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Turmeric dye extraction residue for use in bioactive film production: Optimization of turmeric film plasticized with glycerol

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ABSTRACT

Four turmeric flour fractions (F1, F2, F3, and F4) were produced from the turmeric dye extraction residue by wet milling and sieving for film production. F2, which presented the lowest lignocellulosic and the highest starch content, provided the best material for film production. The main objectives of the present study were (i) to evaluate the effect of heating temperature (T) and pH on the mechanical and functional properties of F2 film plasticized with glycerol, and (ii) to optimize the process conditions (T, pH) by response surface method and multi-response analyses. Higher T (>90 °C) and alkaline pH produced a denser and mechanically stronger polymer matrix and more soluble film. In contrast, less soluble films were produced at intermediate T and pH. The effect of T and pH on the mechanical properties and solubility of turmeric films is associated with starch gelatinization and protein solubilization and denaturation. High T produced loss of the curcuminoids and antioxidant activity of turmeric films. The optimal conditions were T of 85.1 °C and pH of 8.1. These conditions yielded films with high mechanical strength (9 MPa), low solubility (37%), and low water vapor permeability (0.352 g mm h^{-1} m^{-2} kPa^{-1}).

1. Introduction

Edible and/or biodegradable films from biopolymers originating from agricultural sources could substitute non-degradable synthetic plastics in packaging materials. Researchers are currently exploring different materials such as flour extracted from agricultural sources in an attempt to improve the properties of biodegradable films. Because this flour consists of natural blends of starch, protein, and lipid that remain in their original system, it is a new candidate material in the area of biodegradable and edible films (Baldwin, Nisperos-Carriedo, & Baker, 1995; Tapia-Blácido, Mauri, Menegalli, Sobral, & Añón, 2007). Researchers have obtained flour from raw materials of plant origin such as fruits, cereals, tubers, and rhizomes to prepare biodegradable films (Andrade-Mahecha, Tapia-Blácido, & Menegalli, 2012; Dias, Muller, Larotonda, & Laurindo, 2010; Gontard, Guilbert, & Cuq, 1993; Pelissari, Andrade-Mahecha, & Sobral, 2013; Tapia-Blácido et al., 2007; Tapia-Blácido, Sobral, & Menegalli, 2011). A recent trend has been to employ industrial residues to produce flour from natural mixtures of biopolymers, which may provide the packaging market with a competitive product.

Curcuma longa L. belongs to the family Zingiberaceae, commonly known as turmeric. This herb is bright yellow because it contains the curcuminoids curcumin, demethoxycurcumin, and bisdemethoxycurcumin. Curcumin is a diphenolic compound that exerts anti-inflammatory action; it can potentially treat cystic fibrosis, Alzheimer's, malarial diseases, and cancer (Joshi, Jain, & Sharma, 2009; Yallapu, Jaggi, & Chauhan, 2012). The turmeric resin oil usually contains 30–45% of curcuminoids and 15–20% of volatile oil. Extraction of this resin oil generates a residue that consists mainly of starch and fibers; this residue may also present residual levels of curcuminoids with antioxidant properties (Braga, Leal, Carvalho, & Meireles, 2003; Braga, Moreschi, & Meireles, 2006; Kuttigounder, Rao, & Bhattacharya, 2011). In a previous study, our research group demonstrated that the film produced from the turmeric dye extraction residue and containing sorbitol as plasticizer exhibited antioxidant activity and could constitute an active packaging material. However, these films had poor elongation at break (Maniglia, Domingos, de Paula, & Tapia-Blácido, 2014).

In this scenario, the present work reports on the development of a bioactive turmeric flour film plasticized with glycerol. The heating temperature and the pH used during turmeric flour film production

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were optimized. Glycerol was selected because it is a highly hygroscopic molecule, glycerol addition to film-forming solutions prevents film brittleness (Karbowiak et al., 2006), and glycerol is almost always systematically incorporated in most hydrocolloid films (Cuq, Gontard, Cuq, & Guilbert, 1997; Zhang & Han, 2006).

2. Material and methods

2.1. Materials

The dried, milled, and sieved (0.250 mm) turmeric (*Curcuma longa* L) was supplied by the industry "Flores and Ervas" (Campinas, Brazil). The turmeric residue (R) was obtained from turmeric dye extraction by the Soxhlet method at ~47 °C, for 3 h. A mixture of ethanol/isopropanol (1:1 v/v) (Synth-São Paulo, Brazil) was used as solvent during Soxhlet extraction, because this is the mixture employed by the dye industry in Brazil.

Glycerol, used as plasticizer, was purchased from Sigma–Aldrich (São Paulo, Brazil).

2.2. Preparation of turmeric flour from the turmeric dye extraction residue

Wet milling of R yielded the turmeric flour (Fig. 1). First, R was steeped in water at 5 °C, for 24 h. Next, R was milled and passed through 80-mesh screens four times. Then, the material was sieved through 200- and 270-mesh screens. The solids retained in the 80-(F1), 200-(F2), and 270-(F3) mesh screens were dried at 35 °C, for 24 h, in an oven with forced circulation (MA Q314M, Quimis, Brazil). The liquid fraction was centrifuged at 6000 × *g* and 10 °C, for 20 min, to separate the solids, which were dried at 35 °C, for 24 h. This material was called F4.

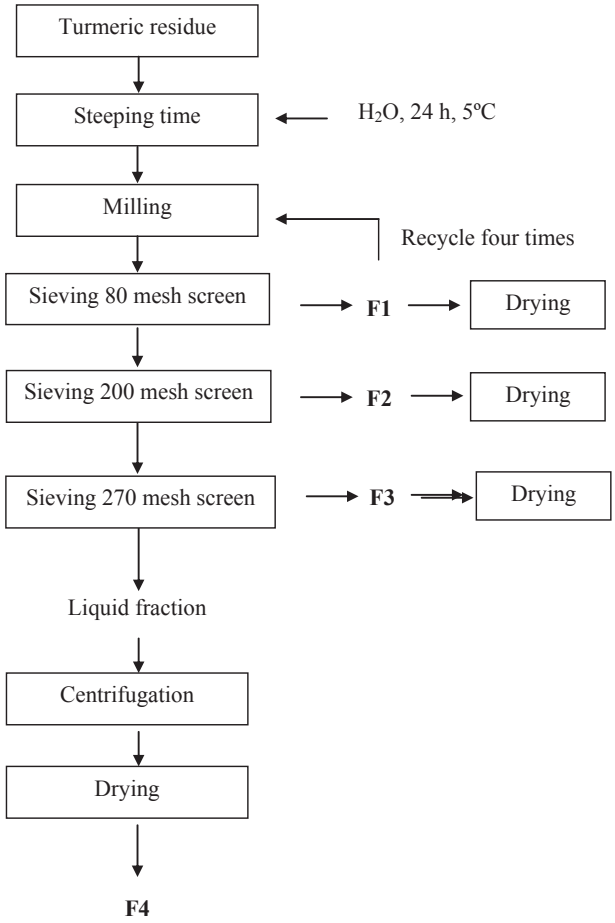

Fig. 1. Procedure employed to produce turmeric flour fractions.

2.3. Turmeric flour characterization

The turmeric flour moisture, crude protein, and ash contents were analyzed according to standard AOAC methods (AOAC, 1997). The lipids content was calculated by the method of Bligh and Dyer (1959). Cellulose was determined on the basis of the methodology described by Sun (2004). Hemicellulose was determined by HPLC, according to the methodology described by Gouveia, Do Nascimento, & Souto-Maior (2009). Klason lignin was determined by the TAPPI T 222 om-22 (2002) method, and soluble lignin was analyzed by measuring the absorbance at 280 nm. All the analyses were performed in triplicate.

2.4. Film preparation

The turmeric flour films were prepared by casting of the F2 turmeric flour fraction. Initially, a suspension of 5 g of flour/100 g was prepared in deionized water and homogenized for 30 min in a magnetic stirrer (IKA MAG ® C-HS7-Marconi, Brazil). The pH was adjusted, and the solution was heated for 4 h. During this period, the solution was submitted to 2-min homogenization cycles at 12,000 rpm at every hour; an ultra-turrax homogenizer (Ultra-cleaner 1400, Unique, Brazil) was employed. The pH (6.59, 7.0, 8.0, 9.0, or 9.41) and T (78, 80, 85, 90, or 92 °C) were varied according to a 2^2 central composite design (star configuration). Next, 22 g of glycerol/100 g of flour was added, and the mixture was heated for 20 min. The solution was poured onto acrylic plates (a weight of 0.15 g m^{-2} was maintained), and dried at 35 °C, for 7 h, in an oven with forced circulation (MA Q314M, Quimis, Brazil). Before characterization, all the films were preconditioned for at least 48 h in desiccators containing saturated NaBr solution (58% RH).

2.5. Mechanical properties

The mechanical tests were performed on a texture analyzer TA TX Plus (TA Instrument, England). The tensile strength (TS) and elongation at break (E) were obtained according to the ASTM D882-95 method (ASTM, 1995), by taking an average of five determinations in each case. The crosshead speed was 1.0 mm s^{-1}, respectively. Young's modulus (YM) was calculated as the inclination of the initial linear portion of the stress versus strain curve, with the aid of the software Texture Expert V.1.22 (SMS).

2.6. Solubility in water (S) and moisture content (MC)

Solubility was calculated as the percentage of dry matter of the solubilized film after immersion in water at 25 ± 2 °C for 24 h (Gontard, Guilbert, & Cuq, 1992) as described by Tapia-Blácido et al. (2011). The moisture content in the films was also determined by drying the materials in an oven at 105 °C until constant weight (~24 h). The analyses were performed in triplicate.

2.7. Water vapor permeability (WVP)

The water vapor permeability (WVP) test was performed at 25 ± 2 °C, in triplicate; a modified ASTM E96-95 (ASTM, 1995) method was used. Film samples were sealed over the circular opening of a permeation cell containing silica gel. Then, the cells were placed in desiccators containing distilled water. After the