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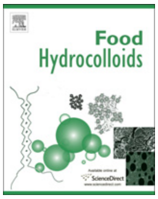

Defatted mustard seed meal-based biopolymer film development

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ABSTRACT

Edible films were developed from a defatted mustard seed meal (*Sinapis alba*) (DMM), a byproduct from the bio-fuel industry. Films were formed by casting DMM suspensions (3-g DMM/100-g suspension) that were treated by high-pressure homogenization (HPH, 138 MPa), ultrasound (400 W, 30 min), or gamma irradiation (10 or 20 kGy), and mixed with glycerol and soy lecithin. Rheological properties, water vapor permeability, water solubility, tensile strength (TS), percentage elongation (%E), elastic modulus (EM), color, and structural properties of film-forming suspensions or films were determined. Films were successfully produced using the HPH-processed suspension with 0.6% glycerol. Rheology results indicated the polymer network structure of the film-forming suspension was loosened by HPH, but tightened by heating at 90 °C. The ranges for the properties of WVP, WS, TS, %E, and EM of the films were 3.4–5.0 g mm/kPa h m^2, 30.3–34.4%, 1.3–5.5 MPa, 0.9–18.1%, 33.2–294.7 MPa, respectively. L, a, and b by CIELAB coordinates were 73.3–77.9, 0.4–3.5, and 29.5–45.7, respectively. HPH increased TS and %E of the films and decreased EM, whereas the ultrasound and the 20 kGy-irradiation treatments increased %E and decreased EM. The TS and EM decreased and E% increased with increasing glycerol and soy lecithin. DMM is a promising material to produce edible biopolymer films and coatings for food packaging.

1. Introduction

Edible biopolymer films may be formed as either food coatings or stand-alone film wraps, layers, casings, and pouches. The purpose of using edible films includes inhibiting the migration of oxygen, carbon dioxide, aromas, oils, and moisture (Kester & Fennema, 1986). Edible plant-based films are preferred by consumers who wish to avoid animal-based proteins (e.g., collagen) and obtain sustainability from food packaging.

Although edible biopolymer biodegradable/edible films and coatings from various sources have shown potential for commercial application, utilization of the films and coatings in the food industry is not popular. One major factor limiting practical application is the high cost of biopolymer materials. Low-value agricultural byproducts may represent a viable source of biopolymer feedstocks that can be used to reduce the cost of manufacturing edible films and coatings.

Mustards are functional foods having beneficial physiological effects in humans (Cui, 1997). *Sinapis alba*, commonly known as yellow mustard, can be used as a source for a wide range of active components including isothiocyanates, phenolics, dithiolthiones, and dietary fiber (Cui, 1997). Defatted mustard seed meal (DMM) is a byproduct left after crushing mustard seeds for oil removal.

Developing biopolymer films possessing favorable mechanical and moisture barrier properties is important for film application to food systems (McHugh & Krochta, 1994). Heterogeneous biopolymers or biopolymer aggregates/particles in natural biomaterials not easily dissolving in water require disaggregation for them to be used in preparing a film-forming suspension (hydrocolloid) in water, that allows uniform matrix formation for films and coatings during water evaporation.

High-pressure homogenization (HPH), ultrasound, and irradiation may be used to prepare film-forming suspensions containing adequately small biopolymer particles that form films with appropriate tensile and barrier properties. HPH disaggregates biopolymer particles using shear forces and pressure by accelerating the biopolymer to a high velocity within (micro) channels and then impinging them on a solid surface (Bouaouina, Desrumaux, Loisel, & Legrand, 2006; Hayes & Kelly, 2003; Sanchez, Pouliot, Gauthier, & Paquin, 1997). Biopolymer suspensions introduced to a HPH system are accelerated to a high velocity within (micro) channels and then made to simultaneously impinge with each other on a solid surface. Intense disruptive forces are generated under high pressure when the fluids collide with each other,

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causing the suspensions to be sheared and the biopolymer aggregates/particles to be disrupted (McClements, 2005). Size reduction of aggregates or particles of biopolymers by HPH has been reported (Hayes & Kelly, 2003; Sanchez et al., 1997). Irradiation can degrade biopolymers by free radicals (•OH and •H formed from water molecules exposed to irradiation) that interrupt molecular bonding and structuring of the polymers (Woods & Pikaev, 1994). Biopolymers are disrupted by free radicals formed from irradiation through their effects on molecular bonding and polymer structure (Woods & Pikaev, 1994). Irradiation has been applied to pectin, alginate, pectin, milk protein, and gelatin, and chitosan to produce low molecular weight substances (Cho, Kim, & Rhim, 2003; Choi, Ahn, Lee, Byun, & Park, 2002; Kang, Jo, Lee, Kwon, & Byun, 2005) and to form film-forming materials (Brault, D'Aprano, & Lacroix, 1997; Jo, Kang, Lee, Kwon, & Byun, 2005). Ultrasound treatments involve formation, growth, and implosion of tiny gas bubbles or cavities in biopolymer suspensions. Implosion of cavities results in micro-streaming and localized high shear that can be used for the separation of biopolymers (Zhang, Feng, Niu, & Eckhoff, 2005).

We hypothesized that HPH, irradiation, and ultrasound treatments can de-polymerize or deagglomerate biopolymers in the film-forming suspension of DMM, allowing the formation of continuous and uniform films after drying film-forming suspensions. Thus, the objectives of this research were to (1) develop edible biopolymer films from DMM and (2) evaluate the effects of HPH, irradiation, and ultrasound treatments and those of the film formulation (concentrations of glycerol and soy lecithin) on the formation of DMM-based films and rheological, moisture barrier, water solubility, tensile, color, and structural properties of the film-forming suspensions or films.

2. Material and methods

2.1. Materials

Oil was extracted from *S. alba* 'IdaGold' seeds by cold pressing (Brown, Davis, Erickson, Brown, & Seip, 1998). Approximately 90% of the oil was removed from the seed using a mechanical seed crusher (Borek & Morra, 2005; Peterson, Auld, & Thompson, 1983). The material produced during the pressing procedure consisted of irregularly shaped flakes approximately 1 mm thick and ranging in diameters from 1 to 3 cm. The meal remaining after oil extraction was not treated in any additional manner prior to use as the film base material (DMM). The DMM contained polysaccharides, proteins, and lipids of 45.2, 35.0, and 8.7 g/100 g meal, respectively (dry basis). The composition analysis was conducted by Korea Food Research Institute (Sungnam, Korea). Glycerol, used as a plasticizer to improve film flexibility, was purchased from Sigma–Aldrich Co. Ltd. (St. Louis, MO, USA). Soy lecithin (hydrophilic–lipophilic balance (HLB) value: 8), used as an emulsifier to help provide uniform distribution of the biopolymers in film-forming suspensions, was purchased from Alfa Aesar Co. (Ward Hill, MA, USA).

2.2. Film preparation

The mustard meal was ground in a Scienceware Micro mill (Bel-Art Products, Pequannock, NJ, USA) and then sieved to prepare a fine powder (<500 μm). The powder was stored in screw-capped glass bottles at 4 °C until used. Mustard meal was mixed with water (3% (w/w)) and the mixture was processed with a high shear probe mixer (Ultra-Turrax, Model T25, IKA-Works, Inc., Wilmington, NC, USA) at 20,000 rpm for 5 min. The homogenate was then treated by HPH (138 MPa), ultrasound (400 W), or gamma irradiation (10 or 20 kGy) to de-polymerize and/or deagglomerate particles of DMM in the colloid. Treatment conditions for preparing film-forming suspensions using HPH, ultrasound, and irradiation were determined from a preliminary study testing different parameters of HPH pressure (20,000 and 30,000 psi) and the number of HPH passes (2, 5, and 10 passes), ultrasound treatment time (10, 30, and 60 min), and temperature (5, 30, 60, and 90 °C). More descriptions on each treatment follow. The treated suspension was maintained at 90 °C for 30 min in a water bath and then cooled on ice. After cooling, glycerol (plasticizer) and soy lecithin (emulsifier) were added to suspensions and homogenized with the Turrax T25 mixer at 20,000 rpm for 5 min. Glycerol and soy lecithin were mixed into the suspension at 20 or 50% (w/w) of DMM powder (16.7 and 33.3% (w/w, dry basis)) and 0, 0.5, or 2.0% (w/w) (dry basis), respectively. The 50% glycerol was used only in the high-pressure-homogenized film-forming suspension. The mixture was then degassed under vacuum. Films were cast by pipetting the degassed mixture onto Teflon plates (15.5 cm i.d.) resting on a leveled granite surface. The amount of the film-forming suspension pipetted was selected to produce a 0.1 mm-thick film. For example, the amount pipetted onto the plate to produce a film that had a WPI–glycerol–soy lecithin ratio of 1:0.5:0.008 (33.3% glycerol and 0.05% soy lecithin (dry basis)) was 77 g. The film-forming mixtures were dried for 3 days at 23 ± 2 °C/35 ± 5% relative humidity (RH). Dried films were peeled intact from the casting surface and stored in a chamber (Tenney-10 model TTUFR-40240, Tenney Engineering, Inc., Union, NJ, USA) controlled at 23 ± 2 °C/50 ± 2% RH for 2 days to equilibrate before testing.

2.3. Depolymerization processes

2.3.1. High-pressure homogenization

A 1-L DMM-water suspension was introduced into a high-pressure homogenizing processor (M-110S Microfluidizer® Materials Processor, MA, USA). The homogenizer with a H10Z (100 μm) interaction chamber was run at 138 MPa. The suspension (1 L) was passed through the processor for 10 passes. The temperature of the suspension was monitored during sample processing and maintained between 23 and 45 °C.

2.3.2. Gamma irradiation

Irradiation was done at the USDA irradiation facility (Food Safety Intervention Technologies Research, ERRC, PA, USA). A Lockheed Georgia Company (Marietta, GA, USA) self-contained ^{137}Cs radiation source was used for all exposures. The dose rate (0.095 kGy/min) was verified according to methodology outlined by ASTM E 2116-00 using dosimeters obtained from the National Institute of Standards and Technology. The temperature during irradiation was maintained at 4.0 ± 1 °C by the gas phase of a liquid nitrogen source that was introduced directly into the top of the sample chamber. The absorbed dose was verified by use of 5-mm alanine pellet dosimeters that were attached to the sides of the sample bags, which were then measured using a Brucker EMS 104 EPR Analyzer (Billerica, MA, USA). Radiation doses of 10 and 20 kGy were used in this study.

2.3.3. Ultrasound treatment

A 500-mL DMM suspension was treated by ultrasound using a ultrasound processor (UP400, Hielscher Ultrasound Technology, Teltow, Germany) at 30 °C for 30 min and 400 W (24 kHz (frequency) and 120 μm (wavelength)). The cycle was set for continuous run and amplitude at 100%. A refrigerated circulating water bath was used to keep sample at 30 ± 2 °C.

2.4. Rheological properties

Rheological properties of film-forming suspensions were measured following the method of Chiou et al. (2006) using