



# Structural and technological characteristics of starch isolated from sorghum as a function of drying temperature and storage time



David Bandeira da Cruz, Wagner Schellin Vieira da Silva\*, Ivonir Petrarca dos Santos, Elessandra da Rosa Zavareze, Moacir Cardoso Elias

Departamento de Ciência e Tecnologia Agroindustrial, Universidade Federal de Pelotas, Av. Eliseu Maciel s/n, 96010-900 Pelotas, RS, Brazil

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## ABSTRACT

The quality of sorghum grains can vary according to the conditions of the drying temperature and storage time. The objective of this study was to evaluate the effects of the drying temperature and storage time of sorghum grain on the structure and technological properties of starch. The sorghum grains were dried at 45, 65, and 85 °C and stored for six months. The grains were stored in an environment with a controlled temperature and humidity, and the starch from sorghum grains was isolated in initial time, after three and six months. The sorghum starches grains dried at 45 and 65 °C present higher relative crystallinity than the starches of sorghum grains dried at 85 °C in three months of storage. A reduction in the solubility of the starches of the sorghum grains dried at 85 °C was observed when the grains were stored during six months. The breakdown and swelling power of the starches were reduced with the increase of the drying temperature.

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## 1. Introduction

Sorghum (*Sorghum bicolor* (L.) Moench) is the fifth most important grain in the world (Singh, Chang, Lin, Singh, & Singh, 2011), being an excellent source of energy used both in animal and human nutrition. The cereal present has an average of 68.8% to 78.6% starch, 8.9% to 11.3% protein, 0.4% to 1.6% ash, 0.8% to 3.9% of fat, all in wet basis, moisture ranging from 9.2% to 14.8%, and is an important alternative to replacing corn (Liu et al., 2012).

Due to its good potential and its low requirement of technological production, sorghum is an important culture for small and medium producers. The human and animal consumption requires that the grain passes through a pre-process which may result in changes in the bioavailability of nutrients. The drying process allows the storage of grain for longer by reducing the moisture content to levels that allow for safe storage and their nutritional value. The nutritional quality of sorghum protein is of concern because, besides being low in the essential amino acid lysine, it has a lower digestibility than the protein of other cereal grains (Oria, Hamaker, Axtell, & Huang, 2000). However, sorghum grain has a high starch content between 70 and 80%, which can be used in various industrial applications, such as food ingredients, paper and

textile industries, alcohol production, and biodegradable containers.

According to Peplinski, Paulis, Bietz, and Pratt (1994), high temperatures used during the drying process can affect the performance in the wet milling of corn and modify the physicochemical properties of the starch granules recovered. When stored, the grains are subject to the action of several factors such as heat, moisture, oxygen, associated organisms, intrinsic enzymatic activity of the grain due to its metabolism, and other factors. Recently, interest has increased for the effect of a high drying temperature of the grains in the physicochemical and functional properties of the starches (Malumba, Massaux, Deroanne, Masimango, & Béra, 2009).

Previous studies have shown that high drying temperatures of the corn grains reduce the swelling power of the starch granules and their rates of solubility during gelatinization (Malumba et al., 2009). It is likely that structural changes possibly occurring within the granules during the drying process may affect the pasting characteristics of the starch in the water system during the subsequent gelatinization. Therefore, changes in the physicochemical and functional properties of starch extracted from grains dried at high temperature cannot be exclusively attributed to the samples residual protein. The aim of this study was to evaluate the effects of drying temperature and storage time of sorghum grain on the structural and technological properties of starch.

\* Corresponding author. Tel.: +55 53 3275 7284; fax: +55 53 3275 7284.  
E-mail address: [wagnersvsvila@yahoo.com](mailto:wagnersvsvila@yahoo.com) (W.S.V.d. Silva).

## 2. Materials and methods

### 2.1. Materials

Sorghum grains [*S. bicolor* (L.) Moench] produced in the 2013 growing season at Aceguá (31°52′07.0″S 54°09′49.1″W), Brazil, were manually harvested with a moisture content around 22%.

### 2.2. Drying and storage of sorghum grains

The sorghum grains were dried in a stationary dryer until around 12.5% of moisture in the air drying temperature of 45, 65 and 85 °C and drying time of 442 min, 152 min and 67 min, respectively. After drying, the samples were divided into portions of 1600 g for storage during six months at 20 °C. The moisture content was measured through weight difference by the equation: final weight  $\times$  (100 – final moisture) = initial weight  $\times$  (100 – initial moisture).

### 2.3. Starch isolation

The starch was isolated from sorghum grains in initial time, after three and six months, and then evaluated. The isolation of the starch was carried out according to the method described by Sandhu, Singh, and Malhi (2005), with some modifications. A sample of 200 g of sorghum grains was added to 500 mL of 0.1% a sodium bisulfite solution at room temperature for 24 h. After this hydration, the grains were drained and underwent wet grinding with 1000 mL of distilled water. After grinding, a sieving was done with an 80 mesh sieve, and then with a 120 mesh, leaving the filtrate to stand for 2 h at room temperature; then the supernatant was discarded, the precipitate was resuspended and centrifuged at 5000  $\times$  g for 20 min, eliminating the protein fraction. This process was repeated again for protein removal. The starch was dried at 40 °C for 12 h until to reach 7 to 10% of moisture and then ground. The nitrogen content of the sorghum starches was determined by the according to AACC method 46-13 according to McGrance, Cornell, and Rix (1998) and after, the protein content was obtained using a conversion factor of nitrogen to protein of 6.25.

### 2.4. Morphology of the starch granules

The morphology of starch granules was examined by a scanning electron microscope (Shimadzu, SSX-550) according to method described by Vanier et al. (2012). Starch samples were initially suspended in acetone to obtain a 1% (w/v) suspension, and the samples were maintained in an ultrasound bath for 15 min, for individualization of the starch granules. A small quantity of the sample was spread directly on the surface of the stub and dried in an oven at 32 °C for 1 h. All the samples were subsequently coated with a thin gold layer and examined under a scanning electron microscope at an acceleration voltage of 15 kV and 1000 $\times$  magnification.

### 2.5. X-ray diffractograms and relative crystallinity

X-ray diffractograms of the sorghum starches were obtained with an X-ray diffractometer (XRD-6000, Shimadzu, Brazil) according to method described by Paraginski et al. (2014). The scanning region of the diffraction ranged between 3° and 45°, with a target voltage of 30 kV, a current of 30 mA, and a scan speed of 1°/min. The relative crystallinity (RC) of the starch granules was calculated as described by Rabek (1980) by the equation: RC (%) =  $(Ac/(Ac + Aa)) \times 100$ ; where *Ac* is the crystalline area (below the peak), and *Aa* is the amorphous area (total area – crystalline area) on the X-ray diffractograms.

### 2.6. Thermal properties

The thermal properties of the sorghum starches were determined according to method described by Vanier et al. (2012) using differential scanning calorimetry (TA-60WS, Shimadzu, Kyoto, Japan). Starch samples (approximately 2.5 mg, dry basis) were weighed directly in an aluminum pan and distilled water was added to obtain a starch-water ratio of 1:3 (w/w). The pan was hermetically sealed and allowed to equilibrate for 1 h before analysis. The samples were heated in a nitrogen atmosphere from 20 to 100 °C at a rate of 10 °C/min. An empty pan was used as a reference. The temperature at the onset of gelatinization ( $T_0$ ), peak temperature ( $T_p$ ), temperature at the end of gelatinization ( $T_c$ ), and the enthalpy ( $\Delta H$ ) of gelatinization were determined.

### 2.7. Pasting properties

The pasting properties of the sorghum starches were determined according to method described by Paraginski et al. (2014) using a Rapid Visco Analyser (RVA-4, Newport Scientific, Australia) with a Standard Analysis 1 profile (Newport Scientific, 1995). The viscosity was expressed in rapid visco units (RVU). Starch (3.0 g of 14 g/100 g wet basis) was weighted directly in the RVA canister, the samples had their moisture acquired by the stove method on stove at 105 °C for 24 h, and adjusted to complete 25 mL with distilled water was then added to the canister. The sample was held at 50 °C for 1 min, heated to 95 °C in 3.5 min, and then kept at 95 °C for 2.5 min. The sample was cooled to 50 °C in 4 min and then kept at 50 °C for 1 min. The rotating speed was maintained at 960 rpm for 10 s, and it was maintained at 160 rpm during the remaining process. Parameters, including pasting temperature, peak viscosity, breakdown, final viscosity, and setback, were recorded.

### 2.8. Swelling power and solubility

The solubility and swelling power of the sorghum starches were determined as described by Leach, McCowen, and Schoch (1959). Samples (1.0 g, dry basis) were mixed with 50 mL of distilled water in centrifugal tubes. The suspensions were heated at 90 °C for 30 min. The gelatinized samples were then cooled to room temperature and centrifuged at 1000  $\times$  g for 20 min. The supernatant was dried at 110 °C to a constant weight to quantify the soluble fraction. The solubility was expressed as the percentage of dried solid weight based on the weight of the dry sample. The swelling power was represented as the ratio of the weight of the wet sediment to the weight of the initial dry sample (deducting the amount of soluble starch).

### 2.9. Statistical analysis

All analytical determination was performed in triplicate and its results were submitted to the analysis of variance by the Tukey test ( $p \leq 0.05$ ) except for the DSC analysis and X-ray diffractograms which were made in duplicates.

## 3. Results and discussion

### 3.1. Morphology of the starch granules

The scanning electron micrographs of the starches of sorghum grains dried at 45 °C, 65 °C and 85 °C and stored for six months are shown in Fig. 1.

The sorghum starches showed the presence of irregular-polyhedral as well as spherical granules. The drying temperature and time of storage of the grain sorghum did not affect the morphology of the starch granules (Fig. 1). The granules of starches

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