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Improving the physical and moisture barrier properties of *Lepidium perfoliatum* seed gum biodegradable film with stearic and palmitic acids

Samira Seyedi, Arash Koocheki*, Mohebbat Mohebbi, Younes Zahedi

Department of Food Science and Technology, Ferdowsi University of Mashhad (FUM), PO Box 91775-1163, Mashhad, Iran

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ABSTRACT

Stearic and palmitic fatty acids (10%, 20% and 30%, W/W gum) were used to improve the barrier properties of *Lepidium perfoliatum* seed gum (LPSG) film. The impact of the incorporation of fatty acids into the film matrix was studied by investigating the physical, mechanical, and barrier properties of the films. Addition of stearic and palmitic fatty acids to LPSG films reduced their water vapor permeability (WVP), moisture content, water solubility and water adsorption. Increasing fatty acid concentration from 10% to 30%, reduced the elongation at break (EB). Lower values of tensile strength (TS) and elastic modulus (EM) were obtained in the presence of higher fatty acids concentrations. Incorporation of fatty acids led to production of opaque films and the opacity increased as function of fatty acids concentration. Results showed that moisture content, water solubility and WVP decreased as the chain length of fatty acid increased. Therefore, LPSG-fatty acids composite film could be used for packaging in which a low affinity toward water is needed.

1. Introduction

Synthetic polymer materials used in the packaging industries are non-biodegradable and therefore lead to environmental pollution, which pose serious ecological problems [1,2]. Thus, the polymer materials which are biodegradable have being paid more and more attention [3,4]. Edible and biodegradable natural polymer films offer alternative packaging with lower environmental cost. The main natural biopolymer films are obtained from polysaccharides, lipids and proteins [5].

The search for new renewable resources for the production of edible and biodegradable materials has steadily increased in recent years. A variety of polysaccharides and their derivatives have been tested for potential use as biodegradable films. Polysaccharide films have relatively good oxygen barrier properties, but are highly permeable to water due to their hydrophilic nature [6,7].

On the other hand, lipids offer high water barrier properties. Fatty acids, lipids and waxes are commonly used in edible film to reduce its water vapor permeability since these materials are hydrophobic and, thus, are good barriers against moisture migration. Addition of lipids was reported to reduce the water vapor permeability in polysaccharide films such as kefiran [8], arabinoxylan [9], mesquite gum [10], chitosan [11,12], hydroxypropyl methyl cellulose [13], gellan [14] and cellulose [15].

Lepidium perfoliatum seed gum (LPSG) can be used as an essential viscosity builder [16]. In general, the thickening action of LPSG in aqueous system could stabilize the dispersions and emulsions [17]. Our previous work showed that LPSG can produce films with good appearance but weak water barrier properties [18]. Since, lipid component can serve as a good barrier to water vapor, stearic and palmitic fatty acids could be used to combine the advantages of both lipid and hydrocolloid components. For this purpose, mechanical, physical, barrier and microstructure properties of LPSG–fatty acid composite films were investigated in this study.

2. Materials and methods

2.1. Materials

L. perfoliatum seeds were obtained from a local medical market in Mashhad, Iran. Glycerol, Palmitic acid, Tween 80 and potassium sulfate (used to equilibrate films at 97% RH) were purchased from Merck Co. (Germany). Stearic acid was obtained from Sigma (America). Anhydrous calcium chloride was procured from Scharlau (Spain). Calcium nitrate $6H_2O$ (used to equilibrate films at 50 ± 2% RH) obtained from Ghatran Chemistry (Iran).

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* Corresponding author. Tel.: +98 915 313 9459; fax: +98 511 878 7430.
E-mail address: koocheki@um.ac.ir (A. Koocheki).

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2.2. Extraction of LPSG

L. perfoliatum seeds were manually cleaned to remove all foreign substances such as stones, dust, dirt, chaff and broken seeds. The LPSG was extracted based on the method described by Koocheki et al. [19].

2.3. Preparation of film-forming emulsions

Aqueous solutions of 0.6% (w/v) LPSG were prepared by dispersing LPSG powder in distilled water at room temperature (23 ± 2 °C). Glycerol (60%, w/w of LPSG) was added and solutions were magnetically stirred for 15 min and kept overnight. Palmitic and stearic fatty acids (10%, 20% and 30% w/w) and Tween-80 (2% w/w of fatty acid) were added to the solutions and heated at 80 °C for 45 min. The solutions were then cooled and homogenized with IKA T25 digital homogenizer (Ultra-Turrax, Germany) for 3 min at 9000 rpm.

2.4. Emulsion droplet size and size distribution measurements

The droplet size of the emulsions was measured using Zetasizer nano laser particle size analyzer (Zetasizer Nano ZS, Malvern Instruments, UK). The *Z*-average diameter would be determined by dynamic light scattering (DLS) technology. The Nanosizer operates by detecting back-scattered laser-light and comparing the coherence of scattering patterns as a function of time. Decay of coherence is converted to apparent particle sizes and distributions through the supplied software, which relies on Mie theory calculations.

2.5. Preparation of films

The solutions were poured onto a series of Petri dishes (diameter 15 cm) and placed on a leveled surface. To control film thickness, volume of each film-forming solution was always 110 ml. Films were dried at 45 °C for 18 h, in a drying box (Soroush oven-so-2005, Iran) with gentle fan forced air circulation. Dried films were peeled off the casting surface and stored in a desiccator containing saturated calcium nitrate to meet the required relative humidity before analysis.

2.6. Film density

Film density (ρ_s) was calculated directly from the film weight and dimensions [20] according to the following equation:

$$\rho_s = \frac{m}{A \times \delta} \tag{1}$$

where A is the film area (4 cm × 4 cm), δ is the film thickness (cm), m is the film dry mass (g) and ρ_s is the dry matter density of the film (g/cm^3).

2.7. Moisture content

The moisture content was determined by film weight loss using oven drying at 103 ± 2 °C for 24 h (Soroush Oven-SO-2005). The weight loss of the sample was determined and the moisture content was calculated using the following equation:

$$\text{Moisture content}\,(\%) = \frac{Mi - Md}{Mi - Mp} \times 100 \tag{2}$$

where *Mi* is the weight of the Petri dish and film sample before drying, *Mp* is the weight of the Petri dish, and *Md* is the weight of the Petri dish and film sample after drying.

2.8. Moisture absorption

The moisture absorption (MA) was determined according to the method of Angles and Dufrense [21]. In brief, dried films (2 cm × 2 cm) were placed in a desiccator containing anhydrous Calcium Chloride (0% RH) at 25 °C for 24 h. After initial weighing, they were conditioned in a desiccator containing saturated potassium sulfate solution (97 ± 1% RH). The weight of the samples was recorded every 24 h until constant weight was reached. The moisture absorption was calculated using Eq. (3):

$$MA = \frac{W_t - W_0}{W_0} \tag{3}$$

where W_t and W_0 are the weights of the sample after time t and the initial weight of the sample, respectively.

2.9. Water solubility

The films' solubility in water was determined according to the method reported by Gontard et al. [22]. Pieces of LPSG films (2 cm × 2 cm) were cut with a scalpel, dried at 103 ± 2 °C for 24 h in a laboratory oven, and weighed to obtain the initial film dry weight. Each piece of film was placed into a plastic tube with 50 ml of distilled water. After 24 h of immersion with gentle mechanical agitation, the remaining pieces of films were filtered and dried at 103 °C to reach a constant weight (final dry weight). The water solubility was calculated using Eq. (4):

$$\text{Water solubility}\,(\%) = \frac{\text{inital dry weight} - \text{final dry weight}}{\text{intial dry weight}} \times 100. \tag{4}$$

2.10. Water vapor permeability (WVP) and water vapor transmission rate (WVTR)

The WVP test was conducted based on ASTM method E96 [23]. The circular test cups containing 3 g anhydrous calcium chloride (0% RH, assay cup) were sealed by films without pinholes or defects (0.00018 m^2 film area). To maintain the gradient of relative humidity of approximately 97% across the film, cups were placed inside desiccators containing saturated potassium sulfate solution at the test temperature of 25 °C. The permeability cups with the films were weighted at intervals of 2 h during the first 12 h and finally after 24, 48 and 72 h. Linear regression was used to calculate the slope of a fitted straight line in a graph of variation of mass versus time. The WVTR and the WVP were calculated from Eqs. (5) and (6) respectively:

$$WVTR = \frac{\text{Slope}}{A} = \frac{\Delta m}{\Delta t \times A} \tag{5}$$

$$WVP = \frac{WVTR \times X}{\Delta p} \tag{6}$$

where $\Delta m/\Delta t$ is the weight of moisture gain per unit of time (g/s), A is the area of the exposed film surface (m^2), X is film thickness (m) and ΔP is the water vapor pressure difference between the two sides of the film (KPa).

2.11. Contact angle

The surface hydrophobicity of the films was estimated by the sessile drop method, based on optical contact angle method. A droplet of distilled water (20 μL) was put over the surfaces of the samples and images were taken right after using a digital camera. The angle between drop and surface of the film was measured using the Image J Software. Measurements were performed at three locations for each specimen.