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Effects of flaxseed oil concentration on the performance of a soy protein isolate-based emulsion-type film

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ABSTRACT

Biodegradable edible films have the potential to either replace or reduce the amount of synthetic packaging utilized by the food industry. The overall goal of this research was to investigate the effect of flax seed oil concentration (1–10%) on the mechanical, moisture barrier and swelling properties of soy protein isolate (SPI) (5.0% w/w SPI, 40% w/w glycerol) emulsion-based films. Film forming solutions showed a bimodal oil droplet distribution with peak sizes occurring at <10 and ~100 μm. As the oil content increased, the size distribution shifted towards smaller droplet sizes. An equal size ratio was noted at the 5.0% oil content level. All film forming solutions were pseudoplastic in nature, where viscosity increased from 18 to 58 mPa (at 1 s^{-1}) as a function of oil content (3% to 10%). Tensile strength of formed films reached a maximum at 5.35 MPa at the 5% w/w oil level, whereas tensile elongation increased from 11.3% to 22.2% with increasing oil content. Puncture strength and deformation, as well as water vapour permeability was relatively independent of the oil content. Moisture content and swelling properties of formed films were found to both decrease from 22.8% to 18.7%, and from 3114% to 1209%, respectively as the oil content was raised from 1 to 10%, and films became darker, redder and more yellow in colour as the percentage of flax seed oil increased.

1. Introduction

Research in the area of edible biodegradable films is on the rise as the food industry seeks to reduce the use of synthetic petroleum-based materials by the food industry (Zhang & Mittal, 2010). Movement away from synthetic petroleum-based materials arises due to its non-renewable nature, which contributes to the cost of these materials and recent insecurity for procuring these resources (Zhang & Mittal, 2010). Edible biodegradable packaging is advantageous due to its renewable nature, ability to degrade by direct consumption or be composted (Denavi et al., 2009; Janjarasskul & Krochta, 2010); and as a carrier for bioactive compounds, nutrients, (Debeaufort, Quezada-Gallo, & Voilley, 1998; Dhall, 2013; Elsabee & Abdou, 2013; Falguera, Quintero, Jiménez, Muñoz, & Ibarz, 2011; Janjarasskul & Krochta, 2010; Mei, Zhao, Yang, & Furr, 2002); or antimicrobial/fungal agents (Appendini & Hotchkiss, 2002; Coma et al., 2002; Ramos et al., 2012). This makes biodegradeable packaging attractive for applications involving improving consumer health or extending a product's shelf life. Despite these advantages, there are also several disadvantages that inhibit their widespread use in the market. These films are costly to produce and its physical properties (*i.e.* control over moisture transfer, gas permeability, colour) are not comparable to petroleum-based products (Zhang & Mittal, 2010).

Edible biodegradable packaging is commonly produced using proteins, lipids and polysaccharides. In general, proteins and polysaccharides tend to form stronger films with good gas barrier properties, but are poor moisture barriers (Falguera et al., 2011; Hettiarachchy & Eswaranandam, 2005; Janjarasskul & Krochta, 2010; Vargas, Pastor, Amparo, McClements, & González-Martínez, 2008). In contrast, lipid-based films tend to be weaker and have poor gas barrier properties, but good moisture control (Falguera et al., 2011; Janjarasskul & Krochta, 2010). However, most research found within the literature focuses on composite-type films, in order to make use of properties of both biopolymers and lipids (Falguera et al., 2011). Composite films are typically produced *via* layer-by-layer lamination or *via* emulsification. The former faces issues surrounding cracking and/or delamination, whereas the latter faces issues surrounding in-homogeneity to the film matrix due to differences in lipid droplet size distributions.

Soy protein isolate (SPI) is a commonly used material for producing biodegradable films. SPI is comprised of two main types of protein: β-conglycinin and glycinin, which are typically referred to as 7S and 11S globulins, respectively (Zhang & Mittal, 2010). These two proteins have a distinct impact on appearance, as films produced from the 11S fraction tend to be opaque, and those produced from the 7S fraction are translucent but contain creases (Zhang & Mittal, 2010). Films are formed through careful formulation of the film forming solution (*e.g.*, addition of plasticizers, pH, temperature pre-treatments of the protein), followed by casting, drying, and conditioning at a constant relative humidity (Brandenburg, Weller, & Testin, 1993). The drying time

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of films was reported to produce differences in the texture and properties of the films. Films which were dried quickly at 95 °C and 30% relative humidity (RH) were reported to be thinner, stronger, and stiffer, but have lower water vapour permeability than those dried at 21 °C and 50% RH (Alcantara, Rumsey, & Krochta, 1998). Additionally, SPI films that are formed without plasticizers (*e.g.*, glycerol) are brittle, therefore plasticizers are required when producing usable films (Park, Hettiarachchy, Ju, & Gennadios, 2002). Protein concentrations in film forming solutions may vary in the range from 5% (w/w) to 10% (w/w) SPI, whereby tensile strength of the film increases with protein content (Park et al., 2002).

Flaxseed oil is a rich source in the essential omega-3 fatty acid, α-linolenic acid (ALA). Flaxseed oil is thought to have health benefits beyond nutrition such as its ability to protect against cardiovascular disease (Fitzpatrick, 2012). The purpose of incorporating it into films was to increase the nutritional value of the film and utilize the hydrophobic nature of the oil to reduce the moisture penetration and water vapour permeability of the films. Factors such as polarity, lipid saturation, and homogeneity have been reported to affect the water vapour permeability of these films whereby no one factor specifically dictates this property (Janjarasskul & Krochta, 2010; Morillon, Debeaufort, Blond, Capelle, & Voilley, 2002; Pérez-Gago & Krochta, 2005).

The overall research goal of this research is to produce an emulsion-based composite film using SPI and flaxseed oil that offers excellent mechanical properties and moisture barrier properties.

2. Materials and methods

2.1. Materials

SPI (94.87% protein wet basis) was donated by Archer Daniels Midland for this study (ProFam 974, Decatur, IL, USA). Milli-Q water was generated by a Millipore Milli-QTM system for water purification (Millipore Corporation, Milford, MA, USA). Chemicals utilized in this study were reagent grade and obtained from Sigma-Aldrich Canada Ltd. (Oakville, ON, Canada), with the exception of L-ascorbic acid, which was acquired from BDH Inc. (Toronto, ON, Canada). Flaxseed oil was donated by Bioriginal Food and Science Corp. (Saskatoon, SK, Canada).

2.2. Formation of SPI films

SPI films were produced by utilizing a casting method reported by Chang and Nickerson (2013). A stock solution comprised of 5% SPI powder (w/w) (corrected for protein content) was dispersed into Milli-Q water, and stirring (500 rpm) for 10 min at room temperature (21–23 °C) with a mechanical stir plate. This stock solution was then adjusted to a pH of 9.0 using 1.0 M NaOH, followed by the addition of glycerol at a ratio of 40% w/w total protein weight which was further stirred for an additional 10 min. This stock solution was then heated to 85 °C and stirred constantly for 30 min utilizing a combination of hot plate and stir plate. The stock solution was then cooled for 45 min at room temperature under continuous stirring. To this stock solution, Tween 80 was first added at proportions of 0.5%, 1.5%, 2.5%, 3.75% and 5.0% w/w; followed by the addition of flaxseed oil at proportions of 1%, 3%, 5%, 7.5% and 10% w/w; and finally with the addition of ascorbic acid, as an antioxidant, at proportions of 0.5%, 1.5%, 2.5%, 2.75% and 5.0% w/w all based on protein weight, respectively. In between each addition, the stock solution was stirred for 10 min to ensure homogeneity. The solution was then homogenized utilizing a Polytron MR 2100 (Kinematica Ag, Switzerland) at 15,000 rpm for 5 min. This film forming solution was immediately cast onto polyfluoroethylene (PTFE) moulds (10 cm length, 10 cm width, 1 mm depth) whereby additional solution on top of the moulds was removed with a straight edge. The films were then dried overnight at room temperature. After drying, the films were removed from the moulds and conditioned in desiccators set at 54% RH (using a saturated magnesium nitrate solution) at room temperature (21–23 °C) for 48 h before testing. The thickness of each film was determined by the average of ten measurements made with a digital micrometer (Model 62379-531, Control Company, USA).

2.3. Rheological properties of the film forming solution

2.3.1. Small deformation rheology

Small deformation rheology was used to capture the viscous properties of film forming solutions. Approximately 1.46 mL of the emulsion was placed onto a AR-G2 Rheometer (TA Instruments, New Castle, DE) equipped with a 40 mm diameter 2° acrylic cone where sample viscosity was measured as a function of shear rate (2 to 200 s^{-1}) and 10 data points were collected per logarithmic decade.

2.3.2. Droplet size

The droplet size distribution of these emulsions was captured using a Mastersizer 2000 equipped with the Hydro 2000s wet dispersion accessory (Malvern Instruments, Malvern, UK). Emulsions were dispersed using the Hydro 2000s wet dispersion accessory until optical obscuration reached ~12%. Droplet size distributions were determined according to the Mie Theory which uses the difference in refractive index between droplets and the dispersing medium to predict the light scattering intensity. The ratio of refractive index (RI) was 1.112 which accounts for the RI of flaxseed oil at 1.479 and the dispersion medium at 1.330. Droplet size measurements were reported as a volume-surface mean diameter ($d_{3,2}$):

$$d_{3,2} = \frac{\sum_{i=1} n_i \cdot d_i^3}{\sum_{i=1} n_i \cdot d_i^2} \quad (1)$$

where n_i is the number of droplets of diameter d_i (μm) (McClements, 2005).

2.4. Mechanical testing

Puncture strength (PS, Newtons (N)) and deformation (PD, mm) were measured for each film for all concentrations of flaxseed oil. Mechanical properties were determined utilizing a texture analyser (Texture Technologies Corp., New York, USA) by the method of Gontard, Guilbert, and Cuq (1992). Triplicate tests for each film were performed, where testing was completed by placing films on a puncture mould (65.6 mm diameter), beneath a cylindrical probe with a smooth edge of 4 mm diameter. Films were punctured at a speed of 1 mm/s with the probe moving perpendicular to the film. A computer was used to record the force-deformation data; where PD was given as the change in length from the original at the rupture point of the film and the PS was expressed as the maximum force in N applied onto the film before puncture.

Percentage tensile elongation (TE) and tensile strength (TS) at the rupture point for each triplicate film were determined by a Texture Analyser (Texture Technologies Corp., New York, USA). Films were tested by stretching preconditioned films cut into 2.5 × 8 cm held in grips 50 mm apart at a speed of 300 nm/min. Stress–strain data was recorded and used to determine TE and TS. TE was defined as the percentage of strain observed by the films when they broke (Chang & Nickerson, 2013). TS was defined as the maximum force a film can accommodate before it breaks.

2.5. Water vapour permeability

Water vapour permeability (WVP) of the films was measured by the method used by Chang and Nickerson (2013). Films were cut specifically to fit the cups designed for this WVP experiment (dimensions: outer cup height: 2.65 cm; outer cup radius: 2.50 cm; inner cup height: