



Structural and physicochemical properties of lotus seed starch treated with ultra-high pressure



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ABSTRACT

Aqueous lotus seed starch suspensions (15%, w/w) were subjected to ultra-high pressure treatment (UHP, 100–600 MPa) for 30 min. The effects of UHP treatment on the structural and physicochemical properties of starch were investigated. The SEM and laser diffraction particle size analysis revealed that UHP treatment affected the shape and size distribution of starch granules. The morphological structure of starch was completely destroyed at 600 MPa, indicating complete gelatinization. Analysis of HPSEC-MALLS-RI suggested that the dispersity index of UHP-treated starch were decreased from 1.28 to 1.11. According to XRD analyses, UHP treatment converted native starch (C-type) into a B-type pattern. The swelling power and solubility presented a significant decrease at 85 and 95 °C, but opposite trends were found at 55–75 °C. The DSC results indicated a reduction in gelatinization temperatures and enthalpy with increasing pressure treatment. The RVA viscograms revealed that UHP-treated starch showed a decreased breakdown and setback viscosity, reflecting lower retrogradation tendency compared to native starch.

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1. Introduction

Lotus (*Nelumbo nucifera* Gaertn.), which belongs to the Nymphaeaceae family, is a plant that has been widely cultivated in China, India, Thailand, South Korea, Japan, and Australia (Bhat & Sridhar, 2008; Kim & Shin, 2012). Lotus seeds, which are rich in carbohydrates, proteins, amino acids, and alkaloids, are widely used as food and pharmaceutical products in India, China, and Japan (Mani, Subramanian, Pillai, & Muthusamy, 2010; Rai, Wahile, Mukherjee, Saha, & Mukherjee, 2006). In our previous study (Zeng, Zheng, Lin, & Zhuo, 2009), we reported that the starch content of lotus seeds is approximately 500 g/kg (dry basis) with a high amount of amylose molecules (~40%, w/w). Lotus seed starch as other starches is used as a thickening, stabilizing, gelling, filling, and water retention agent in food and non-food applications.

However, the applications of native starch are limited by its low solubility in cold water, limited emulsification capability, easy retrogradation, poor storage stability under refrigerated and processing conditions, and variable shearing force and pH values (Singh, Kaur, & McCarthy, 2007; Whistler, BeMiller, & Paschall, 1984).

Ultra-high pressure (UHP) treatment is a non-thermal modification method. Over the last two decades, the use of UHP to gelatinize or physically modify starch molecules has been investigated at different processing conditions, such as pressure treatment, dwelling time, starch concentration, temperature, and starch source, to assess the mechanism of pressure-induced starch gelatinization and to understand the effects of UHP on the physicochemical characteristics of starch granules. It has been shown that UHP treatment is effective on physicochemical properties of starch polymers (Behzad & Saeideh, 2012). Studies have reported that UHP-treated starch has unique gelatinization and retrogradation properties compared to heat-gelatinized starch (Tian, Li, Zhao, Xu, & Jin, 2014; Vallons, Ryan, & Arendt, 2014). Compared to heat-gelatinized starch, UHP-treated starch has intact or partially denatured granules, lower quantity of released amylose, and lower initial rate of enzymatic reactivity (Douzals, Cornet, Gervais, & Coquille, 1998; Stolt, Oinonen, & Autio, 2001; Stute et al., 1996).

Abbreviations: UHP, ultra-high pressure; SEM, scanning electron microscopy; XRD, X-ray diffractometer; SEC-MALLS-RI, size exclusion chromatography connected with multi-angle laser light scattering and refractive index system; RVA, rapid visco analyzer; DSC, differential scanning calorimeter.

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Understanding the relationship between starch functionality and its molecular properties, such as structure and weight-averaged molecular weight (M_w), has been a long-term goal of food scientists (Zhong, Yokoyama, Wang, & Shoemaker, 2006). The differences in structure of amylopectin, such as molecular weight (M_w) and its distribution, can influence the physicochemical behavior of the onset swelling and gelatinization of starches and the texture of food products (Ma et al., 2007). In general, the molecular weight of polymers is determined by size exclusion chromatography (SEC). However, this measurement is challenging for starch samples because calibration standards are necessary and the accuracy of the method is not high. In recent years, SEC coupled to multi-angle laser light scattering (MALLS) and refractive index (RI) detectors has become a powerful tool for determining the molecular weight of starch polymers that does not require the use of calibration standards. (You & Lim, 2000).

The extent of starch gelatinization by UHP treatment is highly dependent on the starch source, pressure treatment, dwelling time, water content, and temperature (Kawai, Fukami, & Yamamoto, 2007). X-ray diffraction analyses reveal that starch molecules can be divided into “A” (cereal starch), “B” (tuber starch), or “C” (legume starch) types. B-type starches are more resistant to pressure than A- or C-type starches (Bauer & Knorr, 2005; Stute et al., 1996). Studies have mostly focused on the effect of UHP treatment on the structural and physicochemical properties of A- and B-type starches; however, there is limited information on the effect of UHP treatment on C-type starches. No studies have assessed the effect of UHP treatment on the structural and physicochemical of lotus seed starch, which is a C-type starch.

Therefore, the objective of this study was to evaluate the effects of UHP treatment on the structural and physicochemical properties of lotus seed starch. The structure of lotus seed starch was characterized by scanning electron microscopy (SEM), laser diffraction particle size analyzer, X-ray diffraction, and SEC–MALLS–RI. Furthermore, the swelling power, solubility, pasting, and thermal properties were studied and compared.

2. Materials and methods

2.1. Materials and sample preparation

Lotus seed starch (Green Field Fujian Food Co., Ltd., Fujian, China) was isolated according to the method reported by Zeng (2007). Fresh peeled raw lotus was homogenized (DS-200, Changzhou Xiangtian Experimentation Instrument Plant Co., Ltd., Jiangsu, China), mixed with a small volume of water, and filtered through a 100-mesh sieve (150 μm). The filtrate was collected and allowed to settle for 6 h at 4 °C. The resulting supernatant was discarded; the sediment was washed twice with distilled water and dried in an oven (DJG-9053A, Shanghai Yiheng Technology Co., Ltd., Shanghai, China) at 45 °C for 24 h. The dried material (starch) was filtered through an 80-mesh sieve (180 μm) and kept in a desiccator at room temperature. Lotus seed starch contained, on average, 10.25% (d.b.) moisture, 0.28% (d.b.) ash, 0.38% (d.b.) protein and 0.23% (d.b.) lipid.

2.2. UHP treatment

Starch was subjected to UHP treatment (high pressure press type 5 L-HPP-600 MPa; KeFa High Pressure Technology Co., Ltd., Baotou, China). In this study, 15% (w/w) of a starch–water suspension was transferred to polypropylene bags that were vacuum packed (–100 kPa). The starch suspensions were subjected at pressures of 100, 200, 300, 400, 500, and 600 MPa at room temperature (25 °C) for 30 min. The samples were pressurized at a rate of

approximately 1 MPa/s. When the UHP treatment was completed, the samples were vacuum filtered, and then dried in an oven at 45 °C for 24 h, filtered through an 80-mesh sieve (180 μm), and then kept in a desiccator at room temperature for further analysis.

2.3. Scanning electron microscope (SEM)

The dried starch samples were mounted on an SEM stub with double-sided adhesive tape and coated with a 50 nm thickness gold layer. Starch granule morphologies were examined in an SEM (PHILIPS-XL 30 ESEM, Philips-FEI, Netherlands) at an acceleration voltage of 20 keV and 1500 \times magnification.

2.4. Particle size distribution

Particle size distribution of the starch samples was assessed using a laser diffraction particle size analyzer (Malvern Mastersizer 2000, Malvern Instruments Ltd., England, UK). Starch samples were sonicated in water for 3 min. The average particle size distribution was determined by three parallel experiments once the shading rate reached 15%. Granule size was the average diameter calculated by the instrument. The refractive indexes of distilled water and starch were 1.33 and 1.52, respectively.

2.5. X-ray diffraction

X-ray diffraction analysis of the starch samples was performed in triplicate using an X-ray diffractometer (Bruker AXS, Bruker Corporation, Germany) under the following conditions, X-ray tube Cu-K α (nickel filter), 40 kV, voltage of 100 mA, scanning form of 2 θ of 5–40°, step width of 0.05°, and continuous scan.

2.6. Molecular weight distribution

HPSEC–MALLS–RI was used to determine the molecular weight distribution of starch. The mobile phase consisted of 90% DMSO with LiBr (50 mmol/L) filtered through a 0.22 μm PTFE filter and then degassed with ultrasound treatment. For SEC analysis, 12.5 mg sample of isolated starch was dispersed in 5 mL of 50 mM LiBr in DMSO at 90 °C for 2 h on a stirrer–heater module followed by stirring for 24 h on a magnetic stirrer at room temperature (Pu, Chen, Li, & Li, 2013). Dispersed samples were centrifuged at 13,500 $\times g$ for 15 min; the resulting supernatant was analyzed by HPSEC–MALLS–RI. Diluted starch dispersions, with concentrations from 0.02 to 0.2 mg/mL, filtered through 5 μm PTFE filter film (Millipore Co., USA) and then injected with a manual injector (1 mL injection loop) directly to a MALLS detector at a flow rate of 0.3 mL/min. The columns used were Ohpak SB-G (guard column, Shodex, Tokyo, Japan), Ohpak SB-806 HQ columns (Shodex, Tokyo, Japan) selected in series and maintained at 35 °C. In this experiment, the wavelength of the laser source was 623.8 nm (Zhong et al., 2006). M_w and M_n were calculated using the Astra V software according to the Zimm model. A second-order Berry method was used for curve fitting. M_w calculations were based on a mobile phase refractive index of 1.4785 and a dn/dc value of 0.066.

2.7. Swelling power and solubility

Swelling power and solubility were determined in triplicate by a modified method of Choi et al. (Choi, Kim, Park, Kim, & Baik, 2009). Starch sample (0.6 g, dry basis) was dispersed in distilled water (30 mL) in the 50 mL centrifuge tubes, and heated in a water bath at 55, 65, 75, 85, and 95 °C for 30 min. Every 5 min, the tubes were mixed in turbine mixer (QL-866, Kylin-bell Instrument manufacturing Co., Ltd., Jiangsu, China). The samples were then

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