this study as a natural source of starch and flour to develop edible films containing only pinhão starch or flour and glycerol. There are few studies applying pinhão starch as base for biofilms (Luchese, Frick, Patzer, Spada, & Tessaro, 2015; Spada, da Silva, & Tessaro, 2014), but, these studies also incorporated xanthan gum into the films. To the best of our knowledge, the development of pinhão flour-based films was not tested yet, nor was the use of pinhão starch in films not including xanthan gum. The use of pinhão flour has the advantage of using the whole seed, decrease the process steps and waste. The aim of this study was to develop edible films based on pinhão starch and pinhão flour plasticized with glycerol, evaluate film physical properties, and compare starch-based and flour-based films with different glycerol contents.

2. Methodology

2.1. Materials

Pinhão seeds harvested in Southeastern Brazil in 2014 were used to develop edible films. One kilogram of pinhão seeds was used to obtain two fractions: starch and flour. The starch was obtained according to Daudt, Külkamp-Guerreiro, Cladera-Olivera, Thys, and Marczak (2014) and the flour was obtained from raw seeds. For the flour, the seeds were peeled, and dried in an oven with air circulation (SL 102/100, Solab, Brazil), at 50 °C for 24 h. After drying, the seeds were pre-milled in a cutting-grinding head (IKA® MF 10.1 basic, Staufen, Germany) to produce a coarse flour with around 2 mm particle size and finely milled to pass through a 0.25 mm mesh with a Cyclone sample mill (Udy Corp., Fort Collins, CO, USA). Vegetable 100% pure glycerol was used as plasticizer (Starwest Botanicals, Sacramento, CA, USA) in film preparation.

2.2. Methods

2.2.1. Physical properties of pinhão starch and flour

Physical properties of pinhão starch and flour were evaluated to understand the mechanisms involved during film formation and the final product properties.

Moisture content of pinhão starch and flour samples was determined according to AOAC method 934.06 (AOAC, 1997).

Water activity of pinhão starch and flour samples was measured using an AquaLab 4TE Water Activity Meter (Decagon, Pullman, WA, USA). It was previously calibrated using saturated salt solutions, according to the calibration procedure for this instrument. Water activity was measured at room temperature (25 ± 2 °C).

To determine true density of powders and films, a gas pycnometer (Micrometrics AccuPyc II 1340, Norcross, GA, USA) was employed to measure the displaced volume from samples. The corresponding mass of the dried sample was previously measured. The true density was calculated by dividing mass by volume of samples in five replicates.

Bulk density was measured according to the Method 616 from the United States Pharmacopeia for powders (USP, 2008) with some modifications. Five replicates made using 1.6×10^{-4} m^3 cylindrical container filled using a funnel, with its discharge opening located 25 mm above the top edge of the container. Since the powder in the funnel bridged, it was stirred with a thin glass bar to provide a continuous flow through the funnel discharge. The powder in the container was leveled by rolling a round, stainless steel bar across the container in two perpendicular directions. Then, the container was weighed. The bulk density was calculated by dividing the mass of the sample by the volume of the container. The apparent porosity ($\varnothing$) was calculated from the true density (ρ_t) and bulk density (ρ_b) according to Eq. (1) (Datta, 2007).