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Physicochemical properties and digestibility of hydrothermally treated waxy rice starch

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ABSTRACT

Waxy rice starch was subjected to annealing (ANN) and heat-moisture treatment (HMT). These starches were also treated by a combination of ANN and HMT. The impact of single and dual modifications (ANN–HMT and HMT–ANN) on the molecular weight (M_w), crystalline structure, thermal properties, and the digestibility were investigated. The relative crystallinity and short-range order on the granule surface increased on ANN, whereas decreased on HMT. All treated starches showed lower M_w than that of the native starch. Gelatinization onset temperature, peak temperature and conclusion temperature increased for both single and dual treatments. Increased slowly digestible starch content was found on HMT and ANN–HMT. However, resistant starch levels decreased in all treated starches as compared with native starch. The results would imply that hydrothermal treatment induced structural changes in waxy rice starch significantly affected its digestibility.

1. Introduction

Starch is the principal carbohydrate in cereal grains and an important source of nourishment for humans. From a nutritional point of view, starch is generally classified as rapidly digestible starch (RDS), slowly digestible starch (SDS) and resistant starch (RS) based on the rate and extent of its digestibility (Englyst, Kingman, & Cummings, 1992). RDS causes an increase in blood glucose levels immediately after ingestion and SDS is digested completely in the small intestine but this process is slow. RS is not digested in the small intestine but fermented in the large bowel into short-chain fatty acids (Cummings, Beatty, Kingman, Bingham, & Englyst, 1996). SDS offers the advantage of a slow increase of postprandial blood glucose level and sustains blood glucose levels over time compared to RDS with its drastic fluctuation. SDS might be helpful in controlling and preventing hyperglycemia related diseases. Consequently, starch ingredients with high levels of SDS and RS can improve the nutritional function of foods.

Hydrothermal treatments, including annealing (ANN) and heat-moisture treatment (HMT), are physical modifications that change the physicochemical properties of starch without destroying its granule structure (Zavareze, Storck, de Castro, Schirmer, & Dias, 2010). Both ANN and HMT are involved in the starch to moisture ratio, with temperature and heating time needed to be controlled. Annealing occurs under an excess of water (>40%) and low temperature (below the gelatinization temperature), while the HMT is carried out under restricted moisture content (10–30%) and higher temperatures (90–120 °C) (Maache-Rezzoug, Zarguili, Loisel, Queveau, & Buléon, 2008).

A substantial amount of studies have been focused on the effect of ANN and HMT on the physicochemical properties and digestibility of various starches (Dias, Da Rosa Zavareze, Spier, de Castro, & Gutkoski, 2010; Jacobs, Eerlingen, Spaepen, Grobet, & Delcour, 1997; Lee, Kim, Choi, & Moon, 2012; Singh, Chang, Lin, Singh, & Singh, 2011; Varatharajan et al., 2011). The above studies have shown that ANN and HMT result in structural changes within the amorphous and crystalline regions to different extents, which in turn influence granular swelling, amylose leaching, pasting properties, gelatinization parameters, molecular structure, crystalline structure, and susceptibility towards enzymes and acids. However, only a few studies have reported the effect of the combination of ANN and HMT on starch structure and properties (Chung, Hoover, & Liu, 2009; Chung, Liu, & Hoover, 2010; Stute, 1992). These studies showed the effect of combinative hydrothermal treatments on the crystalline structure, thermal properties,

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nutritional fractions of various starches and led to either an increase or decrease in SDS and RS content. However, data is still scarce on the effect of dual hydrothermal treatments on starch. Further studies are still essential on the combinative effect of ANN and HMT on starch molecule structure and properties.

Rice starch is used as an additive in various foods, industrial products, desserts, bakery products and as a fat mimetic in foods such as ice cream, yoghurt and salad dressings. Because of its wide-ranging food and industrial applications, waxy rice starch has been extensively studied. To our knowledge, no further report was found using a combination of ANN and HMT treatment on the physicochemical properties of waxy rice starch. Thus, the objective of this study was to investigate to what extent changes to molecular weight, crystalline structure, gelatinization properties and nutritional fractions of waxy rice starch on ANN, HMT, HMT–ANN and ANN–HMT are influenced by amylopectin structure.

2. Materials and methods

2.1. Materials

Waxy rice starch (0% amylose) was obtained from Jiangsu Baobao Group (Nantong, China). Porcine pancreas α-amylase (EC3.2.1.1, 16 U/mg) type-B and amyloglucosidase (EC 3.2.1.3, 300 U/ml) from *Aspergillus niger* were purchased from Sigma–Aldrich Chemical Co. (St. Louis, MO, USA). Megazyme glucose assay kit (GOPOD method) was bought from Megazyme International Ireland Ltd. (Wicklow, Ireland). Other chemicals and solvents were all of analytical grade.

2.2. Hydrothermal treatment

For annealing treatment (ANN), native waxy rice starch (50 g dry basis) slurries (80% moisture) were incubated at 50 °C in a water bath with shaking for 24 h. After the incubation period, samples were centrifuged (6000 rpm) for 10 min and the supernatant was decanted. The annealed starches were washed once with deionized water and air dried at 40 °C overnight. With regard to heat-moisture treatment (HMT), native waxy rice starch (50 g dry basis) samples were weighed and the moisture content was adjusted to 25% by adding appropriate amount of distilled water. Specimens were weighed into polytetrafluoroethylene containers and sealed, then packed into stainless steel reaction still. The containers were kept for 12 h at 4 °C, and then placed in a forced air oven at 110 °C for 8 h. Afterwards the containers were opened, and the starch samples were air-dried to uniform moisture content (about 12%). For dual modification, annealed starch samples were subjected to heat-moisture treatment (ANN–HMT) and heat-moisture treated starch samples were subjected to annealing (HMT–ANN) as referred to by Stute (1992) and Chung et al. (2009). Finally, all starch samples were dried at 40 °C overnight then gently ground by a pestle and mortar to pass through a 100-mesh sieve.

2.3. Swelling power and solubility

Swelling power of starch samples was determined in duplicate by adopting the method of Tester and Morrison (1990). Swelling power is the ratio in weight of the wet sediment to the initial weight of dry starch. The solubility of starch was measured according to the method of Schoch (1964) with modifications. The solubility is the ratio of the dried supernatant weight to the initial weight of dry starch. Experimental data are the means of duplicates.

2.4. ATR-FTIR analysis

ATR-FTIR analysis of starches was obtained with an FT-IR spectrometer (TENSOR27, BRUCK, Germany) equipped with a deuterated triglycine sulphate (DTGS) detector using an attenuated total reflectance (ATR) mode. For each spectrum, 16 scans were recorded at a resolution of 4 cm^{-1} at room temperature. Spectra were baseline-corrected and then deconvoluted over the range of 1200–800 cm^{-1}. A half- width of 22 cm^{-1} and a resolution enhancement factor of 2.2 were used. The amplitudes of absorbance for each spectrum at 1022 and 1047 cm^{-1} were noted and the ratio of amplitudes of absorbance at 1047 cm^{-1} and at 1022 cm^{-1} was calculated per sample to estimate the degree of order of starch at the surface (Sevenou, Hill, Farhat, & Mitchell, 2002).

2.5. X-ray diffraction and relative crystallinity

X-ray diffraction analysis was performed with an X-ray diffractometer (D8 ADVANCE, Bruker, Germany) operated at 40 kV and 40 mA producing Cu Kα radiation of 1.5418 Å wavelength, scanning through the 2θ range from 3° to 35° at a rate of 2°/min. The moisture of a specimen was regulated to about 15% by storage in a sealed desiccator over water at 25 °C. Relative crystallinity was calculated by the ratio of the crystalline area to the total diffractogram area (Nara & Komiya, 1983).

2.6. High-performance size-exclusion chromatography (HPSEC) and multi-angle laser-light scattering (MALLS) with refractive index (RI) detector

Starch sample (12.5 mg) was stirred in 25 ml of dimethyl sulfoxide (DMSO) contain 50 mM LiBr and heated in a boiling water bath for 30 min. After that, the liquid system was stirred for 24 h at room temperature. The solutions were then filtered through a nylon filter (0.22 μm type membrane, Millipore, USA) before injection into the MALLS system (Wyatt Technology, Santa Babara, CA, USA) consisting of a pump (P2000, Spectra System, San Hose, CA, USA), an injector valve with a 1 ml loop, SEC column (P8514-806, Showa Denko, Tokyo, Japan), a MALLS (Dawn DSP-F, Wyatt Technology, Santa Barbara, CA, USA) fitted with an argon laser (488 nm), and an Optilab 903 RI detector (Wyatt Technology, Santa Barbara, CA, USA). The sample (1 ml) was injected into the system and ran at a flow rate of 0.3 ml/min. The mobile phase was DMSO and degassed under vacuum. The column oven temperature was controlled at 40 °C. The molecular weights were calculated using ASTRA 6.1 software program (Wyatt Technology).

2.7. Differential scanning calorimetry

The thermal transitions of starches were investigated with the use of a differential scanning calorimetry (DSC 8000, Perkin Elmer Inc., Norwalk, USA). A starch sample (3 mg) was weighed in a DSC pan and the excess water was added to obtain a starch/water ratio of 3:7. The pans were then sealed, equilibrated for 4 h at room temperature, then heated from 30 to 130 °C at the rate of 10 °C/min. Gelatinization onset temperature (T_o), peak temperature (T_p), conclusion temperature (T_c), gelatinization range (ΔT) and enthalpy values (ΔH) were measured to characterise the thermal properties of starch.

2.8. In vitro digestibility

Starch nutrition fractions were analysed according to the method of (Englyst et al., 1992) with minor modifications. Enzyme solution containing porcine pancreas α-amylase and amyloglucosidase was