



Gelatin-based films additivated with curcuma ethanol extract: Antioxidant activity and physical properties of films



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ABSTRACT

Curcuma longa L. rhizomes contain curcuminoid pigments (more specifically, curcumin), which are phenolic compounds. These compounds can be incorporated into films for their functional properties (such as their antioxidant capacity), allowing this film to be used as food packaging. Thus, the aim of this study was to use curcuma ethanol extract to prepare gelatin-based films and evaluate the effects of the extract incorporation on the antioxidant and physical properties of the films. The gelatin-based films were produced by casting technique, and the curcuma ethanol extract (CEE) was incorporated at concentrations of 0, 5, 50, 100, 150, and 200 g/100 g of gelatin. The color parameters, light transmission, gloss measurements, microstructure, infrared spectroscopy characteristics, mechanical properties, moisture content, water-soluble matter, water vapor permeability and antioxidant capacity of the films were evaluated. Adding CEE to the gelatin-based films increased their ultraviolet and visible light barrier. Infrared spectroscopy analysis suggested there were interactions between the phenolic compounds of the extract and the gelatin, which may have improved the mechanical properties (tensile strength and elongation at break) and reduced both the water-soluble matter and water vapor permeability of the films. The antioxidant capacity of the films increased with increasing concentrations of CEE. The incorporation of curcuma ethanol extract conferred barrier properties and antioxidant capacity to the gelatin-based films.

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1. Introduction

Films produced with biopolymers from renewable sources have the ability to carry active compounds (Muñoz-Bonilla & Fernández-García, 2012); thus, they can be used as active packaging for food. Active packages may contain substances that interact with the packaged product (Pereira-de-Abreu, Cruz, & Paseiro-Losada, 2012; Suppakul, Miltz, Sonneveld, & Bigger, 2003) such as antioxidants. In active packages, the active compounds may be incorporated into the films rather than directly adding the compounds to foods, providing functional effects at the food surface (Coma, 2008), which is where the oxidation process is mostly found.

Lipid oxidation in food products may be a serious problem for the food industry. Studies by Bondet, Brand-Williams, and Berset (1997) showed that reactive species (alkyl radicals and peroxide) that produced hydroperoxides were formed, and these compounds are responsible for changing the nutritional and organoleptic characteristics of food. The use of antioxidants may prevent or

inhibit the damaging actions of these radicals (Park, Lim, & Hwang, 2012).

Several studies showed that the incorporation of natural extracts into films based on renewable sources can improve the properties of gelatin-based films (Bodini, Sobral, Fávoro-Trindade, & Carvalho, 2013; Giménez, López-de-Lacey, Pérez-Santín, López-Caballero, & Montero, 2013; Hoque, Benjakul, & Prodpran, 2011; Wu et al., 2013), in addition to conferring functional properties to the film.

Wu et al. (2013) found that films based on silver carp (*Hypophthalmichthys molitrix*) skin gelatin showed antioxidant capacity when incorporated with green tea extract. Moreover, the additivated films presented better mechanical properties and a higher water vapor barrier in relation to the control film (without extract). Bodini et al. (2013) found that gelatin-based films containing propolis ethanol extract reduced water vapor permeability in comparison with the film without added extract, in addition to maintaining the antimicrobial activity and concentration of polyphenols by 177 days.

Curcuma (*Curcuma longa* L.) contains phenolic compounds (curcuminoid pigments) in its rhizomes that are responsible for the functional properties of the plant (Katz, Trask, & Lucchesi, 2009;

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Kowsalya & Krishnaveni, 2011), such as antioxidant (Jayaprakasha, Jaganmohan-Rao, & Sakariah, 2006), antimicrobial (Arutselvi, Balasaravanan, Ponnuragan, Muthu-Saranji, & Suresh, 2012), anti-inflammatory (Menon & Sudheer, 2007), and anticancer activities (Jiang et al., 2012).

The curcuminoid pigments comprise curcumin, demethoxycurcumin, and bisdemethoxycurcumin. Curcumin is the component that is found in the greatest concentrations in the *Curcuma longa* L. rhizome (Govindarajan, 1980). According to Chatterjee, Padwal-Desai, and Thomas (1999) and Ak and Gülçin (2008), the curcuminoid pigments may exhibit higher or equivalent antioxidant capacity than some synthetic compounds (BHT, BHA and α -tocopherol). Therefore, there is interest in producing active films with added curcuma ethanol extract as active packaging for food.

Thus, the aim of this study was to use the curcuma ethanol extract to prepare gelatin-based films and to evaluate the effects of extract incorporation on the antioxidant and physical properties of the films.

2. Materials and methods

2.1. Materials

Curcuma rhizome powder (Oficina de Ervas, Ribeirão Preto, Brazil), ethanol (Synth), and pig skin gelatin type A (Bloom 260, 40 Mesh) were acquired from GELITA of Brazil Ltda. (São Paulo, Brazil) and used together with sorbitol plasticizer (Cromoline, Diadema, Brazil) for film production. Standard curcumin (Merck, Darmstadt, Germany), Folin–Ciocalteu reagent (Sigma–Aldrich, USA), sodium carbonate (Synth), and standard gallic acid (Sigma–Aldrich, USA) were used to determine the curcumin content and total phenolic compound concentration of the extract. DPPH• radical (2,2-diphenyl-picrylhydrazyl, Aldrich, USA), ABTS•+ radical (2,2-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid), Aldrich, USA), and potassium persulfate (Synth) were used to characterize the film (antioxidant capacity).

2.2. Production and characterization of curcuma ethanol extract

Curcuma rhizome powder (15 g) of a standardized particle size (48 mesh) was added to 150 mL of ethanol (70%) under mechanical stirring (Fisaton-713, São Paulo, Brazil) at 100 rpm and 40 °C for 1 h in the absence of light by using a thermostatic bath (Marconi MA-415, Piracicaba, Brazil). After this period, the solution was stored under refrigeration for 24 h, and curcuma ethanol extract (CEE) was obtained after filtering the mixture. The extract was stored for 7 days under refrigeration in the dark.

2.2.1. Curcumin content of curcuma ethanol extract

The curcumin content of the CEE was determined by the spectrophotometric method of Kumar, Kishan, Roa, Duganath, and Kumanam (2010). A standard curve was constructed by using standard curcumin (98% purity) at 1.6, 2.4, 3.2, 4.0, 4.8 and 5.6 $\mu\text{g}/\text{mL}$ (diluted in ethanol), and readings were performed at 427 nm in a spectrophotometer (Biospectro, SP-22, São Paulo, Brazil). For the readings, the extract was diluted in ethanol and the results were expressed as mg curcumin/mL of extract. The experiment was performed in triplicate.

2.2.2. Total phenolic compound contents of curcuma ethanol extract

The total phenolic compound contents of CEE were determined by Folin–Ciocalteu method (Singleton, Orthofer, & Lamula-Raventós, 1999). An aliquot of the extract diluted in ethanol (70%) was added to 2.5 mL of Folin reagent (1:10 diluted in distilled

water), and after 5 min, 2.0 mL of sodium carbonate was added (7.5%, diluted in distilled water). After 2 h in the absence of light, the absorbance of the solution was determined at 740 nm. The result was expressed as mg of gallic acid/mL of extract by using a standard gallic acid curve.

2.3. Production and characterization of films

The films were produced by a casting technique. The concentrations of gelatin (2 g/100 g filmogenic solution) and sorbitol (30 g/100 g gelatin) were kept constant. First, the gelatin was hydrated (room temperature, 30 min) and solubilized in distilled water by using a thermostatic bath (55 °C, 10 min). Previously dissolved sorbitol in distilled water was then added, and the solution was magnetically stirred (2 min). Then, the filmogenic solution was kept in a thermostatic bath (55 °C, 10 min). CEE was added to the solution at concentrations of 0, 5, 50, 100, 150 and 200 g/100 g of gelatin. The solution was magnetically stirred (2 min) and kept in an ultrasonic bath (Unique, Ultra Cleaner 1400 A, São Paulo, Brazil) for 2 min. The filmogenic solution was distributed into acrylic plates (12 × 12 cm) and subsequently dried for 24 h at 30 °C in an air circulation drying oven (Marconi MA-037, São Paulo, Brazil). The film thickness was controlled as a function of the mass of the filmogenic solution/plate area and determined by using a digital micrometer (Mitutoyo, Japan) through the arithmetic average of 10 random measurements of the film surface.

Prior to characterization, the films were stored in desiccators containing saturated NaBr solution (at a relative humidity of 58%) at 25 ± 2 °C for 5 days. With the exception of the infrared spectroscopy and microstructure analyses, the films were stored in desiccators containing silica gel at 25 ± 2 °C for a period of 10 days. Analyses were performed in a climate-controlled room at 25 ± 2 °C under controlled relative humidity (50–60%).

2.3.1. Visual aspect

Visual analyses of the films were performed by evaluating their homogeneity (for uniform color and the presence of insoluble particles).

2.3.2. Color parameters

The color parameters L^* (lightness), a^* (chroma a^*), and b^* (chroma b^*) were determined for the films according to Gennadios, Weller, Hanna, and Froning (1996) by using a colorimeter (HunterLab, Miniscan XE plus, Reston, USA) controlled by a Universal Software program. The results were obtained in triplicate from 10 random measurements of the film surface. The films were placed on a white standard plate with color values of $L^* = 93.9$, $a^* = -0.8$, and $b^* = 1.2$.

2.3.3. Light transmission

The ultraviolet and visible light barrier properties were determined according to Fang, Tung, Britt, Yada, and Dalgleish (2002) by using a Biochrom spectrophotometer (Libra S22, Cambridge, England). The analyses were performed in triplicate. Film samples (10.0 cm in length and 1.5 cm in width) were fixed in place of the cuvette so the light beam could pass over the film surfaces. Transmittance measurements were taken at wavelengths between 200 and 800 nm.

2.3.4. Gloss measurements

The gloss of the films was determined according to the methodology described by Villalobos, Chanona, Hernández, Gutiérrez, and Chiralt (2005) by using a gloss meter (Rhopoint, Novo-Gloss 20/60°) at 20° and 60° angles. The gloss values were obtained in triplicate from 10 random measurements of the film surface.

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