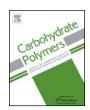
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# Characteristics of starch isolated from maize as a function of grain storage temperature



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#### ABSTRACT

Considering the importance of maize starch and the lack of knowledge about the effects of storage temperature on the isolated starch properties; maize grains were stored during 12 months at different temperatures (5, 15, 25 and 35 °C). The extraction yield and the physicochemical, thermal, pasting, crystallinity and morphological properties of starches were determined. The starch isolated from grains stored at 35 °C was yellowish and showed a 22.1% decrease in starch extraction yield compared to freshly harvested maize grains. At 35 °C, a reduction in crystallinity was observed by the end of 12 months, despite a parallel rearrangement of the starch chains which resulted in an increase in X-ray peak intensities, gelatinisation temperatures and enthalpy. The starch isolated from maize grains stored at 35 °C appears to have smaller granules, which presents some points in their surface, potentially attributed to the protein matrix compressing the granules within maize grains.

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#### 1. Introduction

Starch is widely used in the food industries, especially in the preparation of soups, sauces, baked goods, dairy, confectionery, snacks, pasta, coatings and products made with meat (Davies, 1995). The ability of starch to form a viscous paste when heated in water followed by the cooling property makes starch suitable for various uses in the food and non-food industries (Nguyen, Jensen, & Kristensen, 1998). The main botanical source used for extraction of starch is maize, accounting for about 80% of the world market (Jobling, 2004). Among all kinds of starches, maize starch is an important ingredient in the production of foodstuffs, and has been widely used as a thickener, stabiliser, colloidal gelling agent, water retention and as an adhesive (Singh, Singh, Kaur, Sodhi, & Gill, 2003). Starch is the main constituent of maize kernels, about 72–73% of the total weight (Sandhu, Singh, & Lim, 2007).

After harvested, the maize grains are subjected to various postharvest steps, such as cleaning, drying and storage. Several studies have elucidated the effects of drying temperature on the properties of isolated starches (Altay & Gunasekaran, 2006; Eckhoff & Watson, 2009; Haros, Tolaba, & Suarez, 2003; Lasseran, 1973; Malumba et al., 2010; Malumba, Massaux, Deroanne, Masimango, & Béra, 2009; Setiawan, Widjaja, Rakphongphairoj, & Jane, 2010). According to Malumba et al. (2009), drying temperatures of maize grains up to 100 °C cause changes in the pasting and texture properties of the starch gel, and reduce the extraction yield as well as purity of starch.

The storage results in reduced solubility and digestibility of grain proteins (Rehman, Habib, & Zafar, 2002), increased free fatty acids (Park, Kim, Park, & Kim, 2012), and these may form complexes with amylose or amylopectin short chains, altering the nutritional properties and the physical characteristics of the final products (Hasjim et al., 2010; Salman & Les, 2007). Long periods of storage reduce the yield of cassava starch extraction during wet-milling, as a result of starch degradation and the interactions between starch and other constituents (Abera & Sudip, 2004). Setiawan et al. (2010) stored maize grains at 27 °C and around 85-90% of relative humidity for 6 months, reporting changes in the pasting, thermal, morphological and crystallinity properties of starch; without considering the effects of storage temperature on the properties of isolated starch. Yousif et al. (2003) reported an increase in gelatinisation temperature of adzuki bean (Vigna angularis) starch with increasing storage temperature. Rupollo et al. (2011) evaluated the effects of storage

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conditions of common bean (*Phaseolus vulgaris* L.) grains on physicochemical, pasting, crystallinity and morphological properties of isolated starch, observing changes in the thermal properties and crystallinity of starch isolated from grains stored at  $25\,^{\circ}\text{C}$  during 360~days.

Considering the importance of maize starch in the world market and the lack of knowledge about the effects of temperature during maize grains storage on the isolated starch properties, the aim of this study was to evaluate the physicochemical, pasting, thermal, morphological and crystallinity properties of starches isolated from maize grains stored for 12 months at different temperatures.

#### 2. Materials and methods

#### 2.1. Storage of grains

Maize grains produced in the 2012 growing season at Santo Augusto (27°53′18″ S, 53°47′20″ W, 489 m) in the State of Rio Grande do Sul, Brazil, were used. The grains were placed into raffia bags after harvested and immediately transported to the Postharvest, Industrialisation and Quality of Grains Laboratory of DCTA-FAEM-UFPel, where the experiments were carried out. The grains were harvested mechanically, subjected to artificial drying with air temperature of 35 °C until 14% of moisture was achieved, and subsequently purged using aluminium phosphide to prevent the interference of insects in the experiment. The maize grains were stored in polyethylene bags of 0.2 mm thick plastic film with a capacity of 0.9 kg at temperatures of 5, 15, 25 and 35 °C for 12 months, in triplicate. The grains were maintained covered from the light by an aluminium foil.

#### 2.2. Starch isolation

The isolation was performed according to the method described by Sandhu, Singh, and Malhi (2005), with some modifications. Maize grains (200 g) were added to 500-ml of 0.1% sodium bisulfite (NaHSO<sub>3</sub>) in distilled water, and maintained for 20 h at 50 °C. After this period, the water was drained and the grains were crushed in a grinder (Electronic Filter 600 W, Britânia, São Paulo, Brazil) until the smallest possible fraction (wet milling) was achieved. The crushed samples were double filtered through 100 and 270mesh sieves. The protein-starch filtrates were decanted for 4 h. The supernatant was removed and the sedimented protein-starch layer was resuspended in distilled water to be centrifuged at  $5000 \times g$ for 20 min. The resulting protein rich supernatant was removed and the remaining starch slurry was resuspended once again in distilled water before further centrifugation to completely remove any remaining protein content. The collected starch was dried at 40 °C for 12 h in an oven until 11% of moisture was achieved. Once dry, the starch was placed in a laboratory mill (Perten 3100, Perten Instruments, Huddinge, Sweden) with 70-mesh sieve for attaining uniform particle size distribution. A total of 100 g kernels were used to determine the percentage extraction yield by weighing the starch obtained after drying. The starch was isolated from freshly harvested maize grains, before storage, and considered as the initial treatment. Then, the starch was isolated from maize grains and stored under time-temperature conditions mentioned above.

#### 2.3. Colour parameters

The colour of the isolated starches was determined using a colorimeter (Minolta, CR-310, Osaka, Japan). The colour parameters used were  $L^*$  (100 = white and 0 = black) and  $b^*$  (positive = yellow and negative = blue).

#### 2.4. Protein and fat contents

The nitrogen content was determined using the AACC method 46-13 (AACC, 1995), and the protein content was obtained using a conversion factor of nitrogen to protein of 6.25. The fat content was determined in accordance with the AACC method 30-20 (AACC, 1995)

#### 2.5. Swelling power and solubility

The swelling power and solubility of the starches were determined as described by Leach, McCowen, and Schoch (1959). Samples (1.0 g) were mixed with 50 ml of distilled water in centrifuge tubes. The suspensions were heated at 90 °C for 30 min. The gelatinised samples were then cooled to room temperature and centrifuged at  $1000 \times g$  for 20 min. The supernatants were dried at 110 °C until a constant weight was achieved so that the soluble fraction could be quantified. Solubility was expressed as the percentage of the dried solid weight based on the dry sample weight. Swelling power was represented as the ratio of wet sediment weight to initial dry sample weight (deducting the amount of soluble starch).

#### 2.6. Pasting properties

The pasting properties of the maize starches (3.0 g, 14% wet basis) were determined with a Rapid Visco Analyser (RVA-4; Newport Scientific, Warriewood, Australia) and profile Standard Analysis 1. The viscosity was expressed in rapid visco units (RVU). The samples were held at 50 °C for 1 min, heated to 95 °C at 3.5 min and held at 95 °C for 2.5 min. The samples were then cooled to 50 °C in 4 min and held at 50 °C for 2 min. The rotating speed was held at 960 rpm for 10 s and then maintained at 160 rpm during the process. Parameters including pasting temperature, peak viscosity, breakdown, final viscosity and setback were recorded.

#### 2.7. Differential scanning calorimetry (DSC)

Gelatinisation characteristics of the maize starches were determined using differential scanning calorimetry (TA-60WS, Shimadzu, Kyoto, Japan). Starch samples (approximately 2.5 mg on a dry basis) were weighed directly in an aluminium pan (Mettler, ME-27331), and distilled water was added to obtain an aqueous suspension containing 75% water. The pan was hermetically sealed and allowed to equilibrate for 1 h before analysis. An empty pan was used as a reference. The sample pans were then heated from 40 to 140 °C at the rate of 10 °C min<sup>-1</sup>. The onset temperature of gelatinisation ( $T_0$ ), peak temperature ( $T_p$ ), conclusion temperature ( $T_c$ ) and gelatinisation enthalpy ( $\Delta H$ ) were determined. The range of gelatinisation was calculated by subtracting  $T_0$  from  $T_c$ .

#### 2.8. Crystallinity

The crystallinity of starches was determined with an X-ray diffractometer (XRD-6000, Shimadzu, Brazil). The scanning region of the diffraction ranged from  $5^{\circ}$  to  $30^{\circ}$  with a target voltage of  $30 \, \text{kV}$ , current of  $30 \, \text{mA}$  and scan speed of  $1^{\circ} \, \text{min}^{-1}$ . The relative crystallinity (RC) of the starch granules was calculated as described by Rabek (1980) using following the equation:

$$RC(\%) = \frac{A_c}{A_c + A_a} \times 100$$

where  $A_c$  is the crystalline area; and  $A_a$  is the amorphous area on the X-ray diffractograms.

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