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Relationships between physicochemical, thermal, rheological and *in vitro* digestibility properties of starches from pearl millet cultivars

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ABSTRACT

Pearl millet starches from different cultivars were studied for physicochemical, thermal, rheological, morphological and *in vitro* starch digestibility properties and correlations among these properties were calculated. The starches had low protein, fat, and ash contents. Amylose content, transition temperature, ΔH_{gel}, PV and PT of starches ranged from 13.6 to 18.1 g/100 g, 63.4–76.3 °C, 10.6–12.4 J/g, 4647–8303 mPa·s and 72.4–73.9 °C, respectively. Steady and dynamic shear properties were studied using rheometer. Plots of shear stress (σ) versus shear rate for starch pastes were plotted and fitted to Herschel-Bulkley model. Yield stress (σ_o), flow behaviour index (n) and consistency index (K) ranged from 25.6 to 183.4 Pa, 1.69–54.5, and 0.29–0.81, respectively. Dynamic rheological properties revealed that starch suspensions had elastic behaviour. Frequency sweep measurement of starch pastes revealed higher magnitude of G′ as compared to G″ with increase in ω, indicating visco-elastic behaviour. The micrographs of starches show granules size and shape from small to large, spherical and polygonal. Digestibility study revealed that resistant starch (RS) fraction varied from 9.7 to 16.5 g/100 g, with cv.GHB-732 having higher RS. Principal component analysis (PCA) studies revealed that two components represent 76.6 g/100 g of total variability.

1. Introduction

Pearl millet (*Pennisetum glaucum* L.), one of the important type of millets, belongs to the family gramineae. It is a drought tolerant crop grown primarily as food grain and fodder in India and Africa (Freeman & Bocan, 1973). India is the largest producer (1,14,20,000 tonnes) of millets in the world followed by Niger (33,21,753 tonnes) (FAO, 2016). Sade (2009) reported 14.0, 5.7, 2.1, 2.0 and 76.3 g/100 g of crude protein, fat, ash, crude fibre and carbohydrate content, respectively in pearl millet. Suma and Urooj (2015) reported starch content in pearl millet in the range from 62.8 to 70.5 g/100 g among different Indian genotypes.

Starch is the major storage polysaccharide of higher plants and it is deposited in partially crystalline granules varying in morphology and molecular structure between and within plant species (Blazek & Copeland, 2008). Starch is of commercial importance because of its numerous functional properties, particularly due to its ability to modify the texture of a product (Kaur, Sandhu, & Lim, 2010). There is growing interest in starch digestion kinetic due to the increase in diseases such as diabetes and consumers' awareness of the relationship between food, nutrition and health (Naidoo, Amonsou, & Oyeyinka, 2015). The dynamic rheological tests for small-deformation oscillatory measurements generate valuable information on visco-elastic properties of starch pastes without breaking their structural elements. These tests are important for studying the molecular structure of starches (Gunasekaran & Mehmet, 2000). Chakraborty, Tiwari, Mishra, and Singh (2015) optimized ingredients for bread made from wheat flour and millets and studied the rheological properties of dough under thermo-mechanical stress.

Due to decreased water supplies, decreased area under maize cultivation, adverse salinity, low cost of growing pearl millet crop, its multipurpose applications in food and feed, starch from pearl millet needs to be explored more so that it can become better substitute than other conventional starch sources. Many researchers have studied the properties of starches from conventional sources including wheat starch (Li et al., 2016; Rosicka-Kaczmarek et al., 2016; Zhang et al., 2016), rice starch (Ashogbon & Akintayo, 2012; Jang et al., 2016; Lawal et al., 2011) and maize starch (Sandhu & Singh, 2007; Sandhu, Singh, & Kaur, 2004). As

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pearl millet is largely underutilized, therefore limited work has been done on various properties of pearl millet starch as compared to starch from conventional sources such as maize, rice and potato. Previously, some workers have characterized pearl millet starch for different properties (Choi, Kim, & Shin, 2004; Suma & Urooj, 2015; Wu, Lin, Cui, & Xiao, 2014). Zhu (2014) reported structural, physicochemical and use of millet starches. However, there is dearth of information on relationship between physicochemical, rheological and digestibility properties of pearl millet starches. Keeping this in view, the present study was conducted to establish relationships among different functional properties of starch from pearl millet cultivars. The relationships established will help in further exploration and starch utilization from pearl millet.

2. Materials and methods

2.1. Materials

Pearl millet cultivars (cv.) (HC-10, HHB-67, HHB-223, HHB- 226 and W-445) of 2013 harvest were procured from Chaudhary Charan Singh Haryana Agriculture University (CCSHAU), Hissar, India. The cultivars procured from CCSHAU were grown under similar conditions. One pearl millet cultivar, cv.GHB-732 was procured from Pearl Millet research station, Jamnagar, Gujarat, India from 2013 harvest. All the cultivars were grown in rainy season (June–September) and their maturity duration was 75–85 days. The yields of the cultivars ranged between 2900 and 4045 kg/ha. These cultivars were the results of breeding programmes for pearl millet improvement. 1000 kernel weight of HC-10, HHB-67, HHB-223, HHB- 226, W-445 and GHB-732 was 10.4, 11.2, 8.8, 9.0, 11.3, and 9.8 g, respectively. The grains from cv.W-445 were whiter in colour whereas those from other cultivars were light brown in colour.

2.2. Starch isolation

Starch was isolated from pearl millet grains by following the method described by Sandhu and Singh (2005). About 500 g of clean, sound and whole grains (10–20 g/100 g moisture content) were added to 1.25 L of distilled water containing sodium *meta*-bisulphite (0.1 g/100 g). The mixture was maintained at 50 °C for about 18–20 h with intermittent circulation of liquid. After 20 h, the steep water was drained off and grains were ground in laboratory grinder (Maxie Plus, New Delhi, India). About 250 g of steeped grains were ground with 250 ml of distilled water. The ground slurry was passed through 0.250, 0.150, 0.100, 0.075, 0.045 mm sieve. The starch-protein slurry was then allowed to stand for 4–5 h. The supernatant was removed by suction and the settled starch layer was re-suspended in distilled water and centrifuged in wide mouthed cup centrifuge (Remi, New Delhi, India) at 605 *g* for 10 min and the upper non-white layer was scrapped off. The white layer was re-suspended in distilled water and re-centrifuged 3–4 times. The starch was then collected and dried in an oven (NSW-143, New Delhi, India) at 45 °C for 12 h. The starch samples were estimated for their moisture, ash, fat, and protein content by employing the standard methods of analysis (AOAC, 2000). The yield of starch depends on the method adopted for starch isolation. In our case the yield of the starch from different cultivars ranged from 42.7 to 47.5 g/100 g.

2.3. Amylose content

The amylose content of starch was determined by following the method described by Williams, Kuzina, and Hlynka (1970). Starch (0.020 g) was thoroughly mixed with 10 ml of 0.5 mol/L KOH. The dispersed sample was transferred to 100 ml volumetric flask and diluted to the mark with distilled water. An aliquot of test starch solution (10 ml) was pipetted into 50 ml volumetric flask and 5 ml of 0.1 mol/L HCl was added followed by the 0.5 ml of iodine reagent. The volume was diluted to 50 ml and the absorbance was measured at 625 nm in a spectrophotometer (Systronics, Ahmadabad, India). The measurement of the amylose was determined in triplicate from a standard curve developed using amylose and amylopectin blends. The samples were not defatted so the amylose content reported is apparent amylose content.

2.4. Swelling power and solubility

The swelling power and solubility of starches were determined by following the method described by Leach, McCowen, and Schoch (1959). Starch (1 g) was added to 99 ml of distilled water and heated to 90 °C for 1 h. The heated samples were cooled rapidly in ice water bath for 1 min, equilibrated at 25 °C for 5 min and then centrifuged at 605 *g* for 30 min. The supernatants were drained into pre-weighed moisture dishes, evaporated to dryness in a hot air oven at 100 °C and cooled to room temperature in a desiccator prior to reweighing.

2.5. Thermal properties of starches

Thermal characteristics of starches from different cultivars were studied by using a Differential Scanning Calorimeter (DSC) (Mettler Toledo, Switzerland) equipped with a thermal analysis data station. Starch (3.5 mg, dry weight) was loaded into a 40 μl capacity aluminium pan (Mettler, ME-27331) and distilled water was added with the help of Hamilton microsyringe to achieve a starch-water suspension containing 70 g/100 g water. Samples were hermetically sealed and allowed to stand for 1 h at room temperature before heating in DSC. The DSC analyzer was calibrated using indium and an empty aluminium pan was used as reference. Sample pans were heated at a rate of 5 °C/min from 40 to 100 °C. Thermal transitions of starch samples were defined as T_o (onset), T_p (peak of gelatinization) and T_c (conclusion), and ΔH_{gel} was referred to enthalpy of gelatinization. Enthalpies were calculated on starch dry basis. These were calculated automatically. The gelatinization temperature range (R) and peak height index (PHI), was calculated as $2(T_p\text{-}T_o)$ and $\Delta H/(T_p\text{-}T_o)$, respectively.

2.6. Pasting properties

The pasting properties of pearl millet starch were determined using a starch cell of Modular Compact Rheometer (Model-52, Anton Paar, Austria) by following the method described by Rincón-Londono, Millan-Malo & Rodríguez-García (2016b) with slight modifications. The starch was adjusted to 12 g/100 g moisture content. 2.5 g of starch and 15 ml of distilled water were used for the pasting profile that was carried out under the following conditions: Initially, the temperature of the system was 50 °C, and it remained constant for 1 min. The sample was heated for 5.3 min from 50 to 90 °C and then held at a constant temperature of 90 °C for 5.3 min. After this, the samples were cooled down to 50 °C for 5.3 min, and this temperature was kept constant for 1 min. All tests were carried out at a constant frequency of 193 rpm.

2.7. Rheological properties

2.7.1. Dynamic properties

A small amplitude oscillatory rheological measurement was made for different starches with a Modular Compact Rheometer (Model-52, Anton Paar, Austria) equipped with parallel plate system (0.04 m diameter). The gap size was set at 1000 μm. The strain