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Optimization of process conditions for the production of films based on the flour from plantain bananas (*Musa paradisiaca*)

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ABSTRACT

In this work, the casting process has been employed for the production of flour films from plantain bananas (*Musa paradisiaca*); glycerol has been used as plasticizer. The influence of process conditions such as the glycerol concentration (Cg), the process temperature (Tp), the drying temperature (Td), and the relative humidity (RH) on the mechanical, barrier, and optical properties of banana flour films has been evaluated by means of a central composite design. The results have been statistically analyzed by the response surface methodology and desirability function, and the optimal process conditions for film formation have been determined. The process variables have a significant impact on the mechanical properties, water vapor permeability (WVP), and opacity of the films, but these features are mostly affected by the Cg parameter. Compared to other biodegradable films, the banana flour film displays high opacity, low solubility in water, good WVP and flexibility, and excellent mechanical strength and rigidity. The desirability function employed here has allowed for the establishment of the optimal process conditions, which are Cg = 19 g of glycerol/100 g of flour, Tp = 81 °C, Td = 54 °C, and RH = 48%, proving to be an effective tool for this type of study.

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1. Introduction

Polysaccharides and proteins of animal and vegetable origin are natural biodegradable polymers that have traditionally been used to produce environmentally-friendly films (Chandra & Rustgi, 1998; Krochta & De Mulder-Johnston, 1997). Starch is the most widely employed polysaccharide for film production, because it is naturally abundant and inexpensive (Alves, Mali, Beléia, & Grossmann, 2007; Mali, Grossmann, García, Martino, & Zaritzky, 2004). Starch films present good mechanical and oxygen barrier properties, but their sensitivity to moisture is a major drawback. To improve the characteristics of these materials, some authors have designed films based on starch and protein mixtures (Coughlan, Shaw, Kerry, & Kerry, 2004; Jagannath, Nanjappa, Das Gupta, & Bawa, 2003). Researchers have also added lipids to the formulation of some films, to enhance the water vapor barrier (Bravin, Peressini, & Sensidoni, 2004; García, Martino, & Zaritzky, 2000).

When developing films, another alternative is to use flours, which are naturally occurring complex blends of starch, protein, lipids, and fibers. Some authors have reported on the potential application of flours obtained from whole materials such as amaranth, soy, and wheat for film production (Mariniello et al., 2003; Rayas, Hernandez, & Ng, 1997; Tapia-Blácido, Sobral, & Menegalli, 2005a). The excellent characteristics of these films stem from the natural and intrinsic molecular interactions taking place between their starch, protein, lipid, and fiber components.

An interesting renewable raw material for the preparation of edible and biodegradable films is the unripe banana fruit. Originating in Southeast Asia, bananas (genus *Musa*) are an important food crop that is extensively grown in tropical and subtropical regions. Unfortunately, post-production losses are huge due to the highly perishable nature and inadequate post-harvest handling of banana fruit. Processing both the surplus fruit and the fruit that is inappropriate for fresh consumption reduces these losses. Starch is the major constituent of unripe bananas and comprises over 70 g/100 g of their dry weight. Moreover, this fruit contains a significant amount of protein (1.0–2.5 g/100 g), lipids (0.2–0.5 g/100 g) and fiber (1.5–2.5 g/100 g) (Zhang, Whistler, BeMiller, & Hamaker, 2005), which could be interesting for the production of biodegradable films. In this sense, the flour of unripe bananas may be an attractive alternative for the attainment of a continuous matrix.

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Romero-Bastida et al. (2005) recently used starches isolated from banana, okenia, and mango to produce edible films. Okenia and banana films were the most (32%) and the least (23%) soluble in water, respectively. The banana film had the highest tensile strength value (25 MPa) compared with mango (18 MPa) and okenia (17 MPa) films. A group of researchers studied the effect of banana starch modified by oxidation and acetylation on the properties of films (Zamudio-Flores, Bautista-Baños, Salgado-Delgado, & Bello-Pérez, 2009; Zamudio-Flores, Vargas-Torres, Pérez-González, Bosquez-Molina, & Bello-Pérez, 2006). The oxidation level increased but acetylation decreased the solubility and WVP of the film. Oxidation enhanced the tensile strength of the film, and acetylation of the oxidized starch improved this property. Elongation at break diminished when the oxidation level rose.

More recently, Pitak and Rakshit (2011) employed banana flour/chitosan composite film bags to preserve freshly cut vegetables. The composite yellowish film exhibited great water permeability of $4.5–4.8 \times 10^{-10}$ g/m s Pa. Tensile strength and elongation were in the range of 5.2–14.2 MPa and 1.6–2.6%, respectively, while solubility ranged between 40.9 and 64.2%. The presence of starch in the composite film furnished water soluble and sealable bags or wraps, while the presence of chitosan provided them with the antimicrobial property. In another work, Sothornvit and Pitak (2007) prepared films from the banana flour developed; however, they evaluated the oxygen permeability and the mechanical properties of these films, but they did not determine solubility or the water vapor barrier and the optical properties.

Among the different varieties of banana, the cultivar "Terra" (*Musa paradisiaca*), a type of plantain, exhibits excellent features for the preparation of biodegradable films (Pelissari, Andrade-Mahecha, Sobral, & Menegalli, 2012). Because a few studies reported in the literature have dealt with this cultivar, we have selected it for the present investigation.

Considering that the several parameters employed during film production influence the film properties, a study was carried out to verify the effect of glycerol concentration (Cg), process temperature (Tp), drying temperature (Td), and relative humidity (RH) on film features, so that banana flour films with the desired characteristics can be obtained. In this context, the present study aimed to determine the optimal formulation of banana flour film by using response surface methodology and multi-response analysis, in order to obtain films with low water vapor permeability, moderate elongation, and high resistance to break.

2. Materials and methods

2.1. Materials

The flour was prepared from unripe plantain bananas of the variety "Terra" (*M. paradisiaca*), according to the methodology described by Pelissari et al. (2012). The fruit was obtained from the harvest occurred in March 2010 in the state of Espírito Santo, Brazil, but it was not subjected to any postharvest treatment. All the chemicals used in this work were reagent grade.

2.2. Physicochemical analysis of the banana flour

The size of flour particles was determined in triplicate with a laser diffraction analyzer (Laser Scattering Spectrometer Mastersizer S, model MAM 5005 – Malvern Instruments Ltd., Surrey, England) using ethanol as solvent. The moisture, ash, protein, and crude fiber contents were analyzed by the AOAC standard methods (2005). Lipids were assayed by the method of Bligh and Dyer (1959), as described by Cecchi (1999). The amylose content was obtained according to the methodology reported in ISO 6647 (1987), and total starch was identified by using the method proposed by Diemair (1963). All the experiments were performed at least in triplicate, and the results are presented as mean values.

2.3. Film production

Films were produced by the casting method. This process consists in drying a film-forming suspension (FFS) that has been applied onto a support. The procedure developed herein involved the homogenization of a water solution of 4 g/100 g (d.b.) of banana flour by mechanical stirring for 30 min, followed by heating to the process temperature (Tp: 75–95 °C) under gentle stirring. Glycerol (Cg: 15–30 g of glycerol/100 g of flour) was added at this point, and the solution was maintained at this temperature for 15 min. Next, the FFS was sonicated for 10 min, and 70 g of the solution was poured onto acrylic plates (18 × 21 cm), so as to obtain a constant thickness. The films were dried in a chamber with air circulation under controlled temperature (Td: 35 to 55 ± 0.5 °C) and relative humidity (RH: 30 to 70 ± 0.5%).

Before the characterization of the films in terms of moisture content, mechanical properties, and water vapor permeability, they were conditioned in desiccators under 58% RH, at 25 °C, for 48 h.

2.4. Film characterization

2.4.1. Thickness and density

The thickness of the films was measured using a manual micrometer (Fowler, model FOW52-229-001, Pennsylvania, USA) with an accuracy of 0.0001 mm. The mean thickness of each film was determined from an average of 10 random measurements.

To determine the density, samples of each film were cut into 20 × 20 mm squares, and the thickness of these films was measured (3 random measurements). The film samples were dried at 105 °C for 24 h and weighed, and the density was calculated as the ratio between the weight and volume (thickness × area) of the film. The density experiments were accomplished in triplicate, and the data are reported as mean values.

2.4.2. Moisture content

The moisture content of the films was analyzed gravimetrically, in triplicate, according to the standard method D644-99 (ASTM, 1999), by drying the samples at 105 °C for 24 h.

2.4.3. Mechanical properties

The tensile properties were investigated with the aid of a texture analyzer (Stable Micro Systems, model TA.TXplus, Surrey, England) according to the standard method D882-02 (ASTM, 2002), by taking an average of six determinations in each case. The samples were cut into 25-mm wide and 115-mm long strips by means of a scalpel and mounted between the tensile grips. The initial grip separation and the crosshead speed were set at 80 mm and 1.0 mm/s, respectively. The tensile strength (force/initial cross-sectional area) and the elongation at break were computed directly from the strength × elongation curves using the Texture Exponent 32 software, and Young's modulus was calculated as the slope of the initial linear portion of this curve.

2.4.4. Solubility in water

The solubility (S) values were determined by employing the methodology described by Gontard, Guilbert, and Cuq (1992). To this end, three discs (diameter = 20 mm) of each film were stored in a desiccator containing silica gel (~0% RH) for 48 h. The samples were weighed, to obtain the initial dry weight (W_i), and they were then immersed into 50 mL water containing sodium azide (0.2 g/L) at 25 °C for 24 h, under sporadic agitation. After this period, the

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