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Physicochemical Property Changes of Sweet Potato Starch by Ultra Fine Pulverization

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Abstract

This study was performed to analyze the effects of ultra fine pulverization (UFP) on the physicochemical properties of sweet potato starch (SPS). The average diameter and specific surface area of the SPS was decreased from 22.94 to 10.25 μm and from 0.879 to 1.909 m^2/g throughout UFP, respectively, and the damaged starch content was increased from 13.7 to 99.2%. The pulverized sweet potato starch (PSPS) had higher swelling power, solubility, and transmittance values than the SPS. X-ray diffractograms revealed that the SPS had a C-type pattern, which disappeared in PSPS. The rapid visco analysis (RVA) characteristics, peak viscosity, break down, and set back of SPS ceased to exist in PSPS. According to differential scanning calorimetry (DSC) curves, the peak temperature (T_p) and gelatinization enthalpy (ΔE) of SPS were 71.95°C and 10.40 J/g, respectively, while these remained undetected in PSPS. The enzymatic digestibilities of SPS and PSPS were 61.7 and 84.7%, respectively.

Key words: sweet potato starch, ultra fine pulverization, high impact planetary mill, physicochemical property

Introduction

Starches are obtained from grains such as corn, wheat, and rice as well as from tubers and roots, sweet potatoes, potatoes and cassava (Zaidul et al., 2007). Starches consist of four types of supramolecular structures differing in macromolecular organization and characteristic sizes. They include: crystalline (~4-6 nm) and amorphous lamellae (~2.2 nm), amylopectin clusters (~9 nm), semi-crystalline, and amorphous growth rings (~120-400 nm), as well as granules themselves (~0.5-100 μm) (Manners, 1989; Weigh et al., 2000). The different levels of supramolecular structuring guarantee the biological functioning of the molecular entity (Noda et al., 2009). Sweet potato starch exhibits wide variations in granule size (3-40 μm) and amylose content (15-30%), and has a gelatinization temperature between 61 and 70°C (Noda et al., 1997) based on planting and harvesting dates. It also gives both A- and C-type X-ray diffraction patterns (Moorthy, 1994).

The relative high molecular weight and extensive network mainly formed by the hydrogen bonds of starch lead to a high gelatinization temperature, lower fluidity, and chemical reactivity (Zu et al., 2007). For specific purposes, the market sometimes prefers starch with less extensive crystalline regions, thereby improving its physicochemical properties and reactivity for planned applications. Consequently, there is a great interest in methods for modifying the structures of crystalline regions (Fiedorowicz et al., 2001; Liang et al., 2004). Mechanical treatment, depending on the treatment type and its severity (Becker et al., 2001; Dhital et al., 2010), is known to alter starch granule structure, impacting its physicochemical properties and digestibility. Ultra-fine pulverization techniques can be used to modify the crystalline structure by friction, collision, impingement, shear, or other mechanical actions, which ultimately altering physicochemical properties of starches. Mechanical damage to starch granules caused by ultra-fine pulverization techniques induces a progressive loss of crystalline order, and the conversion of large-ordered regions into essentially disordered amorphous materials that are freely accessible to external agents, including such solvents such as water and amylolytic enzymes (Morrison & Tester, 1994a; 1994b).

The goals of this study were to investigate the physicochemical property changes of sweet potato starch, in which particle structures were broken by an ultra fine pulverization technique using a high impact planetary mill.

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Materials and Methods

Sample preparation

The sweet potato starch (SPS) was purchased from Konia Food Co., (Seoul, Korea) and moisture content was 11.2% (dry weight basis). The approimate composition of the SPS was as follows: 0.1% protein, and 0.2% ash based on wet weight basis. A high impact planetary mill (Pulverisette 6, Fritsch Co., Idar-Oberstein, Germany) was used for the ultra fine pulverization of SPS. Ten grams of SPS sample were put into a jar with 250 g each of 11- and 5-mm ziloconium oxide beads.

Scanning electron micrograph (SEM)

The samples were coated with gold-palladium using SEM ion sputter coater (E1030, Hitachi, Tokyo, Japan). The sizes and shapes of the coated samples were then viewed under SEM (S4300, Hitachi, Tokyo, Japan) at 15 kV.

Particle size analysis

Particle size distributions of the samples were determined using a particle size analyzer (Compagnie Industrielle Des Lasers, CILAS 1064, Orleans, France). The circulating liquid was sonicated with ultrasound (20 kHz) for 30 sec to break the remaining agglomerates before recording the observations (Becker et al., 2001).

Damaged starch

Damaged starch was determined using the AACC method 76-30A (AACC, 1992).

Swelling power, solubility, and transmittance

Aqueous suspensions of 1.7% (w/v) sample were heated in a water bath at constant temperatures (40, 50, 60, 70, 80 and 90°C) and shaking for 30 min. Each suspension was cooled and centrifuged at 3000 ×g for 5 min. The supernatant was set aside in an aluminum tray and dried at 105°C for 24 hr. The precipitate was directly weighed. The obtained data were used to calculate the swelling power and the solubility of the samples (Giovanna et al., 2009). For transmittance (Perera & Hoover, 1999), aqueous suspension (1%, w/v) of the sample was placed in a water bath at constant temperatures (40, 50, 60, 70, 80 and 90°C) and constant shaking for 1 hr. The samples were placed at room temperature (25°C) for 30 min and then absorbance was measured at 625 nm by a spectrophotometer (UV 1201, Shimazu, Kyoto, Japan).

X-ray diffractometry

The samples were analyzed using an X-ray diffractometer (XD-D1, Shimadzu, Kyoto, Japan) under the following conditions; 30 kV, 30 mA, the θ-2θ method using a Cu tube, and 4.0 deg/min.

Thermal analysis

Thermal analyses of samples were performed using a differential scanning calorimeter (DSC 2010, TA instrument, Newcastle, USA). The sample (3 mg, dry weight basis) was weighed and then put into an aluminum hermetic pan (40 μL), after which of deionized water (8 μL) was directly added to the pan. The pan was sealed hermetically and conditioned 24 hr at room temperature (25°C) for the equilibrium of the sample. The pan was scanned from 30 to 120°C at a heating rate of 10°C/min. An empty pan was used as a reference. Gelatinization onset (T_o) and peak temperatures (T_p), and gelatinization enthalpy (ΔE) were recorded.

Pasting property

A rapid visco analyzer (RVA-3D, Newport Scientific Ltd., Warriewood, Australia) was employed to determine the pasting properties of the samples. The sample (3.0 g, dry weight basis) and 25 mL of distilled water were combined and stirred in the aluminum RVA sample canister. A programmed heating and cooling cycle was used, where the sample was held at 50°C for 1 min, heated to 95°C in 3 min 30 sec, held at 95°C for 2 min 30 sec, and cooled to 50°C in 3 min 50 sec. Parameters such as peak viscosity, hold viscosity, final viscosity, peak time, and pasting temperature were determined.

Digestibility with α-amylase

Enzymatic digestibility with α-amylase was performed according to the method described by Liu et al. (1999). Approximately 1 g of sample was added to 30 mL of phosphate buffer (0.2 mol, pH 6.9) in a test tube and allowed to stand for 30 min in a 95°C water bath. After cooling at 25°C, α-amylase (320 unit, Sigma, St. Louis, MO., USA) was added and incubated with shaking at 30°C for up to 14 hr. After digestion, the undigested sample was removed by centrifugation, and analyzed by gravimetric methods.

Statistical analysis

All samples were analyzed at least in three times and the mean values and standard deviations were reported. Data were analyzed using the Statistical Analysis System Software (SAS

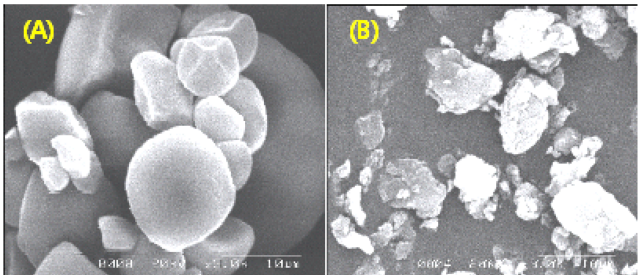

Fig. 1. Scanning electron microscope (×3,000) of sweet potato starch (A) and pulverized sweet potato starch (B).

8.2; SAS Institute Inc., Cary, NC, USA).

Results and Discussion

Microstructure

The scanning electron micrographs (SEM) of the sweet potato starch (SPS) granules before and after ultra fine pulverization (UFP) using a high impact planetary mill are shown in Fig. 1. The operation conditions were 300 rpm for 6 hr. The high impact planetary mill consisted of rotating grinding bowls mounted eccentrically on a rotating support disc. The balls would strike the inner wall of the bowl vertically (impact energy), approach each other tangentially (friction), or just roll down the inner wall of the bowl (centrifugal mills). The UFP of SPS particles resulted in more irregular forms in terms of morphological appearance with reduced sizes as a whole. SEM revealed the existence of minute cracks covering their surfaces and the gathering of small particles around large size starch particles, possibly due to the electrostatic attraction between particles. Accordingly, agglomeration via interactions between amorphous regions of particles of different sizes sometimes results in increased mean diameters, although each individual particle becomes smaller (Dhital et al., 2010). This particle agglomeration is due to hydrogen bonding between hydroxyl groups that are formed due to the breakage of glycosidic bonds during starch pulverization (Chen et al., 2003).

The average diameter and specific surface area of the particles changed from 22.74 to 10.25 μm and from 0.879 to 1.909 m^2/g throughout UFP, respectively, (Table 1).

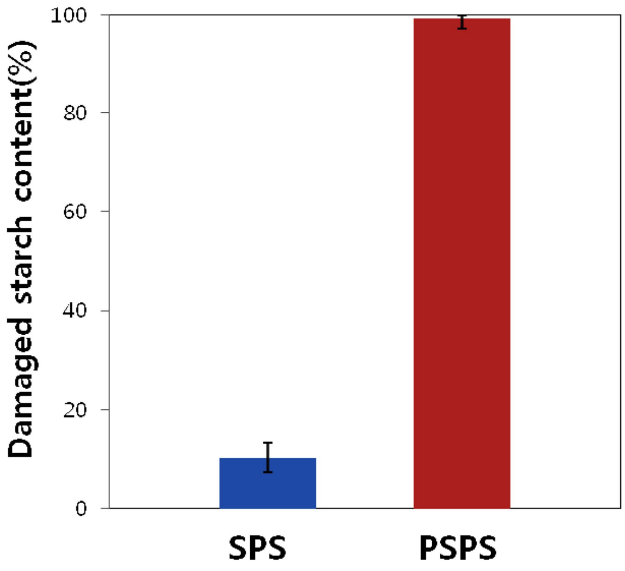

Fig. 2. Damaged starch content of sweet potato starch (SPS) and pulverized sweet potato starch (PSPS).

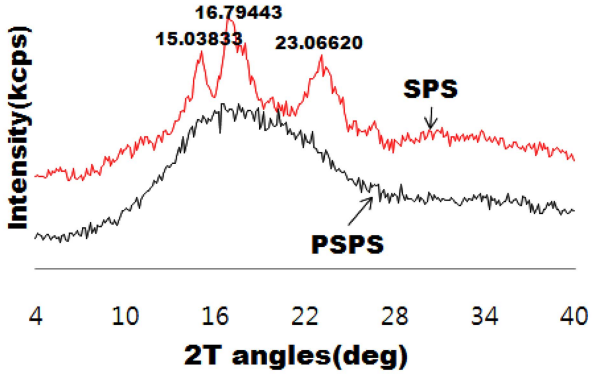

Fig. 3. X-ray diffractograms of sweet potato starch (SPS) and pulverized sweet potato starch (PSPS).

Damaged starch

UFP caused a significant increase in damaged starch content, from 10.25 to 99.22% (Fig. 2). In UFP starch is subjected to various forces such as compression, impact, shear, and attribution, which likely caused physical breakdown of the SPS (Morrison & Tester, 1994a; 1994b). Crystalline materials are generally more brittle than non-crystalline materials, so greater damage in high amylopectin starches may be due to

Table 1. Particle size distribution, mean diameter, and specific surface area of sweet potato starch (SPS) and pulverized sweet potato starch (PSPS)

	Mean diameter at 10% (μm)	Mean diameter at 50% (μm)	Mean diameter at 90% (μm)	Mean diameter (μm)	Specific surface area (m^2/g)
SPS	5.80±1.97	22.14±2.47	38.94±3.78	22.74±4.01	0.879±0.02
PSPS	1.36±0.45	9.46±1.95	20.43±3.34	10.25±2.32	1.909±0.04

greater amounts of crystalline double helices (Htoon et al., 2009).

X-ray diffractograms

As shown in Fig. 3, the SPS spectrum showed definite diffraction peaks that presumably reflect crystalline regions in the starch. According to the characteristic diffraction peaks at 15.0, 16.7 and 23.0, the SPS structure is a C-type pattern. This type of crystallinity is most susceptible to enzymatic hydrolysis (Martinez et al., 2007). The peak diffraction of PSPS disappeared completely and showed that it had been largely converted to a non crystalline state. Consequently, the diffraction spectrum shows a broad featureless peak that is a typical spectrum of amorphism.

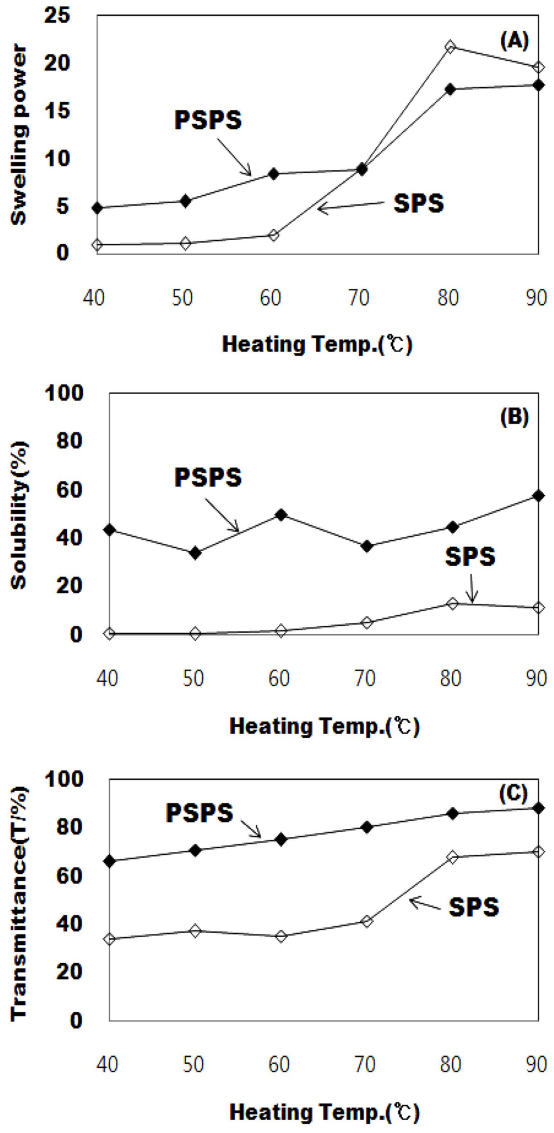

Fig. 4. Physical properties of sweet potato starch (SPS) and pulverized sweet potato starch (PSPS). (A) Swelling power, (B) Solubility, (C) Transmittance.

Physical property

The swelling power, solubility, and transmittance of SPS and PSPS are presented in Fig. 4. PSPS had higher swelling power, solubility, and transmittance values than SPS throughout a temperature range of 40°C to 90°C. Starch such as amylose in a reduced proportion shows low solubility when heated in excess water (Singh et al., 2003). Intact starch granules are insoluble in water. Water soluble fractions obtained after UFP are shown to be low molecular weight fragments produced; water molecules are bonded to the free hydroxyl groups of amylose and amylopectin by hydrogen bonds, which produce an increase in solubility (Dhital et al., 2010).

Pasting property

The RVA characteristics of SPS and PSPS were given in Fig. 5. The peak viscosity of SPS decreased from 661.75 to 24.50 RVU after UFP. The higher peak viscosity of SPS than PSPS may be due to the presence of granules with a wide size distribution range, leading to different swelling patterns in SPS (Lovedeep et al., 2007). The break down value of SPS, the difference in peak and final viscosities, decreased from 411.75 to 3.67 RVU after UFP. During break down, swollen granules are disrupted and amylose molecules generally leach out into the solution. The set back value of SPS also decreased from 63.59 to 30.75 RVU after UFP. Set back is an increase in viscosity resulting from the reassociation among leached amylose molecules during cooling. Other pasting properties also significantly changed and presented a linear pattern. This is a result of SPS granule breakdown and/or rupture by UFP treatment.

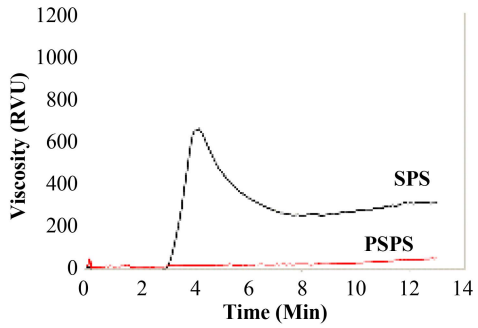

Fig. 5. RVA profile changes of sweet potato starch (SPS) and pulverized sweet potato starch (PSPS).

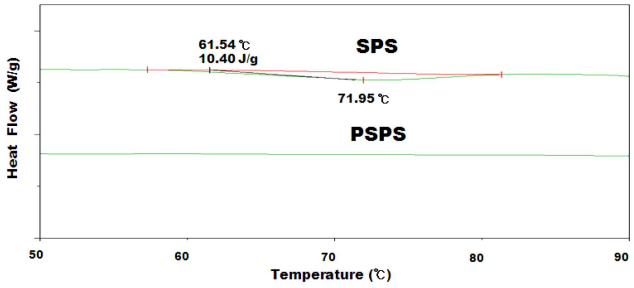

Fig. 6. Differential scanning calorimeter thermograms of sweet potato starch (SPS) and pulverized sweet potato starch (PSPS).

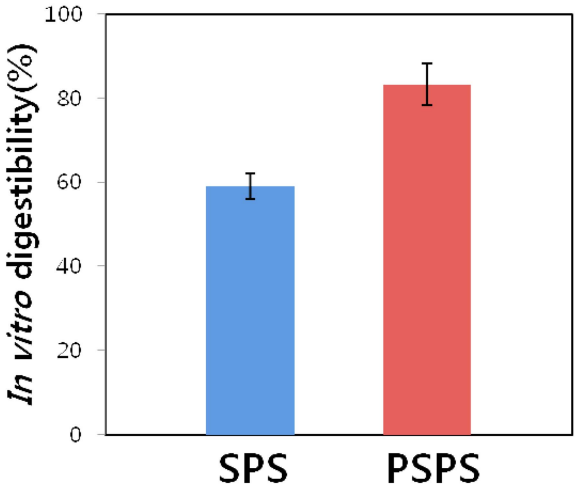

Fig. 7. *In vitro* digestibility of sweet potato starch (SPS) and pulverized sweet potato starch (PSPS).

Thermal analysis

The DSC curves of SPS and PSPS were shown in Fig. 6. The peak temperature (T_p) and gelatinization enthalpy (ΔE) of SPS were 71.95°C and 10.40 J/g, respectively, while these were not detected for PSPS. The SPS crystalline structure was damaged by UFP. The onset temperature (T_o), T_p, and ΔE ranges for SPS were 55.8-73.1°C, 61.3-76.0°C, and 13.7-16.3 J/g, respectively (Noda et al., 1997).

Digestibility with α-amylase

The *in vitro* starch digestibility by α-amylase of SPS and PSPS was shown in Fig. 7. Starch digestibility differences can be attributed to the interplay of many factors such as starch source, granule size, amylose/amylopectin ratio, the extent of molecular association between starch components, degree of crystallinity, and amylose chain length (Tester et al., 2004). SPS (59.0%) had a lower digestibility value than PSPS (83.2%). After UFP of SPS, most of the crystalline state is changed to a non-crystalline state, with a reduction in mean granule particle size and an increase in granule specific surface area, which makes it susceptible to enzymatic hydrolysis (Lovedeep et al., 2007). Zang and Oates (1999) suggested that extensive surface erosion indicated a high degree of hydrolysis by α-amylase, whereas less surface erosion indicated less degradation.

Acknowledgement

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