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Starch behaviors and mechanical properties of starch blend films with different plasticizers

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ABSTRACT

The main objective of the study was to gain insight into structural and mechanical starch behaviors of the plasticized starch blend films. Mechanical properties and starch behaviors of cassava (CS)/and mungbean (MB) (50/50, w/w) starch blend films containing glycerol (Gly) or sorbitol (Sor) at 33% weight content were investigated. It was found that tensile strength TS and %E of the Gly-CSMB films were similar to those of MB films; but%E of all Sor-films was identical. TS of plasticized films increased when AM content and crystallinity increased. When Sor was substituted for Gly, crystallinity of starch films and their TS increased. The CSMB and MB films had somewhat a similar molecular profile and comparable mechanical properties. Therefore, it was proposed the starch molecular profile containing amylopectin with high $\overline{M}_w$, low $\overline{M}_w$ of amylose, and the small size of intermediates may impart the high TS and%E of starch films.
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1. Introduction

Production and functional properties of edible starch films and coatings have been extensively investigated during the past decade, due to their vast potential uses in the food and pharmaceutical industries (Pavlath & Orts, 2009). Among their various attributes, the mechanical properties of starch films are of interest to researchers since they have important functions for film applications (Alves, Mali, Beléia, & Grossmann, 2007; Bae, Cha, Whiteside, & Park, 2008; Mali, Sakanaka, Yamashita, & Grossmann, 2005; Parra, Tadini, Ponce, & Lugão, 2004; Zullo & Iannace, 2009).

Amylose (AM) content, crystallinity, glass transition temperature (T_g), types and contents of plasticizers, and sources of starch are major factors governing the mechanical properties (Gupta, Brennan, & Tiwari, 2012). Starches containing high AM display excellent film-forming ability and good mechanical properties (Bae et al., 2008; Chaudhary, Torley, Halley, McCaffery, & Chaudhary, 2009; Li, Liu, Zou, Yu, Xie, Pu, Liu, & Chen, 2011). The content of B-type crystallinity also increased film stiffness as well as TS at break (Chaléat, Halley, & Truss, 2014). In addition, brittleness of starch films increased when the films possessed high glass transition temperature (T_g) (Myllärinen, Partanen, Seppälä, & Forssell, 2002).

Types and contents of plasticizers also affect the mechanical properties of starch films. Glycerol (Gly) and sorbitol (Sor) are the most widely used polyol plasticizers for starch-based films. Their excellent ability to prevent film cracking during handling and storage was reported; and their effectiveness is possibly due to the close similarity between their chemical structures and the structure of starch polymer (Mali et al., 2005). Gly exhibited a superior plasticization effect to Sor at equivalent weight content (Laohakunjit & Noomhorm, 2004; Mali et al., 2005; Talja, Helen, Roos, & Jouppila, 2007). Nonetheless, Cuq, Gontard, Cuq, & Guillbert (1997) disputed that the effect was mainly due to higher molecular content of glycerol. And yet, Zhang and Han (2006) demonstrated the reverse effect which was the Gly- plasticized pea starch films had lower%E than Sor-plasiticized films at the same molar content. Both plasticized films also exhibited similar TS.

Mungbean (MB) starch, containing high AM (~30%), is available in tropical countries. The costly starch produces clear, colorless, and high-strength gel. Thus, it is considered as the best raw material for cellophane noodle production. MB starch films displayed the highest TS among other starch film samples, and the starch imparted an excellent ability to form pharmaceutical hard capsules (Bae et al., 2008; Prachayawarakorn, Hommannee, Phosee, & Chairapaksatien, 2010).

Cassava (CS) starch, one of the major industrial starches, is abundant and inexpensive. CS starch films were odorless, tasteless, and colorless. The edible films also provided good flexibility. The drawback of CS films was their rather low tensile strength (TS) compared

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to that of films from other starches (Alves et al., 2007; Mali et al., 2005; Thirathumthavorn & Charoenrein, 2007).

Blending of starches can alter functional properties of starch products such as gelatinization, pasting, rheological, gelation, and mechanical properties (Hongsprabhas, 2007; Liu & Lelièvre, 1992; López, Lecot, Zaritzky, & García, 2011; Rachtanapun, Pankan, & Srisawat, 2012; Waterschoot, Gomand, Fierens, & Delcour, 2015). The properties of starch blends can be classified into additive behavior, i.e., the property of the blends can be predicted from each starch component, and non-additive behavior, i.e., the property of the blends cannot be predicted, indicating any interactions occurred between starches (Puncha-arnon, Pathipanawat, Puttanlek, Rungsardthong, & Uttapap, 2008).

Based on the available information, it is beneficial to develop edible films from CS starch partly substituted for MB starch, yet maintaining their mechanical properties comparable to those of MB films. Besides, little information is known about the molecular profile, microstructure, and morphology of starch blend films. In this study, the CS/MB (50/50, w/w) starch blend films containing glycerol or sorbitol at the same weight content were prepared. Their mechanical properties, starch molecular weight profile, and polymer morphology were investigated, and compared with individual starch films. The objectives of the study were to gain insight into structural, thermal, and mechanical starch behaviors of the plasticized starch blend films, and to produce the plasticized CS/MB blend films possessing mechanical properties closed to those of plasticized MB films.

2. Materials

Mungbean (MB) starch (Sitthinan, Bangkok, Thailand) and cassava (CS) starch (E.T.C. International Trading, Bangkok, Thailand) were purchased locally. Glycerol and sorbitol were supplied by Ajax Finechem (New South Wales, Australia).

3. Chemical composition analyses

Protein, ash, moisture and crude fat content of starch were determined using standard official methods of AOAC (2000). Apparent amylose content was measured by the colorimetric method of Chrastil (1987).

4. Film-casting experimental design

The study was a 3 × 2 factorial design using two starches – cassava (CS) and mungbean (MB) – a CSMB starch blend, and two types of plasticizers. Native starches were mixed with distilled water at a concentration of 5% w/w (dry basis, db). Each suspension was heated at 80 °C while magnetically stirring at a speed of ~350 rpm. The CS and MB starch suspensions were stirred for 1 h and 3 h, respectively, to obtain clear solutions. A blend of CS and MB solutions was prepared by mixing the clear solutions of CS and MB starches, prepared separately as described, at a weight ratio of 50:50. The MB starch solution was prepared 2 h prior to the CS starch solution. The CSMB blend was then further stirred for 20 min at 80 °C in order to obtain a homogeneous solution. Then, two types of plasticizer (glycerol and sorbitol) were individually added to samples of each solution at 33% of starch weight. The molar ratio of glycerol and sorbitol per 100 g of starch dispersion was 0.018 mol and 0.009 mol, respectively. The film-forming solutions were continuously stirred using the same speed at 80 °C for 15 min. Each film solution (35 g) was poured into a plastic Petri dish (140 mm diameter). The dishes containing solutions were dried in a hot-air oven at 60 °C for 6 h. The films were peeled off and kept in a desiccator at 52% relative humidity (RH) and ambient temperature for not more than 4 weeks, to be used for further analyses. Unplasticized films (CS, MB and a 50:50 blend of CS and MB starches) were also prepared and stored in the same manner, to be used as controls.

5. Characterization of starch-based films

5.1. Mechanical and thermal properties

Tensile strength, elongation at break and Young's modulus of the films were determined using a universal testing machine (model 5965; Instron, Norwood, MA, USA), according to ASTM standard method D882-02 (ASTM, 2002).

Thermal properties of unplasticized and plasticized starch films were determined by differential scanning calorimetry (DSC) (model DSC 1, Star^e System; Mettler-Toledo, Greifensee, Switzerland). Each sample (10 mg, db) was weighed in an aluminum pan, and the analysis was performed under nitrogen atmosphere. The samples were heated from −70 °C to 250 °C at 5 °C/min, then cooled to 25 °C at 20 °C/min, and reheated again from 0 °C to 250 °C at 5 °C/min.

Glass transition temperatures of the films (10 mm width × 50 mm length) were determined in the tension mode using a dynamic mechanical analyzer (DMA) (Eplexor®; Gabo Qualimeter, Ahlden, Germany). The static load and dynamic load were 1 N and 0.3 N, respectively. The maximum static strain and dynamic strain were set at 0.3%. Measurements were performed at a frequency of 1 Hz using a temperature range from −100 °C to 100 °C at 2 °C/min.

5.2. Crystallinity and microstructure of native starches and starch films

Crystallinity of native starches and unplasticized and plasticized starch films was measured by an X-ray diffractometer (X'Pert; Philips, Amsterdam, Netherlands). Each sample was scanned over a diffraction angle range of $2\theta = 5-35$ °C using a scan rate of 0.03°/s. Percent of relative crystallinity was calculated as the ratio of the area above a smooth curve connected peak baselines to the total area over the diffraction angle 5°–35°. The data was analyzed by Jade software.

Plasticized starch films (20 mm × 20 mm) were iodine vapor stained using Lugol's solution (1% iodine (I_2) and 2% potassium iodide (KI) in water) in a closed chamber for 2 h. The microstructure of starch films was investigated under a light microscope (LSM 5 PASCAL; Zeiss, Oberkochen, Germany) with and without a polarized filter.

5.3. Molecular weight profiles of native starches and starch films

The molecular weight profiles of native starches, a dry blend of both starches (50:50 w/w), and three unplasticized starch films (CS, MB and CSMB blend) were determined using the a high-performance size-exclusion chromatography (HPSEC) system, according to the method of Israkarn, Na Nakornpanom, and Hongsprabhas (2014). Six pullulan standards (1 mg/mL) (Showa Denko, Tokyo, Japan) were used to construct a standard curve. All samples were defatted by the AOAC method (AOAC, 2000) prior to the analyses. The amylose content of all samples was analyzed using the ratio of the peak area of amylose and amylopectin.

5.4. FTIR spectroscopy

Fourier transform infrared (FT-IR) spectra of unplasticized and plasticized films were recorded in attenuated total reflectance (ATR) mode using a Bruker Tensor 27 FTIR spectrometer (Bruker, Billerica, MA, USA). The spectra were recorded from 500 cm^{-1} to 4000 cm^{-1}.

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