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Impact of continuous or cycle high hydrostatic pressure on the ultrastructure and digestibility of rice starch granules

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ABSTRACT

The effects of continuous or 2-cycle high hydrostatic pressure (HHP) treatments (200 and 600 MPa) on the microstructure and digestibility of rice starches were investigated. The morphological and structural changes were characterized using polarized light microscopy, scanning electron microscopy, atomic force microscopy, X-ray scattering and ^{13}C CP/MAS NMR, and the starch digestibility was examined by *in vitro* hydrolysis. Results showed that HHP at 600 MPa significantly alters the microstructure and lowers the resistant starch (RS) compared with HHP at 200 MPa. Under the same pressure level, the two 15-min cycle treatment induced more structural disruption, gelatinization, disappearance of surface protrusion, and lower RS of rice starches than that of the continuous HHP treatment (30 min). Based on the results on RS, the two 15-min cycle HHP treatment at 200 MPa could be beneficial for improving the functionality of the rice starch.

1. Introduction

Rice starch comprises two homoglucans named amylose (AM) and amylopectin (AP) (Boluda-Aguilar et al., 2013). In the starch granule, the composition, shape, size, AM–AP ratio, ultrastructure of amylopectin, and crystalline structure of the granules contribute to the specific functionalities of rice food, including texture, consistency, and storage stability (Liu, 2005; Liu et al., 2010). In-depth understanding on the structure–function relationship is essential for the selection of appropriate processing technology and the development of high value-added rice products in the food industry.

High hydrostatic-pressure (HHP) processing is a non-thermal food processing method, in which foods are subjected to a high pressure ranging from 200 to 600 MPa for a given time with water as the pressure transmitting medium (San Martin et al., 2002). Previous studies have indicated that HHP is one of the advantageous processing treatments for improving palatability, flavor, and safety of cooked rice (Boluda-Aguilar et al., 2013; Deng et al., 2013; Yamakura et al., 2005). The resistance of starch microstructure to pressure is affected by various factors such as pressure level, model of pressure application (continuous or cycle), time, temperature, as well as the constituent and phase state of the food (Boluda-Aguilar et al., 2013; Katopo et al., 2002; Li et al., 2012; Liu et al., 2010; Vallons and Arendt, 2009). For example, Li et al. (2012) found that high pressure treatment at 120–600 MPa for 30 min converts the C-type X-ray pattern to the B-type-like pattern in mung bean starch. Nasehi and Javaheri (2012) suggested that potato starch is more resistant to pressure than rice, corn or tapioca starches due to their higher water content. X-ray diffraction showed that under 600 MPa (5% suspension, 15 min, 20 °C), A-type starch of taro still presents obvious birefringence, while the B-type starch of corn is partially destroyed (Liu et al., 2010). The sorghum starch treated by 400 MPa at 65 °C showed a significant loss in birefringence, and all granules lost their “Maltese Cross” after treatment at 600 MPa (Vallons and Arendt, 2009).

Compared with the continuous HHP treatment, the iterative compression and decompression during cycle pressurization could produce more powerful percussive action and shear effect, which leads to more significant structure damage of biomacromolecules and inactivation of microorganisms, thus shortening the treatment time and saving the cost (Liu et al., 2008a). The two-cycle HHP

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treatment resulted in slightly higher gelatinization but lower values in chewiness and resilience for jasmine rice in comparison with the continuous mode (Boluda-Aguilar et al., 2013). Multiple-cycle HPP treatment exerted significantly more inactivation on *Salmonella* and *Escherichia coli* O157:H7 compared to single cycle treatment (Morales et al., 2008). These previous studies demonstrated that cycle HHP treatments possess some definite advantages for improving the functionality and safety of food. However, little information is available about the comparable impact of continuous and cycle HHP on the ultrastructure modifications of starch or protein to date.

Rice has a relatively high glycaemic index (GI) compared with other starch foods (Mir et al., 2013). It has been well known that the rate and extent of starch digestion in the gastrointestinal tract vary depending on its amylose-amylopectin ratio, granule size, architecture, crystalline pattern, degree of crystallinity, surface pores or channels, degree of polymerization of starch, and processing techniques (Angioloni and Collar, 2012; Lerdluksamee et al., 2013; Mir et al., 2013). Therefore, various physical and chemical modifications have been employed to improve the starch digestibility by manipulating the microstructure, amylose and resistant starch content in starch granule. However, the effect of HHP on the digestible properties of rice starch has not been investigated yet. More understanding of the relationship between ultrastructural characteristics and digestible properties of rice starches after HHP treatment is very important for optimizing industrial applications and allowing consumers to select suitable rice products.

Therefore, the objectives of this study were to investigate the uniqueness of continuous or cycle pressure treatment on the granule morphology, structural characterizations and digestible properties of rice starch and *in vitro* digestion, and to explore the correlation between the ultrastructure and digestibility of rice starch.

2. Materials and methods

2.1. Materials

Rice starch powder (Y-KNA20120824, Jiangxi Golden Agriculture Biotech Co., Ltd., Jiangxi, China) was purchased at a local rice store. It was composed of 56.8% amylose, 39.1% amylopection, 0.74% protein, 7.34% moisture, 0.54% fat, and 0.17% ash based on the dry basis.

2.2. Sample preparation and HHP treatment

Starch–water suspensions were prepared by mixing rice starch powders with distilled water at 25 °C to a final concentration of 20% (w/w) total solids, and sealed in polyethylene-polyamide plastic bags. HHP treatments were performed in a HHP-750 unit (Kefa High Pressure Food Processing Inc., Baotou, China) with a 2.5 L of cylindrical pressure vessel and operational pressure range of 0–700 MPa. The bags were immersed in the high pressure vessel filled up with water and the temperature was maintained at 25 °C by a circulating water system. Continuous HHP treatments were carried out for 30 min at 200 MPa and 600 MPa, while cycle treatments consisted of two 15-min cycles at 200 MPa and 600 MPa. After the pressure treatments, the sample bags were opened and the samples were freeze dried in a Freezone 2.5 L Triad system (Labconco Inc., USA). All dried starch samples were kept in an airtight container at ambient temperature for further analysis.

2.3. Polarized light microscopy (PLM)

Freeze-dried rice starches (control and HHP treated samples) were dissolved in distilled water to prepare 2% (w/w) starch solution and 0.1 mL of this starch solution was carefully dripped on to a glass slide and covered by a glass lid. After water evaporation, the sample was observed using a Leica DM LP polarizing light microscope (Leica Microsystems GmbH, Germany) with a ×20 magnification.

2.4. Scanning electron microscopy (SEM)

Microstructural observations were performed on a JSM-7401F Scan Electric Microscope (SEM) (JEOL Ltd., Japan) at an acceleration voltage of 10 kV. The samples were dried by a freeze-dryer at 25 °C, 50 Pa for 24 h, then dehydrated stepwise after successive immersions in a graded ethanol series of 70%, 80% and 90% (w/v), 10 min per step. Samples were washed with 100% ethanol for 3 × 10 min, and then further conditioned in a vacuum-assisted desiccator overnight. Thereafter, the dried specimens were attached to the stainless stubs with double sticky tabs, sputtered immediately with gold to approximately 10 nm.

2.5. Atomic force microscopy (AFM)

The starch morphology was characterized using an atomic force microscopy (AFM) according to the modified method of Thys et al. (2008). Aliquots of rice starch suspensions (12.5 mg/mL) were deposited onto a mica plate, dried at room temperature, and adhered to the stainless plate by double-sided tape. The sample surface topography was measured by Multimode Nanoscope AFM (Vecco Metrology Group, Digital Instruments, USA) and triangular silicon cantilever with a nominal spring constant of 48 N/m (NSC11/AIBS, NT-MDT Co., Russia). Imaging was taken in tapping mode with a cantilever resonant frequency around 330 kHz and scan rate of 2.441 Hz. A total sample area of 1 × 1 μm^2 was recorded at ambient conditions (25 °C and 50% relative humidity). Trace and retrace procedures were performed to prove that the samples were not modified during the scanning. All AFM images contained 512 × 512 data points. The raw data images were processed using the Nanoscope 5.30r3sr3 software (Digital Instruments, America).

2.6. X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) was obtained by using a D8 ADVANCE X-ray powder diffractometer (BRUKER-AXS, Germany), with Cu Kα radiation ($\lambda = 1.542A^{\circ}$) at settings of 40 kV and 20 mA. The scattered radiation was detected in the region of 5–50° (2θ), with a step of 0.2° by the scanning speed of 4° (2θ) min^{-1}. The degree of relative crystallinity was calculated following the method of Zhang et al. (2009), considering the 2θ range from 10° to 45°.

2.7. NMR (solid-state ^{13}C CP/MAS NMRs) analysis

High-resolution solid-state 13C cross-polarization/magic angle spinning (CP/MAS) NMR analysis of native and HHP treated starches were carried out at a ^{13}C frequency of 100.6 MHz using a 400 MHz nuclear magnetic resonance spectrometer (AVANCE III 400, Bruker, Germany). Starch samples (200–300 mg) were packed in a 7 mm diameter zirconia rotor and spun at 5–6 kHz at the magic angle (54.7°). A contact time of 1 ms and a remulti-step delay of 2 s were applied in the CP experiments. At least 1000 scans were accumulated for each spectrum.

2.8. In vitro starch digestion assay

Starch (100 mg, dry weight) was mixed with 975 units of α-amylase and 70 units of amyloglucosidase in 20 mL of sodium acetate-acetic acid buffer (pH = 6) and incubated at 37 °C in a