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Comparison of the structural characterization and physicochemical properties of starches from seven purple sweet potato varieties cultivated in China

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ABSTRACT

In this study, the structural characterization and physicochemical properties of starches from seven purple sweet potato varieties (Fuzi No. 1, Guang No. 161, Jihei No. 1, Ningzi No. 1, Ningzi No. 2, Xuzi No. 6 and Xuzi No. 8) cultivated in China were compared. Starch granules of purple sweet potatoes all exhibited round, polygonal and hemispherical shapes with granule sizes ranging from 4.3 to 23.6 μm. X-ray powder diffraction patterns and ^{13}C nuclear magnetic resonance spectra revealed purple sweet potato starches were C_A-type with relative crystallinity varying from 34.0% to 37.3%. Small-angle X-ray scattering spectra indicated the lamellar repeat distances of starch granules were in the range of 9.962–10.137 nm. Ratios of 1045/1022 cm^{-1} and 1022/995 cm^{-1} of Fourier transform infrared spectra varied in the range of 0.689–0.887 and 0.850–0.974, respectively. Amylose contents of purple sweet potato starches differed from 18.2 to 27.2%. Purple sweet potato starches exhibited different gelatinization properties but similar thermal stability. Moreover, resistant starch contents varied from 29.25% to 43.50%. Our study indicated the granule size, relative crystallinity, the degree of short-range order, amylose content, gelatinization property and *in vitro* digestibility of purple sweet potato starches were greatly influenced by the variety of purple sweet potato.

1. Introduction

Starch, the major source of carbohydrate in the human diet, is produced by plants for energy storage. Nowadays, starch has been widely used in food, paper, textile, pharmaceutics and materials [1–3]. Starch is a biopolymer consisting of amylose and amylopectin. Amylose is a linear polysaccharide with α (1–4)-linked glucopyranose units, making up to 15–35% in most starch granules; whereas amylopectin is a highly branched polymer with α (1–4)-linked glucopyranose backbones linked by α (1–6)-linked glucopyranose branches [4]. Native starch is highly variable in both structure and function, depending on the origin of starch [5]. Moreover, the physicochemical and functional properties of starch are closely associated with its structural characterization [6,7]. In the past few years, starches from various plant sources, such as wheat, maize, rice and potato have received extensive attention in relation to structural and physicochemical properties [8].

Sweet potato [*Ipomea batatas* (L.) Lam.], a member of *Convolvulaceae* family, is one of the most important root crops worldwide and highly easy to manage and cultivate [6,9]. In recent years, sweet potato has become a research focus due to its unique nutritional and functional properties [10]. Starch is one of the main components in the root of sweet potato, which is widely used as a food ingredient for processing sweet potato products, such as noodles, soup, sauce, snacks and breads [11,12]. Based on the color of peel and flesh, sweet potato can be classified into white, yellow, orange and purple ones [13]. Till now, most studies have focused on starches from white, yellow and orange sweet potatoes [14–18]. Purple sweet potato, a special cultivar of sweet potato, contains abundant anthocyanins and starch [12,19]. However, studies on starch isolated from purple sweet potato are very limited [16,17,20–23]. Existing studies have demonstrated that the structural, physicochemical and functional properties of starch are closely related to the genotype and growing condition of purple sweet potato [16,20,22]. Nonetheless, whether the structure, physicochemical and functional properties of starch are affected by the variety of purple sweet potato is still unknown.

In this study, the structural characterization and physicochemical properties of starches from seven purple sweet potato varieties (Fuzi No. 1, Guang No. 161, Jihei No. 1, Ningzi No. 1, Ningzi No. 2, Xuzi No. 6 and Xuzi No. 8) cultivated in China were compared. The structural

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characterization of starches from purple sweet potatoes was measured by scanning electron microscope (SEM), X-ray powder diffraction (XRD), small-angle X-ray scattering (SAXS), attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy and solid-state ^{13}C cross polarization magic angle spinning nuclear magnetic resonance (^{13}C CP/MAS NMR) spectroscopy. Then, the amylose content, thermal property and *in vitro* digestibility of starches from purple sweet potatoes were determined.

2. Materials and methods

2.1. Materials and reagents

Seven kinds of purple sweet potatoes with varieties of Fuzi No. 1, Guang No. 161, Jihei No. 1, Ningzi No. 1, Ningzi No. 2, Xuzi No. 6 and Xuzi No. 8 were used in this study. These materials were obtained from Xuzhou Institute of Agricultural Sciences in Jiangsu Xuhuai District (Jiangsu, China). Amylose from potato, amylopectin from corn, porcine pancreatic α amylase (EC 3.2.1.1) and amyloglucosidase from *Aspergillus niger* (EC 3.2.1.3) were all purchased from Sigma Chemical Co. (St. Louis, USA). All other reagents were of analytical grade.

2.2. Isolation of purple sweet potato starches

Starches were isolated from seven purple sweet potato varieties according to the method of Wei et al. [24] with some modifications. The fresh root tubers of each purple sweet potato were washed with tap water, peeled, cut into small cubes and milled in a mortar. The ground sample was treated with sodium hydroxide solution (pH 10) for 4 h to remove protein and anthocyanins. Afterwards, the resultant slurry was extruded through four layers of gauze and then successively passed through 100- and 300-mesh sieves. The filtrate was centrifuged at 8500*g* for 15 min. The obtained precipitate was repeated washed with deionized water for 10 times and dehydrated with anhydrous ethanol. Finally, the resultant starch sample was dried at 35 °C for 2 days, ground into powders and passed through a 100-mesh sieve.

2.3. Scanning electron microscopy (SEM)

Morphology of purple sweet potato starch was observed by S-4800 SEM (Hitachi Ltd., Tokyo, Japan). Starch sample was mounted on an aluminum stub and then sputter coated with gold. Micrograph of each sample was taken at an accelerating voltage of 15 kV with 1000× magnification.

2.4. X-ray powder diffractometry (XRD)

Crystalline structure of purple sweet potato starch was analyzed by Bruker AXS D8 Advance X-ray diffractometer (Bruker AXS GmbH, Karlsruhe, Germany). The diffractometer was operated at 40 kV and 40 mA with Ni-filtered Cu Kα radiation. The scanning region was 3–40° (2θ) with a scanning rate of 0.3°/min. The relative crystallinity of each starch was calculated according to the method of Miao et al. [25].

2.5. Small-angle X-ray scattering (SAXS)

The lamellar architecture of purple sweet potato starch was analyzed by SAXS according to the method of Yuryev et al. [26] with some modifications. Starch sample was dispersed in distilled water to form slurries. Then, SAXS pattern was recorded on Bruker NanoStar SAXS system (Bruker AXS GmbH, Karlsruhe, Germany) equipped with Vantec 2000 area detector. The optics and sample chamber were under vacuum to minimize air scattering. The obtained SAXS data were analyzed using Bruker NanoFit software. The relative position of SAXS peak (S_{max}) on the scattering vector axis was used to determine the Bragg spacing (2π / S_{max}) of starch. The intensity of scattering peak (I_{max}) was calculated according to the method of Cai et al. [27].

2.6. Fourier-transform infrared (FT-IR) spectroscopy

Short-range ordered structure of purple sweet potato starch was characterized by Varian 670 FT-IR spectrometer (Varian Inc., Palo Alto, USA) equipped with attenuated total reflectance (ATR) accessory. Spectrum was corrected by a baseline in the range of 1200 to 800 cm^{-1} before deconvolution by using Omnic software of version 8.0 (Thermo Fisher Scientific Inc., Waltham, USA). Then, the band intensity ratios of 1045/1022 cm^{-1} and 1022/995 cm^{-1} were calculated.

2.7. ^{13}C solid-state cross polarization magic angle spinning nuclear magnetic resonance (CP/MAS NMR) spectroscopy

Solid-state ^{13}C NMR spectrum of purple sweet potato starch was recorded on Bruker Avance III 400WB spectrometer (Bruker Biospin GmbH, Rheinstetten, Germany) equipped with a double resonance H/X CP-MAS 4 mm probe. Spectrum was obtained by using standard CP/MAS technique with the spinning rate of 6 kHz and the magic angle of 54.7°. The contact time, acquisition time and recycle delay were set as 1.2 ms, 15.7 ms and 3 s, respectively.

2.8. Determination of amylose content

Amylose content of purple sweet potato starch was determined according to the method of Man et al. [28]. Starch sample (10 g) was dissolved in 5 mL of urea dimethyl sulphoxide (UDMSO) solution (nine parts DMSO and one part of 6 mol/L of urea) at 95 °C for 1 h with stirring. Then, 1 mL of starch-UDMSO solution was mixed with 1 mL of iodine solution (0.2% iodine and 2% potassium iodide, w/v) and 48 mL of deionized water. The mixture was reacted in the dark for 20 min and the absorbance of the reaction mixture was measured at 620 nm. Amylose content was calculated from the standard curve established by amylose from potato and amylopectin from corn.

2.9. Differential scanning calorimetry (DSC)

The gelatinization property of purple sweet potato starch was measured by DSC 8500 (PerkinElmer Ltd., Waltham, USA). Starch sample (5 mg) was mixed with deionized water (15 μL) and sealed in an aluminum pan. The hermetically sealed pan was equilibrated at room temperature for 12 h, and then heated from 30 to 110 °C at a rate of 10 °C/min. Gelatinization parameters including onset temperature (T_o), peak temperature (T_p) and conclusion temperature (T_c), and enthalpy change (ΔH) were recorded.

2.10. Thermogravimetric analysis (TGA)

The thermal stability and decomposition of purple sweet potato starch were measured by Pyris 1 TGA instrument (PerkinElmer Ltd., Waltham, USA). Starch sample (2 mg) was heated from 50 to 800 °C at a heating rate of 10 °C/min under nitrogen flow of 20 mL/min. The characteristic peak of starch degradation was identified through the derivative TGA curve (DTG).

2.11. In vitro digestibility

The digestibility of purple sweet potato starch was carried out following the method of Liu et al. [29] with some modifications. To prepare enzyme solution I, 62.5 mg of porcine pancreatic α amylase (16 U/mg) was dispersed in 10 mL of sodium phosphate buffer (pH 6.0) and centrifuged at 3000*g* for 15 min. To prepare enzyme solution II, 0.05 mL of amyloglucosidase (100,000 U/mL) was dispersed in 50 mL of sodium phosphate buffer (pH 6.0). Afterwards, 20 mg of starch was dissolved