



Effects of acid hydrolysis on the physicochemical properties of pea starch and its film forming capacity

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ABSTRACT

The effects of acid hydrolysis treatment on the properties of pea starch (PS) and PS-based films by solution casting were investigated. Native pea starch granules were partially hydrolyzed with 2% hydrochloric acid for 0.5, 1.0, 1.5, 2.0, and 2.5 h. Acid hydrolysis treatment lowered the molecular weight of amylopectin from 27.94×10^7 g/mol for native PS to 9.16×10^7 g/mol for the sample of 2.5 h hydrolysis, but had no obvious effect on the molecular weight of amylose. Micrographs of the hydrolysis starch granules exhibited surface erosion and fragmentation. The relative crystallinity slightly increased, whereas the peak, trough, final and setback viscosities decreased during acid hydrolysis. After acid hydrolysis treatment, the tensile strength of the pea starch film significantly increased and reached a maximum of 21.21 MPa with 2.0 h of acid hydrolysis.

1. Introduction

Starch is regarded as one of the most promising raw material for the production of edible and biodegradable films as it is a renewable, biodegradable, abundant, and widely available biopolymer (Jiménez, Fabra, Talens, & Chiralt, 2009). However, the poor mechanical properties and moisture barrier properties of the films made from native starch limit their extensive application in many fields (Mali, Debiagi, Grossmann, & Yamashita, 2010). The modification of starches by physical (Deep, Bawa, Riar, & Saxena, 2009; Zavareze et al., 2012) and chemical (Lopez, Garcia, & Zaritzky, 2008) processes has been an effective way to improve the performance of starch films.

The molecular weight, chain length distribution and crystallinity are important factors that influence the properties of polymer films (Cheng, Abd Karim, & Seow, 2007; Chung & Lai, 2007; Fonseca et al., 2015; Lazaridou, Biliaderis, & Kontogiorgos, 2003; Sothornvit and Krochta, 2000a, 2000b). Although the main industrial applications of acid-modified starches are found in the fields of paper, textile and gum candy fields, their use in edible and biodegradable films is increasing due to their good film-forming capacities (Lopez et al., 2008; Luchese, Frick, Patzer, Spada, & Tessaro, 2015). Acid-modified starches are particularly suited for applications utilizing the film-forming properties

of starch (Wurzburg, 1986, pp. 17–40). Acid modification could increase water solubility, degree of crystallinity, and gel strength of starch. Meanwhile, it could decrease molecular weight and viscosity of starch (Hoover, 2000; Luchese et al., 2015). These properties are desired for the ease of handling of the dispersion, pasting, casting and drying processes in the preparation of starch films.

Chung and Lai (2007) reported the effects of modification with 0.36% HCl in methanol at 25 °C and 45 °C on the properties of corn starch and its films. The results showed that acid modification increased the homogeneity of the corresponding films and decreased the moisture absorption rate and maximum moisture content. Acid modification with 0.25, 0.5 and 1.0 mol/L hydrochloric acid for 2 h increased the puncture and tensile strength of water chestnut starch films (Deep et al., 2009). Luchese et al. (2015) reported that hydrolysis by a specific concentration of hydrochloric acid could increase the homogeneity and water vapor permeability of a pinhao starch film. Up to now, the relationships between the physicochemical properties of acid-modified starches and the performance of the corresponding films are still not clear. Furthermore, the effects of the acid hydrolysis time on the properties of pea starch-based films have not yet been reported.

In this study, pea starch (PS) was chosen as the film-forming material because of its high amylose content, which favors film production

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(Li et al., 2011; Muscat, Adhikari, Adhikari, & Chaudhary, 2012). Pea starch hydrolysates were prepared by hydrochloric acid treatment. The properties of the hydrolysis pea starch and its films were studied. The aim of this study was to investigate the effects of acid hydrolysis treatment on the properties of pea starch and its film forming capacity and to obtain the appropriate hydrolyzation extent of pea starch for film preparation.

2. Materials and methods

2.1. Materials

Pea starch was provided by the Yingfei Food Co. Ltd. (ZhaoYuan, China). Standard P-82 pullulans were purchased from Showa (Denko KK, Tokyo, Japan). Sodium azide was analytical grade and obtained from Bodi Chemical Reagent Co. Ltd. (Tianjin, China). Sorbitol was of food grade standard and purchased from Jinqian Chemical Reagent Co. Ltd. (Jinan, China). All other chemicals were of at least analytical grade standard.

2.2. Methods

2.2.1. Acid hydrolysis of pea starch

Pea starch (172.46 g, dry base) was suspended in 350 mL HCl solution (2% by weight) in a 500-mL flask. The suspension was magnetically stirred at 45 °C for 0.5, 1.0, 1.5, 2.0 and 2.5 h, followed by filtering through What-man No. 1 filter paper. Then, samples were washed with 1M NaOH until the washings became neutral. After that, the acid-modified starches were washed four times with distilled water. The filter cake was dried at 50 °C for 24 h in an air oven. Finally, the dried samples were ground and passed through a 100 mesh sieve. The starches were sealed within separate airtight containers for at least 48 h. The samples were coded as PS for native pea starch and PS-0.5 h, PS-1.0 h, PS-1.5 h, PS-2.0 h and PS-2.5 h for samples treated with 2% HCl for 0.5, 1.0, 1.5, 2.0 and 2.5 h, respectively.

2.2.2. X-ray diffraction of the starches

At room temperature, a certain amount of starch prepared in section 2.2.1 was packed tightly into a sample cell compacted by a glass slide, the crystallinity of starch was determined by X-ray diffraction (XRD) on the sample table. X-ray diffractograms were recorded over an angular range (2θ) of 5°–30° at 0.02 intervals by a D8 Advance diffractometer (Bruker AXS GmbH, Karlsruhe, Germany) at 40 kV and 40 mA. From the scattering spectrum, the percentage of efficacious crystallinity was calculated as the ratio of the integrated crystalline intensity (sharp peaks) to the total intensity (both amorphous background and crystalline peaks) (Flores, Fama, Rojas, Goyanes, & Gerschenson, 2007; Liu, Wang, Kang, Cui, & Yu, 2018).

2.2.3. Starch granule morphology

A scanning electron microscope (QUANTA-FEG250, FEI, U.S.) was used to study the starch granule morphology. Starch samples were mounted on a circular aluminum stub, coated with gold, and then examined at an acceleration potential of 10 kV, with images being recorded.

2.2.4. Molecular weight distribution of the starches

The molecular weight distribution of the acid-modified starches was determined with high performance size exclusion chromatography (HPSEC). Methods for dissolution of samples and determination of molecular weight all referred to the method of Lin, Lee, and Chang (2003). Each sample solution was filtered through a 5.0- μ m syringe filter, 100 μ L of filtrate was injected into an HPSEC system.

The HPSEC system consisted of a refractive index (RI) detector (Optilab rEX, Wyatt Tech., USA), and a multiangle laser lightscattering (MALLS) detector (DAWN HELEOS, Wyatt Tech., USA), with a helium-

neon laser light source ($\lambda = 658$ nm). The chromatography columns were two TSK gel columns (7.8 mm ID \times 30 cm per column) in series (G4000 PWXL, G5000 PWXL; Tosohaas, Japan) combined with a PWXL-guard column (Tosohaas, Japan). A 0.1 M sodium nitrate solution containing 0.02% sodium azide was used as the mobile phase. Elution was performed at 60 °C at a flow rate of 0.5 mL/min. The refractive index of mobile phase was 1.331, the refractive index increment of starch molecules in solution was set to $dn/dc = 0.146$. The electronic outputs of the MALLS and RI detectors were collected by ASTRA software (ver. 5.3.4.20, Wyatt Tech., USA). The MALLS and RI signals were used to determine the molecule weight of amylopectin (the first fraction). Because of the reduced sensitivity of MALLS for small molecular weight species, the molecular weight of the second fraction (amylose and degraded amylopectin fragments) of starches was calculated by the RI signal using a calibration curve constructed from a series of pullulan standards with molecular weight of 78.8×10^4 , 40.4×10^4 , 21.2×10^4 , 11.2×10^4 , 4.73×10^4 , 2.28×10^4 , 1.18×10^4 and 0.59×10^4 Da.

2.2.5. Pasting properties of the starches

The pasting properties of the starches were analyzed by a Rapid Visco Analyser (RVA) Super 3 (Newport Scientific Pty Ltd., Warriewood, Australia). The AACC Approved Method 76–21 (American Association of Cereal Chemists, 2000) standard 2 profile for the computer was used. Starch (2.5 g, 14% moisture basis, m.b.) was mixed with 25 mL distilled water in the RVA sample canister. The peak viscosity (PV), trough viscosity (TV), final viscosity (FV), pasting temperature (PT), setback viscosity (SV) and breakdown viscosity (BV) were established.

2.2.6. Film preparation

Native and acid-treated starch films were prepared by solution casting. The amount of sorbitol was weighed and dissolved into distilled water and then starch was added to obtain a film-forming suspension. The starch concentration in the suspension was 4% (w/w) of overall water content independent of sorbitol concentration. Starch: sorbitol ratio was 100:40 (w/w). Film forming suspension was heated with continuous mixing at 90 °C for 30 min. Then, the suspension was cooled down to 50 °C to obtain the film forming solution. The air bubbles that formed during mixing were removed by placing the film forming solution into a desiccator under a vacuum until no bubbles were observed. Film forming solution was poured on teflon-coated plate. Starch-based films were obtained by evaporating water in an oven at 50 °C. All of the tested film samples were equilibrated at 23 ± 2 °C and 53% relative humidity in the constant temperature humidity chamber for at least 48 h prior to testing.

2.2.7. Mechanical properties

The mechanical properties of the films were determined by a TA-XT2i texture analyser (Stable Micro System, UK) according to ASTM D882-02 (ASTM. D882-02, 2002) with some modifications. Film strips (15 mm \times 80 mm) were stretched at 1 mm/s with an initial distance between grips of 50 mm. The tensile strength (TS, MPa) and elongation at break (E, %) were calculated with the following equations (Tang, Alavi, & Herald, 2008):

$$TS = \frac{L_p}{a} \times 10^{-6} \text{ MPa} \quad (1)$$

where L_p is the peak load (N) and a is the cross-sectional area of samples (m^2);

$$E = \frac{\Delta L}{L} \times 100 \quad (2)$$

where ΔL is the increase in length at the breaking point (mm) and L is the original length (mm).

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