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Comparative susceptibilities to alkali-treatment of A-, B- and C-type starches of *Dioscorea zingiberensis*, *Dioscorea persimilis* and *Dioscorea opposita*

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ABSTRACT

The effect of alkali-treatment for 0, 15, and 30 days at 35 °C on amylose content, morphological properties, crystalline properties, swelling power, solubility, absorbance spectra and *in vitro* digestibility of starches from *Dioscorea zingiberensis*, *Dioscorea persimilis* and *Dioscorea opposita* were investigated. The amylose content of all the three starches decreased after 15 days of alkaline treatment and then increased after 30 days of alkaline treatment. There were similar changes in relative crystallinity for *D. zingiberensis* and *D. persimilis* starches. It was observed that the three starches displayed a reduction trend in swelling power with a significant increase in solubility. Adhesion among some of the starch granules was observed after alkali-treatment for 30 days in *D. zingiberensis* and *D. opposita* starches, while *D. persimilis* starch showed some hollows on the granule surface. The rise in the absorbance ratio of 1047/1035 and 1047/1022 cm^{-1} from FT-IR was observed during alkali-treatment for *D. persimilis* and *D. opposita* starches. Alkali-treatment elevated the *in vitro* digestibility with resistant starch values climbing up from 50.16% to 64.95% and from 66.14% to 70.74% for *D. zingiberensis* and *D. persimilis* starches, respectively, but there was no significant change in resistant starch value for *D. opposita* starch.

1. Introduction

Dioscorea (Family Dioscoreaceae) is a large genus consisting of about 630 species and mainly distributed in the tropical and subtropical regions. Nearly 50 *Dioscorea* varieties are grown in China, especially in the south and southwest of China (Ding & Gilbert, 2000). Most *Dioscorea* varieties were used as traditional Chinese medicine, folk medicine and function food amounting to a considerable economic value. For example, *Dioscorea opposita* Thunb, *Dioscorea fordii* Prain et Burkil, *Dioscorea persimilis* Prain et Burkill and *Dioscorea alata* L. known as Chinese yam have been used as important invigorant and food for many years (Ni & Song, 2002). Total carbohydrates of Chinese yam vary from 10% to 30% in the fresh rhizome of which starch is the most abundant carbohydrate (Xie, Gao, Zhang, & Huang, 2004; Xue, Zhu, & Yao, 2008). Starch, as the main storage reserves carbohydrate in higher plants, plays a vital role in industrial applications and human diet. However, there were not enough investigations and comparisons on the properties of starch in *Rhizome Dioscorea*. Therefore, it is desirable to research on these non-conventional starchy sources in order to find new values for potential applications.

Native starch with its functional limitations such as low values of solubility, shear stress resistance, paste clarity, stability and freeze–thaw stability is not always suitable for various industrial applications. The starch modification is a viable means to overcome one or more limitations of native starch for industrial applications. Among various starch modification methods, alkali treatment by sodium hydroxide possessed increasing application in various food products (Nor Nadiha, Fazilah, Bhat, & Karim, 2010). In addition, alkali treatment caused slow disaggregation of crystalline, while degradation did not occur in the supermolecular structures of starch molecules. However, this reaction process required a moderate temperature under a long-term treatment (Praznik, Buksa, Ziobro, Gambuś, &

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Nowotna, 2012). Although alkali treatment was commonly used in food industry, the effects of alkali on characteristics and susceptibilities of starches with different botanical origins received less attention compared with other modification methods, such as acid and enzyme hydrolysis (Bello-Pérez, Romero-Manilla, & Paredes-López, 2000; Nor Nadiha et al., 2010; Wang & Copeland, 2012). Thus, much work should be done on characterizing the structural changes in alkali-treated starch responsible for the specific physico-chemical properties.

Starch consists of two main components: amylose, an unbranched or lightly branched linear molecule with (1 → 4)-α-D-glucan and amylopectin, a highly branched macromolecule consisting of (1 → 4)-α-D-glucan short chains with non-randomly α-(1 → 6) linkages at intervals of approximately 20 unit. According to X-ray powder diffraction, starch can be classified to A-, B- and C-type crystalline structures (Cheetham & Tao, 1998). In the native granular forms, it is known that the A-type polymorphs of starch is mainly presented in cereal or rhizome, such as corn, wheat, rice and *Dioscorea zingiberensis* (Zhang, Zuo, Wu, Wang, & Gao, 2012). While B-type starch is usually obtained from tubers, such as potato, *fritillaria* and *D. persimilis* (Jiang et al., 2013; Li et al., 2012; Yu & Wang, 2008). The C-type pattern is a mixture of A- and B-type polymorphs, and naturally occurred in seeds or rhizomes, such as *Radix Puerariae*, pea and *D. opposita* (Jiang et al., 2013; Wang, Sharp, & Copeland, 2011; Yu & Wang, 2008). In addition, the V-type conformation, unnaturally crystalline structure, displayed the peaks at about 2θ of 13° and 20° observed by XRD, which described the amylose being complexed with substances such as aliphatic fatty acids, emulsifiers, butanol and iodine (Cheetham & Tao, 1998).

Overall, the effects of alkali treatment on functional properties of starches with different crystal types most require investigation to elucidate the action of alkali on starch structure and function. In previous study, *D. zingiberensis*, *D. persimilis* and *D. opposita* starches of the same genus presented A-, B- and C-type patterns, respectively (Jiang et al., 2013; Zhang et al., 2012). The objective was to examine the sus ceptibilities to alkali-treatment of starch with different crystal types, physicochemical properties and *in vitro* digestibility after treatment, which would be valuable for edible nutrition and food applications.

2. Materials and methods

2.1. Materials

D. zingiberensis C. H. Wright (Chinese name: Huangjiang), *D. persimilis* Prain et Burkill. (Chinese name: Huaishanyao) and *D. opposita* Thunb. (Chinese name: Mashanyao) were collected from Yulin City of Guangxi Province, Li County of Hebei Province and Guangyuan City of Sichuan Province. Porcine pancreatic α-amylase (No. 3176, 1 MU/g) and amyloglucosidase (No. 10115, 70 U/mg) were purchased from the Sigma–Aldrich. Dimethylsulfoxide (DMSO) was purchased from Tianjin Jiangtian Chemical Technology Co., Ltd. (Tianjin, China).

2.2. Starch isolation

The starch isolation process from the fresh rhizome was carried out as our previous research (Jiang et al., 2013). The fresh rhizomes were washed, peeled, and cut into slices, and then the slices were ground in a laboratory blender. The homogenate was washed through a 160 mesh sifter with distilled water. After depositing, the supernatant was removed, and the settled starch layer was resuspended in distilled water. After precipitating and resuspending seven or eight times, the slurry containing starch was then centrifuged at 3000 g for 10 min. The upper nonwhite layer was discarded. The white layer was resuspended in distilled water and recentrifuged 2–3 times. The starch layer was collected and then dried at 40 °C. Finally, the dried starch was carefully ground to pass through a 160 mesh sifter, and stored at room temperature in a glass container.

2.3. Preparation of alkali-treated starch

The alkali-treated starches were prepared according to the method reported earlier (Nor Nadiha et al., 2010; Ragheb, Abd El-Thalouth, & Tawfik, 1995; Wang & Copeland, 2012) with some modifications. Aqueous NaOH solution 0.1% (w/v), containing sodium azide (0.1%, w/v) as a chemical preservative, was prepared. Duplicate samples were prepared in the alkaline solution (10%, w/v) and left at the temperature of 35 °C for 0, 15, and 30 days with intermittent shaking by hand twice a day. Each sample was shaken for about 30 s every time to fully resuspend the slurry. After treatment, the undissolved residues were subsequently washed twice with 95% ethanol and distilled water to remove any ions. The alkali-treated starches obtained were dried in an oven at 40 °C for 48 h. The recovery of starch (%, w/w) was then calculated based on starch weight before and after treatment. Starch recovery, relative to untreated starch, decreased after the treatment, After 30 days of alkali treatment, the recovery of starch were 81.97%, 71.24%, and 93.08% for *D. zingiberensis*, *D. persimilis* and *D. opposita* starches, respectively.

Moisture contents of native and alkali-treated starches were determined by drying triplicates of 5 g samples to a constant weight in an air oven at 105 °C.

2.4. Apparent amylose determination

Apparent amylose contents of native and alkali-treated starches were determined by the iodine binding colorimetric method. Starch (10 mg, dry basis) and 2 mL of DMSO were mixed and heated at 85 °C for 15 min. Deionized water was added to the dissolved starch to bring the volume up to 25 mL. Starch solution (1 mL) was pipetted into a 50 mL volumetric flask and made up to volume. Iodine (5 mL) was added. Absorbance was read at 600 nm using a UV–visible spectrophotometer (Nor Nadihaa et al., 2010). Analyses were performed in triplicate.

2.5. Swelling power and solubility

Starch sample (200 mg, dry basis) was weighed accurately in a centrifuge tube with 10 mL of distilled water added. The slurry was heated at 55–95 °C in a water bath for 1 h. After that it was cooled to room temperature and centrifuged at 3000 g for 15 min. The supernatant obtained was carefully removed, and the swollen starch sediment was weighed. The aliquot of supernatant was evaporated overnight (110 °C). Analyses were performed in triplicate. Swelling power and solubility were calculated as follows:

$$\text{Swelling power}(\text{g/g}) = (\text{SW} \times 100)/\text{Starch}_{\text{dwb}} \times (100\% - \%\text{SOL}) \quad (1)$$

$$\text{Solubility}(\%) = \text{Weight of dried supernatant} \times 100/\text{Starch}_{\text{dwb}} \quad (2)$$

SW is the weight of wet sediment,
$\text{Starch}_{\text{dwb}}$ is the dry water basis starch weight.

2.6. Scanning electron microscopy

The morphological features of starches were observed with a scanning electron microscope (ESEM Philips XL-3). The dried samples were mounted on a metal stub and coated with gold powder to make the sample conductive, and the images were taken at an accelerating potential of 20 kV.