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Effect of dry heat treatment on the physicochemical properties and structure of proso millet flour and starch

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ABSTRACT

Proso millet (*Panicum miliaceum* L.) flour and starch were heated in a dry state at 130 °C for 2 or 4 h. The effects of dry heat treatment (DHT) on the pasting, morphological and structural properties of the samples were evaluated. Dry heat treatment had a more significant effect on the pasting viscosity of flour than starch; it increased the pasting viscosity of the flour while it only increased the final viscosity of the starch. After dry heating, the onset of gelatinization and the peak temperatures of the samples increased significantly while the endothermic enthalpy decreased. Scanning electron microscopy showed that the gel structure of the samples became more compact and the particles were plumper when compared with the native ones. Crystallinity of the samples decreased while the X-ray diffraction patterns remained the same after DHT.

1. Introduction

Millets, such as proso millet (*Panicum miliaceum* L.), have excellent nutritional properties and can become a basic resource for crop breeding programs and food diversification (Young-Il et al., 2010). Proso millet, which is also called millet, hog millet, and yellow hog, can be used in many different fields. But now millet is desirable for human food because it is easily digestible and is gluten-free. It can be ground into flour and used to bake flatbreads, tabbouleh or to brew beer (Badau, NkaMa, & Jideani, 2005). The proso millet is rich in protein, mineral substances, and vitamins, and its nutritive parameters are comparable or better than common cereals. Moreover, the quantities of nutrients in the millet are very similar to the recommended ratio of protein, saccharide, and lipids (Kalinova & Moudry, 2006).

Starch, in its native form, has a relatively limited usage in many industries. Physical and chemical modifications are commonly used to produce modified starches with special properties. Although chemically modified starches are available for industrial purposes, most industries (especially the food and pharmaceutical industries) prefer starches without chemical modification. Therefore, physically modified starch, by use of moisture, heat, shearing, or radiation, has gained a wider acceptance for no chemical reagents using (Zavareze, Storck, de Castro, Schirmera, & Dias, 2010). Depending on the presence of moisture, heat treatment can change the granular and molecular structure of the starch (Chung, Min, Kim, & Lim, 2007). Heating starches under dry conditions is a method for producing modified starches. Dry heating treatment (DHT) is a physical modification that changes the physicochemical properties of starch, without destroying its granule structure. Compared with chemical methods, dry heat is a simple, safe, and healthy method. Chiu, Schiermeyer, Thomas, and Shah (1998) introduced a dry heating process for formulating physically modified starches. They reported that thermally treated starches yield a functionality equivalent to that obtained by chemical cross-linking (Chiu et al., 1999; Li et al., 2013). Besides, Sun, Si, Xiong, and Chu (2013) found that the gel structure of the potato starch got compacter after drying-heating with CMC (130 °C, 2 h or 4 h). Heat treatment obviously improved the functional properties of starches.

To sum up, the effect of dry heat treatment or dry heating with ionic gums (Li et al., 2013; Sun et al., 2013) on pasting and thermal properties of starch have been studied, but the changes in the structural and morphological properties of starch and the comparison between flours and starches before and after dry heating have received little attention. To clarify the effects of DHT on the millet flour or starch, and compare the difference between the flours and the starches containing other constituents through dry heating, more studies are needed.

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In this study, the proso millet flour and proso millet starch (8%, w/w, moisture content) were heated in a dry state at 130 °C for 2 h or 4 h. The effects of the DHT on pasting, morphological and structural properties of the proso millet flour and starch were examined.

2. Materials and methods

2.1. Materials

Proso millet grains (Millet Lu 1) were obtained from Qingdao Academy of Agricultural Sciences, Qingdao, China. All analytical grade chemicals were used as obtained without further purification.

2.2. Proso millet flour and starch preparations

The grains were ground into flour using cryogenic milling as described by Hasjima, Li, and Dhitala (2013). The proso millets were steeped in two times distilled water at 4 °C for 2 h, then the water was drained off and the millets were ground with a blender. The milled millet flour was dried in an oven at 40 °C for 48 h, and then passed through a 100-mesh screen. The proso millet starch was prepared using the alkaline steeping method (Ju, Hettiarachchy, & Rath, 2001; Santhanee et al., 2013) with some modifications. Proso millet grains were steeped in 0.3% sodium hydroxide solution and kept at 4 °C for 24 h. The supernatant was discarded and the steeped millets were ground with a blender, then passed through a 100 mesh screen. The slurry was centrifuged at 3000 rpm for 15 min. Then, the supernatant was decanted and the precipitate was re-slurried with water and centrifuged. The step of washing with water was repeated three times. Next, the starch cake was re-suspended in water, neutralized with 1 N hydrochloric acid to pH 7. The supernatant was decanted and the neutralized starch was removed and dried in an oven at 40 °C for 48 h. Standard AOAC methods (1990) were used for the measurement of nitrogen, ash, and lipid. Protein was determined from estimation of total nitrogen using a conversion factor of 6.25. The measurement of the chemical composition (%, dry basis) of the flour and starch was determined according to the method of Puncha-arnon and Uttapap (2013). Protein, lipid, ash, carbohydrate and amylose content in millet flour were 11.43%, 0.91%, 1.3%, 84.82% and 1.5% (dwb, %), whereas those in starch were 0.48%, 0.01%, 0.20%, 97.69% and 1.61% (dwb, %), respectively.

2.3. Modifications using dry heat treatment

The flours and starches were modified by DHT according to Lim, BeMiller, and Lim (2003). The millet flour and starch samples were kept in Petri dish and dried at 40 °C in an air dry oven until the moisture content reached to 8%. Then, all the samples were heated in an electric oven at 130 °C for 2 h or 4 h, respectively. The oven used was 876A-2 digital vacuum oven, Shanghai, China. After DHT, the flours and starches were covered with plastic wraps to prevent them from being affected by damp conditions. Untreated millet starch and flour were used as controls. The measurement of the chemical composition of the DHT samples was according to the same method above. Protein, lipid, ash, carbohydrate and amylose content in DHT2F were 11.44%, 0.9%, 1.31%, 84.83% and 1.5% (dwb, %), in DHT4F were 11.43%, 0.89%, 1.32%, 84.82% and 1.51% (dwb, %), whereas those in DHT2S and DHT4S were the same at 0.48%, 0.01%, 0.20%, 97.7% and 1.61% (dwb, %), respectively. The moisture content of the DHT2 samples and DHT4 samples were 5% and 3%, respectively.

2.4. Paste viscosity

The flour and starch (3.0 g, 14 g/100 g moisture basis) were weighed directly in the RVA canister and distilled water was added to obtain a sample weight of 28.0 g. The slurry was then manually homogenized using the plastic paddle to avoid lump formation before the RVA run. The slurry was heated from 50 °C to 95 °C at a rate of 12 °C/min and maintained at 95 °C for 2.5 min. It was then cooled to 50 °C at the same rate, and held at 50 °C for 2 min. Parameters including peak viscosity (PV), viscosity at the end of 2.5 min at 95 °C or trough viscosity (TV), final viscosity (FV) at the end of cooling, breakdown (BD = PV – TV), setback (SB = FV – TV), and pasting temperature were recorded. All tests were replicated three times.

2.5. Differential scanning calorimeter (DSC)

The thermal properties of samples produced via the heat treatment described above were investigated using a differential scanning calorimeter (DSC 1, Mettler-Toledo, Schwerzenbach, Switzerland) as described by Chanvrier et al. (2007) with minor modifications. Indium was used as the calibration standard. Each product sample (4 mg) and distilled water (8 mg) were placed in a stainless-steel pan, then the container hermetically sealed and then kept at 4 °C for 24 h. Samples were heated at 10 °C/min from 25 °C to 115 °C to observe for the presence of any residual enthalpy gelatinization peak. The endothermic enthalpy (ΔH), onset temperature (T_o), peak temperature (T_p), and conclusion temperature (T_c) were determined.

2.6. Scanning Electron Microscopy (SEM)

The pastes of the millet flours and starches before and after DHT after gelatinization with RVA should be quickly freeze dried. The microstructure of samples after freeze drying for 48 h was observed by scanning electron microscopy (SEM). In addition, the morphology of the flour and starch samples before and after DHT was observed by SEM using the method described by Kim et al. (2008). A dry flour sample was placed on a double-sided Scotch tape, mounted on an aluminum specimen holder, and coated with a thin film of gola under vacuum. Samples were observed under a Jeol scanning electron microscope (JSM 840, Jeol, Japan), and the accelerating voltage was 2 kV.

2.7. X-ray diffraction pattern

The crystal structure of the flour and starch samples was studied with an X-ray diffractometer (Bruker AXS Model D8 Discover) using the conditions described by Watcharatewinkula, Puttanlekb, Rungsardthongc, and Uttapap (2009). Both native and DHT samples were equilibrated in a saturated relative humidity chamber for 24 h at room temperature. The X-ray diffraction was performed on an X-ray diffractometer with copper Ka radiation. Signals of the reflection angle of 2θ, from 4 °C to 45 °C, were recorded. The overall degree of crystallinity was quantified as the ratio of the area of crystalline reflection to the overall diffraction area (Yu, Ma, Menager, & Sun, 2012).

2.8. Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectra of native and DHT samples were recorded on a FTIR spectrophotometer (NEXUS-870, Thermo Nicolet Corporation) as described by Kunal et al. (2008). All the samples (8%, w/w, moisture content) were mixed with KBr and pressed into pellets. The pellets were then subjected to Attenuated Total Reflectance (ATR) spectroscopy in the range of 4000–400 cm^{-1}. Intensity