



Effects of octenylsuccination on physical, mechanical and moisture-proof properties of stretchable sweet potato starch film



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ABSTRACT

The high humidity sensitivity of glycerol plasticized starch films badly limited their applications for various foods. Stretchable sweet potato starch films with improved moisture-proof property were successfully obtained by using equivalently mixed octenylsuccinated/native starches (OSS/NS). FT-IR analysis evidenced the successful synthesis of OSS and its presence in OSS/NS blend films. The effects of the degree of substitution (DS) of OSS on the properties of the resulting blend films were evaluated. The incorporation and DS of OSS were of much importance in determining the properties of the blend films. In contrast with the control film, OSS/NS blend films presented significantly improved moisture-proof property as indicated by the lower values in water solubility and water vapor permeability, and the higher values in contact angle and oil permeability. The amplitudes of these improvements varied with the increasing DS of OSS. Meanwhile, the stretchability, elongation at break and transparency of OSS/NS blend films were higher than those of the control film and increased with the increasing DS of OSS. Oppositely, the results of XRD showed that OSS/NS blend films owned lower crystallinity compared with the control film. These variations were virtually ascribed to the hydrophobicity of OSS and the plasticization of octenyl groups in film formation. Frankly speaking, incorporating OSS into sweet potato starch film is a double-edged sword, providing positive effects by enhancing moisture-proof property, stretchability, elongation at break and transparency while simultaneously triggering deterioration in tensile strength and/or structural homogeneity.

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1. Introduction

Stretchable films produced by polyethylene are widely used as skin packaging or shrink wrapping for foods, such as fruits and vegetables (Rao & Rao, 2002). These films can directly pack foods according to their size and shape, maintaining their quality and increasing their shelf-life. With growing concerns regarding environmental pollution and energy crisis, there has been an increasing interest in biodegradable films, commonly based on renewable and natural polymers, due to their potential as replacements for synthetic plastic packaging. According to previous reports, proteins (Wang et al., 2014), lipids (Fabra, Talens, Gavara, & Chiralt, 2012)

and carbohydrates such as cellulose (Martínez-Sanz, Lopez-Rubio, & Lagaron, 2013), pullulan (Zhu, Sheng, & Tong, 2014), chitosan (Mei et al., 2013) and starches (Laohakunjit & Noomhorm, 2004) are widely used in the fabrication of biodegradable films.

Among these natural polymers, starch has been considered as the most promising candidate for manufacturing biodegradable films because of its low cost, abundance, renewability and biodegradability (Alves, Mali, Beleia, & Grossmann, 2007). However, starch-based films exhibit several shortcomings such as brittleness, high sensitivity to moisture and poor mechanical strength, especially for its elongation at break. Reports of elongation at break values of pure cassava, potato, waxy corn, maize corn and rice starch films (4.60%, 5.29%, 2.28%, 4.23% and 1.37%, respectively) are very low (Alves et al., 2007; Laohakunjit & Noomhorm, 2004; Li et al., 2011; Osés, Niza, Ziani, & Maté, 2009), in contrast with synthetic stretchable films made from high density polyethylene (HDPE) or low density polyethylene (LDPE) (300% and 500%, respectively) (Jiménez, Fabra, Talens, & Chiralt, 2012). Accordingly,

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the poor stretchability of starch-based films restricts their use in food packaging.

Several strategies have been employed to improve the stretchability of starch-based films, such as adding plasticizer (Laohakunjit & Noomhorm, 2004), starch modification (Woggum, Sirivongpaisal, & Wittaya, 2014) and synthetic polymer incorporation (Arvanitoyannis, Biliaderis, Ogawa, & Kawasaki, 1998). Among of these strategies, using plasticizer is the easiest and most effective approach to endow starch films with considerable flexibility and extensibility. The most commonly used plasticizers included glycerol, sorbitol, xylitol, poly(ethylene glycol)200 and poly(ethylene glycol)400 (Laohakunjit & Noomhorm, 2004; Talja, Helén, Roos, & Jouppila, 2007). Glycerol, the most often used small molecule plasticizer, easily gains access between the polysaccharide chains through hydrogen bonds, reducing intermolecular polysaccharide interactions, increasing intermolecular spacing, and therefore increasing film stretchability (Jiménez et al., 2012). Dias, Müller, Larotonda, and Laurindo (2010) found that the elongation at break of rice starch film was substantially improved (from 2.8% to 59.8%) with increasing concentrations of glycerol (from 20% to 30% (w/w, glycerol/starch)). Similarly, the elongation at break of cassava starch film was sharply increased from 5.2% to 153.2% with a glycerol concentration increase from 20% to 45% (w/w, glycerol/starch) (Alves et al., 2007). However, high glycerol content induced stretchability endows films with undesirably high water vapor permeability being sensitive to humidity due to the strong hydrophilic nature of glycerol. This restricts the use of stretchable starch films that use glycerol as the plasticizer of high humidity foods.

To improve the moisture-proof property of biopolymer films, several strategies have been widely investigated: 1) blending with lipids, such as stearic acid, palmitic acid, oleic acid and beeswax (Fabra et al., 2012; Yang & Paulson, 2000). 2) blending with hydrophobic polymers, such as carboxymethyl cellulose (Ghanbarzadeh, Almasi, & Entezami, 2011) and nanocrystalline cellulose (Saxena, Elder, & Ragauskas, 2011). 3) hydrophobization of biopolymer, such as esterification with octenyl succinic anhydride (OSA) (Zhou, Ren, Tong, & Ma, 2009).

Hydrophobization of starch with OSA could potentially produce a modified starch with a higher hydrophobicity than native starch (Sweedman, Tizzotti, Schäfer, & Gilbert, 2013). Moreover, the recent work done by Zhong, Peng, Yang, Cao, and Sun (2013) indicated that the pendent 2-OSA half-ester groups in octenylsuccinated xylan could act as an internal plasticizer for xylan films. In this context, we theoretically hypothesized that high glycerol content induced stretchable starch films made from octenylsuccinated starch would harbor both enhanced water vapor resistance and stretchability. Therefore, the aim of this work was to investigate the effects of octenylsuccination of sweet potato starch on the properties of resultant stretchable starch films, with an emphasis on improving extensibility and moisture-proof property.

2. Experimental

2.1. Materials

Native sweet potato starch (NS) was purchased from Jintian Agriculture Industry Co., Ltd, (Chongqing, China). 2-Octenyl succinic anhydride (Product No. 416487) was purchased from Sigma Chemical Co. (USA). Glycerol was obtained from Guangfu Reagent Chemical Co., Ltd. (China). All chemicals were of analytical grade.

2.2. Preparation of octenylsuccinated sweet potato starch

Octenylsuccinated sweet potato starch (OSS) was prepared according to the method reported by Ruan, Chen, Fu, Xu and He

(2009). Sweet potato starch was suspended in distilled water with agitation. The pH of the suspension was adjusted to locate in the range of 8–10 by adding 3% (w/v) NaOH solution. Then OSA was added and the sweet potato starch suspension was kept at 40 °C for a settled time to allow for thorough octenylsuccination. The reaction was stopped by adjusting the pH of suspension to 6.5 with 3% (v/v) HCl solution. OSS was harvested by centrifugation (4000 rpm, 10 min), and the resultant pellet was washed twice with water and twice with 70% (v/v) aqueous alcohol, and finally oven-dried at 45 °C overnight. The degree of substitution (DS) was determined by titration (Ruan et al., 2009). Four OSSs with significantly different DS were synthesized in the present study, denoted as OSS1, OSS2, OSS3 and OSS4 with DS of 0.0134, 0.0236, 0.0325, and 0.0425, respectively. The detailed reaction parameters for the synthesis of these OSSs were shown in [Supplementary material 1](#).

2.3. Film formation

Starch films in this work were prepared using a casting method. To prepare the film-forming solution, NS (3.5 g) and OSS (3.5 g) were suspended into 175 mL distilled water to achieve a total starch concentration of 4% (w/w). The resultant starch slurry was heated at 95 °C for 30 min with continuous stirring to complete starch gelatinization. Then glycerol (2.1 g) was added as a plasticizer and was stirred for a further 15 min. Then the slurry was placed in a boiling water bath and immediately degassed under vacuum (0.1 MPa) for 30 min. The warm solution was cast onto a polymethyl methacrylate plate and oven-dried at 50 °C for 8 h. After completely cooling at room temperature, the film was peeled from the polymethyl methacrylate plate and balanced at 25 °C and 53% RH by placing in a cabinet over a saturated Mg(NO₃)₂ solution for at least for 5 days prior to analysis. The film prepared with NS was used as control.

2.4. Determination of film thickness, moisture content, color parameters and opacity

Film thickness was measured using a micrometer (Shan Brand, Guilin, China) having a precision of 1 μm. Measurements were taken at nine random locations on the films, and an averaged result was presented. Moisture content was determined by oven-drying at 105 °C until a constant weight was attained (Galus, Mathieu, Lenart, & Debeaufort, 2012). Color parameters of the film were measured by using an UltraScan Pro1166 colorimeter (Hunterlab, USA) and expressed as the lightness (L^*), chromaticity parameter a^* (red–green) and b^* (yellow–blue). The color difference (ΔE) was calculated according to the difference between the color parameters of the sample film and the control film. Film opacity was evaluated according to the method reported by Zhu et al. (2014). Rectangular film specimens were placed across a light path in a spectrophotometer cell (UV-2450, Shimadzu Corporation, Japan), and its light transmittance (T_{600}) was measured at 600 nm against an empty cell. The opacity (T) of the film was calculated using the following equation: $T = -\log T_{600}/\text{thickness}$. The T value was positively related to the opacity of film.

2.5. Film characterization

Film samples were characterized by fourier transform infrared spectroscopy (FT-IR), x-ray diffraction (XRD), and scanning electron microscopy (SEM). FT-IR spectra of dried film sheets (105 °C for 2 h) were recorded over 500–4000 cm⁻¹ using a Perkin–Elmer Spectrum 2000 FTIR spectrometer (Shelton, Connecticut, USA). X-ray diffraction patterns of films were obtained by using an Ultima IV X-ray diffractometer (Rigaku Corporation, Japan) in the grazing-incidence x-ray diffraction mode. The equipment was operated at

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