



Alyssum homolocarpum seed gum-polyvinyl alcohol biodegradable composite film: Physicochemical, mechanical, thermal and barrier properties



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ABSTRACT

Films made from *Alyssum homolocarpum* seeds gum (AHSG) have poor mechanical and barrier (to oxygen) properties. In the present study poly vinyl alcohol (PVA) was used to improve the physicochemical properties of AHSG films. Results indicated that the addition of PVA significantly increased the moisture content, solubility, elongation at break (EB) and transparency while it decreased the density, oxygen permeability, chroma, water contact angle and Young modulus of AHSG based films. Films with higher AHSG to PVA ratios had lower water vapor permeability (WVP). The light barrier measurements presented low values of transparency at 600 nm for PVA/AHSG films, indicating that films were very transparent while they had excellent barrier properties against UV light. Results for FTIR, DSC and SEM showed a clear interaction between PVA and AHSG, forming a new material. These results indicated that PVA/AHSG blend films had good compatibility.

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1. Introduction

Owing to their astatic appearance, low density, low cost and formability, petroleum based plastics are widely used as packaging materials especially for foods. With reducing petroleum resources and rising environmental concerns, chemically synthesized plastics lacking biodegradability are in direct conflict (Mikkonen & Tenkanen, 2012). Therefore, utilizing biodegradable polymers from renewable sources provides opportunities to reduce waste through biological recycling in order to achieve a sustainable ecosystem (Maran, Sivakumar, Sridhar, & Immanuel, 2013). In recent years, using biodegradable materials for food packaging has elevated because of consumer awareness of the environmental damage and their potential use in the replacement of non-biodegradable packaging (Oh, Wang, Field, & Aglan, 2004).

Polysaccharide-based films have the potential to be used in food packaging due to their excellent oxygen, aroma, and oil barrier properties. However, using polysaccharide films is limited by their poor mechanical properties and water vapor and CO₂ barrier properties.

Alyssum homolocarpum seed gum (AHSG) has a great potential to be used as a new source of biodegradable film due to its

proper thickening/gelling action. The majority of AHSG is carbohydrate (85.33%) with small amount of uronic acid (5.63%) (Hesarinejad, Razavi, & Koocheki, 2015). AHSG has low molecular weight (3.66×10^5 Da) with relatively flexible chain and medium intrinsic viscosity (18.34 dl/g) at 25 °C (Hesarinejad et al., 2015). The major sugars compositions of AHSG are galactose (82.97%), glucose (5.7%), rhamnose (5.04%), xylose (2.72%), mannose (3.04%) and arabinose (0.53%) and it is likely a galactan-type polysaccharide. AHSG behaves like a typical polyelectrolyte because of the presence of carboxyl and hydroxyl groups (Hesarinejad et al., 2015). Gum extracted from AHSG can be used as thickening, fat replacer and stabilizer agent (Koocheki, Kadkhodaei, Mortazavi, Shahidi, & Taherian, 2009a; Mortazavi, Shahidi, Razavi, & Taherian, 2009b; Koocheki et al., 2010).

Generally films based on single biopolymer such as carbohydrates are highly sensitive to environmental conditions and generally acquire low mechanical resistance (Kanatt, Rao, Chawla, & Sharma, 2012). Polymer blending is an effective approach to improve mechanical and physical properties of the resulting films (Kanatt et al., 2012). In recent years many researchers have chosen a gum or combination of gum with other biopolymers to achieve desired film properties.

Poly (vinyl alcohol) (PVA) is a synthetic, water-soluble and partially crystalline polymer with technological potential as a biodegradable polymer (El-Kader, Hamied, Mansour, El-Lawindy, & El-Tantaway, 2002). For these reasons, PVA has widely been

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used as textile sizing and adhesive agent in textile and paper industries. Films based on PVA are fully biodegradable, odorless, transparent and non-toxic. Such films have excellent chemical resistance, mechanical strength and physical properties (Ramaraj, 2007). PVA is suitable for blending with natural polymers as it is highly polar and soluble in water (Chiellini, Cinelli, Fernandes, Kenawy, & Lazzeri, 2001). Several investigations have been carried out on blending PVA with other polysaccharides such as starch (Yoon, Park, & Byun, 2012) and chitosan (Bonilla, Fortunati, Atarés, Chiralt, & Kenny, 2014).

To date, use of polysaccharide films for food packaging has been strongly limited because of their poor barrier and weak mechanical properties. Our pre-tests showed that although ASHG film has good appearance, it does not have a firm and flexible structure. Therefore, to improve the physical, mechanical, barrier, thermal and optical properties of AHSG film it was blended with PVA. For this goal, the interactions among two polymers and their effect on properties of blend films were investigated.

2. Material and methods

2.1. Material

Alyssum homolocarpum seeds used in this study were purchased from the local market at Mashhad, Iran. Poly vinyl alcohol (PVA) (M_w : 30000–70000 g/mol, degree of hydrolysis: 89–90%, and viscosity: 4–6 cP), glycerol and calcium chloride were obtained from Sigma Aldrich (Fluka, Sigma-Aldrich, MO, USA). Sodium hydroxide, calcium nitrate, sodium and calcium chloride were purchased from Merck Corporation (USA). Ethanol 96% was purchased from Razi Corporation (Tehran, Iran). At all preparations and analyses, deionised water was used, unless otherwise specified. All other reagents used were of analytical grade.

2.2. Gum extraction

AHSG extracted and purified according to the procedure described by Koocheki et al. (2010). *A. homolocarpum* seeds were first cleaned to remove all foreign substances and then soaked in preheated deionized water at $35 \pm 1.0^\circ\text{C}$ (water to seed ratio of 40:1). The pH was monitored continuously and adjusted at 4 using 0.1 mol/L NaOH and/or HCl. The seed-water slurry stirred with an electric mixing paddle throughout the entire extraction period (1 h). Seeds were then discarded and ultimately, the slurry was precipitated with ethanol (96%) at ratio of 1:3 (slurry:ethanol). The extraction yield of AHSG was 294.1 g kg^{-1} . The precipitate was then freeze dried (Martin Christ Freeze Dryer, 1500w, 380v, 0.9 vacuum, German), milled and sieved using a mesh 100 sifter and stored at room temperature.

2.3. Preparation of PVA/AHSG blend films

Films were obtained by solution casting procedure. In order to prepare the film, AHSG powder (1.5% w/w) was dissolved in distilled water for 20 min at room temperature ($23 \pm 2^\circ\text{C}$). The solutions were then allowed to hydrate for 24 h at 4°C . Thereafter, the solution was homogenized using a rotor-stator homogenizer (Ultraturrax D125, Janke and Kunkel, Germany) at 13,500 rpm for 1 min. PVA (1.5% w/w) was dissolved in an aqueous solution at 95°C for 60 min until complete dissolution. For PVA/AHSG blends, PVA was incorporated into the previously hydrated AHSG solutions in seven different ratios (100:0, 80:20, 60:40, 50:50, 40:60, 20:80 and 0:100). These films were brittle and difficult to peel off. Therefore, glycerol was added with PVA/AHSG to glycerol ratio of 1–0.5. Finally, PVA/AHSG solutions were stirred for 30 min and centrifuged (7000g for 10 min) to remove air bubbles. Film forming

dispersions were spread over petri dishes (diameter 15 cm). To control film thickness, volume of each film-forming solution poured onto a plate was always 75 ml. Films were dried in an air convection heat oven (Soroush oven-so-2005, Iran) at 50°C for 24 h and then, peeled off and conditioned at 53% RH at room temperature ($25 \pm 2^\circ\text{C}$) for further analysis.

2.4. Film thickness

The film thickness was measured at six random positions with a micrometer (QLR digital-IP54, China) to the nearest 0.001 mm.

2.5. Film density

Film samples of $3 \text{ cm} \times 3 \text{ cm}$ were maintained in a desiccator with P_2O_5 (0% RH) for a week and weighed to determine the film density. Film density was calculated using following equation:

$$\rho = \frac{m}{A \times d} \quad (1)$$

where A is film area (9 cm^2), d is film thickness (cm), m is film dry mass (g) and ρ is dry matter density of films (g/cm^3). Film density was expressed as the average of five determinations.

2.6. Moisture content

The moisture contents (MC) of films equilibrated at 53% RH and 25°C , were analysed by drying the samples in an oven (Soroush Oven-SO-2005, Iran) at 110°C until constant weight (dry sample weight). Moisture content (%) was calculated using Eq. (2):

$$\text{MC} = \frac{m_i - m_d}{m_i} \quad (2)$$

where, m_i and m_d are initial and dried sample weight, respectively. Three replicates were analysed per film formulation.

2.7. Water sorption isotherms

Films (1.5–2.0 g) were first conditioned at 0% RH (H_2SO_4) at 25°C for 72 h. After weighing, samples were placed in desiccators at 25°C and equilibrated to different water activity using oversaturated solutions of LiCl, MgCl_2 , $\text{Mg}(\text{NO}_3)_2$, NaCl and BaCl_2 which gave a_w values of 0.11, 0.328, 0.53, 0.75 and 0.902, respectively (Rockland, 1960). Samples were weighed periodically (0.00001 g precision) during 4 weeks until the equilibrium state was reached. Experimental sorption isotherms were fitted to Guggenheime-Anderson-de Bøer (GAB) model Eq. (3) in the entire a_w ranges. The model for sorption isotherm was simulated using MATLAB software (MATLAB r2015b).

$$M_e = \frac{M_0 \cdot C \cdot K \cdot a_w}{(1 - K \cdot a_w)(1 + (C - 1)K \cdot a_w)} \quad (3)$$

where M_e is the equilibrium moisture content on dry basis, a_w is water activity, M_0 is monolayer moisture content, and C and k are equation parameters, both being temperature dependent and related to the water sorption energy in the film.

2.8. Solubility in water

Solubility in water was defined as the percentage of the dry matter of film which is solubilized after 24 h immersion in water (Shi et al., 2007). Film specimens were kept in a desiccator containing dry calcium sulfate till they reached constant weight. Then, about 500 mg of each film were immersed in beakers containing 50 mL of distilled water at 23°C for 24 h with periodical gentle manual agitation. Films were then removed from water and placed back in

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