



Mechanical, barrier and morphological properties of starch nanocrystals-reinforced pea starch films



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ABSTRACT

To characterize the pea starch films reinforced with waxy maize starch nanocrystals, the mechanical, water vapor barrier and morphological properties of the composite films were investigated. The addition of starch nanocrystals increased the tensile strength of the composite films, and the value of tensile strength of the composite films was highest when starch nanocrystals content was 5% (w/w). The moisture content (%), water vapor permeability, and water-vapor transmission rate of the composite films significantly decreased as starch nanocrystals content increased. When their starch nanocrystals content was 1–5%, the starch nanocrystals dispersed homogeneously in the composite films, resulting in a relatively smooth and compact film surface and better thermal stability. However, when starch nanocrystals content was more than 7%, the starch nanocrystals began to aggregate, which resulted in the surface of the composite films developing a longitudinal fibrous structure.

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1. Introduction

A large amount of contemporary research seeks environmentally friendly material to prevent the pollution that petroleum-based plastic waste produces (Famá, Goyanes, & Gerschenson, 2007; Jiménez, Fabra, Talens, & Chiralt, 2012; Mali, Grossmann, García, Martino, & Zaritzky, 2002, 2006; Psomiadou, Arvanitoyannis, & Yamamoto, 1996). The development of entirely biodegradable polymers from agricultural resources has received considerable attention (García, Ribba, Dufresne, Aranguren, & Goyanes, 2011; Lu, Weng, & Cao, 2006; Mathew & Dufresne, 2002; Mathew, Thielemans, & Dufresne, 2008). Starch is probably the most promising renewable naturally biodegradable polymer since it is a versatile, cheap, and abundant biopolymer that does not itself have a fossil source (Savadekar & Mhaske, 2012). High-amylose starches possess more advantages than low-amylose ones and the films prepared from high-amylose starches have better mechanical strength and gas barrier properties (Lourdin, Della-Valle, & Colonna, 1995; Palviainen et al., 2001; Wolff, Davis, Cluskey, Gundrum, & Rist, 1951). Pea starches (PS) have a high amylose content, mostly ranging from 30 to 60%, varying with species (Hoover,

Hughes, Chung, & Liu, 2010), and this makes them very useful in forming films (Palviainen et al., 2001).

However, like other hydrocolloids, starch films exhibit several drawbacks when compared to plastic polymers, such as their hydrophilic character and poor mechanical properties, which can be improved by blending with other compounds in the film (Briassoulis, 2004). Sun, Chu, Xiong, and Si (2013) reported that pea starch films blended with peanut protein isolate have better mechanical properties than pure pea starch film. Singha and Himanshu (2014) found that reinforcing films of blended starch and polyvinyl alcohol with silk fibroin could apparently improve their mechanical and water-resistance properties. Shi, Wang, Li, and Adhikari (2013) reported that water vapor permeability (WVP) of corn starch film is significantly higher than that of starch films containing spray dried and vacuum freeze dried starch nanoparticles.

Starch is a natural semicrystalline biopolymer which is biodegradable, renewable and abundant. Starch-based nanoparticles are widely used as drug carriers due to their non-toxicity, biocompatibility and biodegradability (Han, Gao, & Liu, 2013; Rajisha, Maria, Pothan, Ahmad, & Thomas, 2014). In recent years, nanowhiskers and nanocrystals have also been applied to reinforce biodegradable and non-biodegradable polymeric matrices (Lu, Weng, & Zhang, 2004; Samir, Alloin, Gorecki, Sanchez, & Dufresne, 2004). The diameter of starch nanocrystals (SNCs) prepared by sulfuric acid hydrolysis is about 20 nm, which results in many great achievements when compounded with polymer

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matrices. Chen, Cao, and Huneault (2008) reported the poly(vinyl alcohol)/pea starch nanocrystals nanocomposite films containing 5 and 10 wt% of nanocrystals content possessed improved physical properties than pure PVA film. Using a soy protein isolate (SPI) as a matrix, Zheng, Ai, Chang, Huang, and Dufresne (2009) reported an increase in strength and Young's modulus at pea starch nanocrystal loading levels lower than 2 wt%. Wang and Zhang (2008) described the preparation of high-strength water-borne polyurethane (WPU)-based elastomer reinforced with 1–5% waxy maize starch nanocrystals.

However, the preparation and characteristics of high-amylose starch-based films containing SNCs, to our knowledge, have not been reported so far. Therefore, this work investigates mechanical, barrier, and morphological properties of pea starch films reinforced with different amounts of SNCs to determine whether SNCs can improve the functional properties of pea starch films. This will provide the theoretical basis to produce biodegradable starch films that have better mechanical and barrier properties.

2. Materials and methods

2.1. Materials

Pea starch (about 40% amylose) was purchased from Tianjin Tingfung Starch Development Co., Ltd. (Tianjin, China). Waxy maize starch (98% amylopectin) was kindly provided by the same company. Glycerol was purchased from Sigma–Aldrich Co., Ltd. (St. Louis, MO., United States). Sulfuric acid (H_2SO_4), potassium carbonate (K_2CO_3) and other analytical grade reagents were purchased from Shanghai Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

2.2. Preparation of SNCs

SNCs were prepared by acid hydrolysis of waxy maize starch according to the method of Angellier, Choïnard, and Molina-Boisseau (2004). Typically, 15% starch dispersion was prepared with a diluted sulfuric acid solution (3.16 M, 300 mL) and stirred constantly at 200 r/min with 40 °C water curing treatment for seven days. After hydrolysis, the suspensions were washed using successive centrifugations in distilled water until neutrality, then the precipitate was vacuum freeze-dried to obtain SNCs.

2.3. Preparation of PS/SNCs films

Following the method of Sun, Sun, and Xiong (2013), (5.0 g of pea starch and 1.5 g of glycerol plasticizer were added to 100 mL deionized water to form starch-plasticizer dispersions. Each dispersion was thoroughly stirred for 30 min in a thermostatic water bath at boiling temperature, and then cooled to 50 °C. Aliquots of SNCs (0, 0.05, 0.15, 0.25, 0.35 and 0.45 g respectively) were added to 50 mL deionized water, then treated by ultrasonic wave (KQ-400KDE, Kun-Shan Ultrasonic instrument Co., Ltd., Jiangsu, China) at 100 W for 10 min to ensure uniform dispersion. These SNCs dispersions were then added to the starch-plasticizer dispersions, stirring continuously for 30 min. After that, the film-forming dispersions were degassed, cast at 5.6 mg solids/cm² in a framed and leveled polytetrafluoroethylene plate having a diameter of 15 cm diameter) and dried at 40 °C overnight. The films were peeled from the casting plates and conditioned for at least one week at 43% relative humidity, conditioned by using a K_2CO_3 saturated solution.

2.4. Thickness of PS/SNCs films

The thickness of the films was determined using a digital micrometer (Vernier, China, 0.001 mm accuracy). Reported

thickness values were the mean of ten measurements taken from each film sample. The average value for each film was used to calculate its tensile properties and water vapor permeability.

2.5. Opacity

The transparency of the films were determined by measuring their light absorption at wavelength of 600 nm, using a UV–visible spectrophotometer Shimadzu 1601 PC (Tokyo, Japan), according to the method described by Maran, Sivakumar, Sridhar, and Thirugnanasambandham (2013). The film specimens were cut into strips (1 cm × 4 cm) and placed directly in the spectrophotometer test cell. Air was used as reference. Opacity was expressed as absorbance units per thickness unit.

2.6. Color measurement

Sample surface color was analyzed with a colorimeter (CR-400 Minolta Chroma Meter; Konica Minolta Sensing Inc., Tokyo, Japan) according to the method described by Jang, Shin, and Song (2011) with some modifications. Samples (50 mm × 50 mm strips) were placed onto a white standard plate, and Hunter values (L^* , a^* and b^*) were measured. The Hunter L^* , a^* , and b^* values for the standard plate were $L^* = 96.68$, $a^* = 0.14$, and $b^* = 1.94$. For each sample, five measurements were taken at different locations.

2.7. Mechanical properties

A TA. XT Plus Texture Analyzer (Lloyd Instruments, West Sussex, England) was used to determine the film's tensile strength (TS), elongation at break ($E\%$) and elastic modulus (M). Film specimens were tested as suggested by Mehyar, Al-Ismaïl, Han, and Chee (2012), with some modifications, and the tests were carried out according to the ASTM D828-97 standard test methods (ASTM, 1997). PS/SNCs composite films were cut into strips (1 cm × 10 cm). The strips were then preconditioned at 67% RH for 48 h inside a sealed desiccator containing saturated sodium chloride solution at room temperature (25 ± 1 °C). The films were loaded into the testing system, which was set at an initial sample length and grip speed of 2 cm and 100 mm/min, respectively. TS was calculated by dividing maximum load by the film's cross-sectional area. $E\%$ was expressed as the percentage of change in the original length of a specimen between the grips at break. M was the slope of the tensile stress–strain curve over the elastic (linear) region.

2.8. Measurement of the moisture content of PS/SNCs composite films

The moisture content of the films was determined using the gravimetric method (Carmen, Müllera, João, and Fabio, 2011). The weight of film samples (4 cm × 4 cm) was determined, then the samples were dried at 105 °C for 24 h, after which the weight of the dried samples was determined as well. The moisture content of PS/SNCs composite films is expressed in g water/g dry mass.

2.9. Measurement of water vapor permeability (WVP) and water-vapor transmission rate (WVTR)

The gravimetric method was used to determine the WVP and WVTR of the PS/SNCs composite films, as suggested by Latifa, Pfanstielb, and Makkara (2013) and the tests were carried out according to the ASTM E96-00 (ASTM, 2000) methodology. Circular film samples with a diameter of approximately 10 mm were placed over the mouth of the test cup and sealed using melted paraffin. The cup was pre-filled with anhydrous calcium chloride leaving 3 mm

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