

## Extraction and chemical characterization of starch from *S. lycocarpum* fruits



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### ABSTRACT

In this study the pulp from *Solanum lycocarpum* fruits was used as raw material for extraction of starch, resulting in a yield of 51%. The starch granules were heterogeneous in size, presenting a conical appearance, very similar to a high-amylose cassava starch. The elemental analysis (CHNS) revealed 64.33% carbon, 7.16% hydrogen and 0.80% nitrogen. FT-IR spectroscopy showed characteristic peaks of polysaccharides and NMR analysis confirmed the presence of the  $\alpha$ -anomer of D-glucose. The *S. lycocarpum* starch was characterized by high value of intrinsic viscosity (3515 mPa s) and estimated molecular weight around 645.69 kDa. Furthermore, this starch was classified as a B-type and high amylose content starch, presenting 34.66% of amylose and 38% crystallinity. Endothermic transition temperatures ( $T_o = 61.25^\circ\text{C}$ ,  $T_p = 64.5^\circ\text{C}$ ,  $T_c = 67.5^\circ\text{C}$ ), gelatinization temperature ( $\Delta T = 6.3^\circ\text{C}$ ) ranges and enthalpy changes ( $\Delta H = 13.21 \text{ J g}^{-1}$ ) were accessed by DCS analysis. These results make the *S. lycocarpum* fruit a very promising source of starch for biotechnological applications.

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### 1. Introduction

Starches are ubiquitous semi-crystalline polymers composed mainly by amylose and amylopectin. Amylose is, a mostly linear chain, is typically composed by D-glucopyranosyl subunits interconnected primarily by  $\alpha$ -(1  $\rightarrow$  4) glycosidic linkages with presence of lightly branched molecules. Amylopectin is a large branched polymer formed by  $\alpha$ -(1  $\rightarrow$  4)-D-glucopyranose units with  $\alpha$ -(1  $\rightarrow$  6)-linkages at the branching points (Beninca et al., 2013; Trommsdorff & Tomka, 1996).

This polysaccharide is widely used in food, textile, paper, chemical, pharmaceutical and biotechnological industries (Buléon et al., 1997; Falade & Okafor, 2013; Schirmer, Höchstötter, Jekle, Arendt, & Becker, 2013). The selection of starch for industrial uses is made by considering its availability and also its physico-chemical characteristics, that vary depending on the source (Hoover, 2001; Joshi et al., 2013).

Another important research area where new sources of starch are constantly requested is the biofuel industry. The public concern about greenhouse gas emissions and oil prices spikes resulted in an increased attention on renewable sources of fuel (Lee, Chen,

Chang, & Yang, 2012). In this scenario, bioethanol obtained by starch hydrolysis followed by fermentation justify the study of new sources of starches with chemical structure that provides high yield at industrial scale.

*Solanum lycocarpum* St. Hill (Solanaceae) is a common and abundant plant in the Brazilian Cerrado. It presents a high resistance to hydric and climatic stress, surviving and fructifying along the year. Its fruits weigh from 400 g to 900 g and are consumed fresh or cooked in some regions (Torralbo, Batista, Di-Medeiros, & Fernandes, 2012). The abundance of *S. lycocarpum* in the Brazilian Cerrado and its high fruit production make this plant a very attractive target for biotechnological exploitation (Silva-Filho, Torralbo, Di-Medeiros, Batista, & Fernandes, 2012). The presence of high content of starch in the fruits makes *S. lycocarpum* a potential source of this polysaccharide (Clerici et al., 2011).

However, the functionality of a starch depends on the molecular size, crystallinity degree, amylose content and, specially, on the viscosity properties. (Falade & Okafor, 2013; Joshi et al., 2013). In this sense, the aim of this study was to extract and partially characterize the starch from *S. lycocarpum* fruits. The composition, structure, and physico-chemical properties of *S. lycocarpum* starch were assessed by Fourier transform infrared spectroscopy, nuclear magnetic resonance, scanning electron microscopy, elemental analysis (CHNS) and metal composition, X-ray diffraction, amylose content and viscosity measurements.

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## 2. Materials and methods

### 2.1. Material

The *S. lycocarpum* unripe fruits used in this study were collected in the farm of the Universidade Federal de Goiás (Goiânia-GO, Brazil).

### 2.2. Extraction of starch

After being harvested and selected, the fruits were washed, decorticated and the seeds were removed. The pulp was sliced and milled in a blender, in the presence of 0.8% (w/v) citric acid solution. This material was deposited in a sieve (32 mesh) and washed successively with 0.05 mol L<sup>-1</sup> NaOH and ethanol. Then it was centrifuged at 1430 g and the supernatant discharged. The precipitated starch was dried in an air forced oven at 45 °C until constant weight.

The starch yield was calculated using the following equation:

$$Y_{\text{starch}}(\%) = 100 \cdot \left( \frac{S}{P_i} \right)$$

where  $Y_{\text{starch}}$  is the percentage yield of starch extracted,  $S$  is the amount in grams of starch extracted and  $P_i$  is the amount of initial *S. lycocarpum* pulp in dry basis.

### 2.3. Characterization of starch

#### 2.3.1. Scanning electron microscopy (SEM)

Samples of native starches were examined in a scanning electron microscope JEOL, JSM - 6610, using a secondary electron detector with 15 kV of acceleration.

#### 2.3.2. Elemental analysis (CHNS) and metals

The chemical composition of *S. lycocarpum* starch was determined by using elemental analyzer (Elementary Vario EL III Carlo-Elba, model 1108) for carbon, hydrogen, nitrogen, and sulfur.

The contents of calcium, copper, zinc, magnesium and manganese were analyzed in an atomic absorption spectrometer (PerkinElmer, Inc., MA, USA). The contents of potassium and sodium were analyzed in an atomic emission photometer (Corning 400). All solutions were prepared with distilled and deionized water (resistivity of 18.2 MW cm<sup>-1</sup>). The analytical curve for the quantification of mineral elements was prepared by diluting a standard solution of each metal in the following concentrations range: Ca and Mn 1–4 mg L<sup>-1</sup>; Na and Mg 0.5–2.5 mg L<sup>-1</sup>; Cu 1–5 mg L<sup>-1</sup>; Zn 0.4–1.6 mg L<sup>-1</sup>; and K 0.5–3 mg L<sup>-1</sup>.

#### 2.3.3. Fourier transform infrared (FT-IR) spectroscopy

The FTIR spectrum of *S. lycocarpum* starch was acquired on a PerkinElmer FTIR spectrophotometer (PerkinElmer, Inc., MA, USA) using potassium bromide (KBr) discs prepared from powdered samples mixed with dry KBr. Spectrum was recorded (16 scans) in the transparent mode from 4000 to 400 cm<sup>-1</sup>, at 4 cm<sup>-1</sup> resolution.

#### 2.3.4. NMR analysis

The <sup>13</sup>C NMR measurements were performed with Bruker AVANCE III 500 spectrometer, operating at 500.13 MHz. To acquire <sup>13</sup>C spectrum, samples of starch were pre-hydrolyzed using amylase immobilized onto polyaniline, according to methodology described by Pascoal, Mitidieri, & Fernandes (2011). The product was concentrated by lyophilizing and used in NMR analysis. Samples containing 18 mg of starch were dissolved in deuterated water and 1% (w/v) sodium-3-trimethylsilylpropionate-2,2,3,3-d4 (TMSP-D4) were used as internal standard.

#### 2.3.5. Amylose content

The amylose content was determined according to the AACC method (2000). The color of starch–iodine complex developed at pH 4.5–4.8 in acetate buffer was read in a spectrophotometer at 620 nm. The blue color developed was read against a standard amylose (Sigma–Aldrich) curve plotted from solution with concentration of 0.004–0.024 mg amylose per mL of water.

#### 2.3.6. Viscosity measurement

The viscosity was measured with a Brookfield LVDV-II+ PRO digital rotational viscometer (Brookfield Engineering Labs., Inc., Middleboro, MA, U.S.A.).

Aqueous starch solutions in concentrations varying from 0.2 to 5% (w/v) were heated at 40 °C for 10 min and then left to rest until room temperature was reached. The solutions were transferred into the Teflon® cup reservoir of the viscometer. The recording of output parameters started 2 min after the experiment onset. Changes of the dynamic viscosity values of the system were measured at 25.0 ± 0.1 °C.

The molecular weight of starch from *S. lycocarpum* was estimated by applying the Mark Houwink–Sakurada equation, relating intrinsic viscosity ( $[\eta]$ ) with molecular weight ( $M_w$ ):

$$[\eta] = k_H [M_w]^a$$

where  $k_H$  and  $a$  (0.89) (Foster, 1965) are constants. The intrinsic viscosity was calculated by determination of viscosities of solutions of various concentrations, followed by extrapolation of  $\eta_{sp}/c$  to zero concentration, according to the following equation:

$$[\eta] = \lim_{c \rightarrow 0} \frac{\eta_{sp}}{c}$$

where  $\eta_{sp}$  is the specific viscosity and  $c$  is a concentration of the starch solution. In a range of moderate concentrations, the dependence is linear and the  $k_H$  constant can be calculated using the Huggins equation (Huggins, 1942):

$$\frac{\eta_{sp}}{c} = [\eta] + k_H [\eta]^2 c$$

#### 2.3.7. X-ray

The X-ray analysis was performed in samples of starch in powder form using a Shimadzu XRD-6000 X-ray diffractometer operating under the following conditions: tube Cu K $\alpha$  radiation, 40 kV and 100 mA, scan ranging from 10.000 to 80.000. To avoid interference of relative humidity, samples were dried for 24 h and placed in a desiccator until use.

The degree of crystallinity was quantitatively estimated, following the method of Nara and Komiy (1983). The crystallinity was expressed through the mathematical calculation of the peak area under the curve in X-ray diffraction pattern. The degree of crystallinity was calculated according to the equation:

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

where  $D_c$  refers to the degree of crystallinity;  $A_c$  refers to the crystallized area on the X-ray diffractogram;  $A_a$  refers to the amorphous area on the X-ray diffractogram.

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

Thermal characteristics of *S. lycocarpum* starch were studied with a Mettler Toledo Differential Scanning Calorimeter, Model 822 $^{\circ}$ . Samples (3.5 mg) were weighed into aluminum pans and deionized water (8  $\mu$ L) was added. The pans were hermetically sealed and kept at room temperature overnight before analysis. The samples were heated from 25 to 120 °C, at a rate of 10 °C min<sup>-1</sup> using aluminum crucible with a perforated lid cover. From the

**Table 1**  
Extraction yield and amylose content of starches from different sources.

| Source of starch                     | Yield <sup>a</sup> (%)                                                     | Amylose content (%)                          |
|--------------------------------------|----------------------------------------------------------------------------|----------------------------------------------|
| <i>Solanum lycocarpum</i> (lobeira)  | 51                                                                         | 34.66                                        |
| <i>Manihot esculenta</i> (Cassava)   | 33.5 (Hoover & Hadziyev, 1981)                                             | 20.67–36.23 (Ceballos et al., 2008)          |
| <i>Zeas mays</i> (Corn)              | 24.8 (Hoover & Hadziyev, 1981; Sriroth, Piyachomkwan, Wanlapatit, & Oates) | 15.3–26 (Sandhu, Singh, & Kaur)              |
|                                      |                                                                            | 27.9 (Jane et al., 1999; Singh et al., 2003) |
|                                      |                                                                            | 22.2 (Singh, Chawla, & Singh)                |
| <i>Solanum tuberosum</i> (potato)    | 32 (Hoover & Hadziyev, 1981)                                               | 19.1 (Singh et al., 2004)                    |
| <i>Oryza sativa</i> (rice)           | –                                                                          | 4.2–27.2 (Chung, Liu, Lee, & Wei)            |
| <i>Colocasia esculenta</i> (cocoyam) | –                                                                          | 11.55–33.77 (Falade & Okafor, 2013)          |

<sup>a</sup> Yield values shown above are based on dry basis.

curve, enthalpy of gelatinization ( $\Delta H$ ), gelatinization temperature ranges ( $\Delta T$ ), the onset ( $T_o$ ), peak ( $T_p$ ) and end ( $T_c$ ) temperatures were obtained using the data processing software supplied with the DSC instrument (Kim, Wiesenborn, & Grant, 1997).

### 3. Results and discussion

#### 3.1. Starch extraction

The extraction of *S. lycocarpum* starch resulted in a white fine inodorous powder (Fig. 1a), with a yield of 51% on dry basis. This yield is very high compared to those obtained from other traditional sources of starch (Table 1). The yield obtained for *S. lycocarpum* starch indicates that this fruit is a very promising starch source for commercial exploitation.

#### 3.2. Starch characterization

##### 3.2.1. Scanning electron microscopy (SEM)

Morphological characteristics of starches such as size and shape vary depending on the plant source. Besides the biological origin, the morphology of the starch granule depends on the biochemistry and metabolic routes occurring in the chloroplast or amyloplast, as well as physiology of the plant and agronomic and climatic conditions under which they were grown (Freitas, Paula, Feitosa, Rocha, & Sierakowskic, 2004; Singh, Singh, Kaur, Sodhi, & Gill, 2003). As can be seen in the Fig. 1, the surface of the *S. lycocarpum* starch granules is smooth, with no evidence of fissures. The starch granules were heterogeneous in size, presenting a conical appearance (Fig. 1b), very similar to a high-amylose cassava starch (Ceballos et al., 2008; Rolland-Sabaté et al., 2012). The granules showed sizes varying from 10 to 14.4  $\mu\text{m}$  (314–651.1  $\mu\text{m}^2$ ) for the hemispherical unit and 6.25–8.1  $\mu\text{m}$  (122.3–206  $\mu\text{m}^2$ ) for those subunits with irregular shapes (Fig. 1c). The average size of the *S. lycocarpum* starch granules are close to those presented by starches from tubers and roots (Hoover, 2001) and higher than those from cereals, such as barley (regular and waxy), maize (regular, high-amylose and waxy), regular and B-type wheat, corn and rice (Schirmer et al., 2013; Singh et al., 2003).

##### 3.2.2. Elemental analysis (CHNS) and metals

The elemental analysis showed that the *S. lycocarpum* starch presented the following composition: 64.33% carbon, 7.16% hydrogen and 0.80% nitrogen. The low percentage of nitrogen (traces) evidences of the high quality and the purity of the extracted starch.

The study of metal content is important from the nutritional point of view. Several metals, such as calcium, zinc and magnesium are beneficial to health whereas other, such as sodium, needs a special monitoring (Farahnaky, Farhat, Mitchell, & Hill, 2009). The presence of metal ions is also important under the perspective of enzymatic hydrolysis, since some metals may act as enzyme activator or inhibitors (Pascoal et al., 2011).

In general, the metals content depends on the environmental and climate conditions, soil fertility and composition, and harvesting periods (Clerici et al., 2011). The results presented in this study revealed amounts of 16.6 mg 100 g<sup>-1</sup> sodium, 17.5 mg 100 g<sup>-1</sup> potassium, 8.64 mg 100 g<sup>-1</sup> calcium, and 8.58 mg 100 g<sup>-1</sup> magnesium and lower content of manganese (0.7 mg 100 g<sup>-1</sup>), zinc (0.63 mg 100 g<sup>-1</sup>) and copper (0.006 mg 100 g<sup>-1</sup>). The soil composition may be the explanation for the variation observed in the metal content obtained by Clerici et al. (2011) in *S. lycocarpum* starch from fruits collected in other regions.

The actual concern over health related to the high intake of salt by consumers has stimulated the reduction of sodium chloride in different food products. The content of sodium in starch-based products has been investigated and it is known that it markedly influences the gelatinization and retrogradation, as well as the physical characteristics of the final product (Farahnaky et al., 2009; Maauf, Man, Asbi, Junainah, & Kennedy, 2001). In this case, knowing the sodium content in *S. lycocarpum* starch will allow predicting some properties of the products based on this starch.

On the other hand, the starch is a raw material for the production of glucose-rich syrup and derivatives, including fructose-rich syrup, dextrin, and recently the production of the biofuel ethanol. Enzymatic hydrolysis appears at least in one of the steps of obtaining these products. The enzymes are susceptible to the presence of metal ions which may activate or inhibit the enzymatic reaction. In this sense, is worthwhile quantify these metals before proposing applications for a starch from a new source, since ions such as calcium and magnesium generally actuate as activator for several amylases, whereas copper and zinc generally are inhibitors (Mitidieri, Martinelli, Schrank, & Vainstein, 2006; Pascoal et al., 2011; Sodhi, Sharma, Gupta, & Soni, 2005).

##### 3.2.3. FT-IR spectroscopy

The analysis of the FT-IR spectra was performed in order to identify the major functional groups of the extracted starch (Fig. 2). As it can be seen, the spectrum presents a broad strong band at 3420 cm<sup>-1</sup> due to the stretching vibration of O–H bond. A small peak in the region of 2930 cm<sup>-1</sup> is assigned to the stretching vibration of C–H bond from glucose. An important peak is that at 1644 cm<sup>-1</sup>, which has been frequently assigned to scissoring vibrations of –OH coming from water of hydration. In spite of the presence of nitrogen observed in the elemental analysis, the contribution of amino group deformations is negligible. Peaks in the region of 1460 cm<sup>-1</sup>, 1420 cm<sup>-1</sup> and 1383 cm<sup>-1</sup> are related to the folding of –CH bonds. The peaks at 1158 cm<sup>-1</sup>, 1080 cm<sup>-1</sup> and 1015 cm<sup>-1</sup> are related to the stretching vibration of C–O–C and C–O–H from glycosidic bonds, characteristic of polysaccharides (Pavia, Lampman, Kriz, & Vyvyan, 2010; Silva, Santiago, Purcena, & Fernandes, 2010).

##### 3.2.4. NMR analysis

The <sup>13</sup>C NMR spectrum of the *S. lycocarpum* starch is shown in Fig. 3. Assignments of resonances are consistent with literature



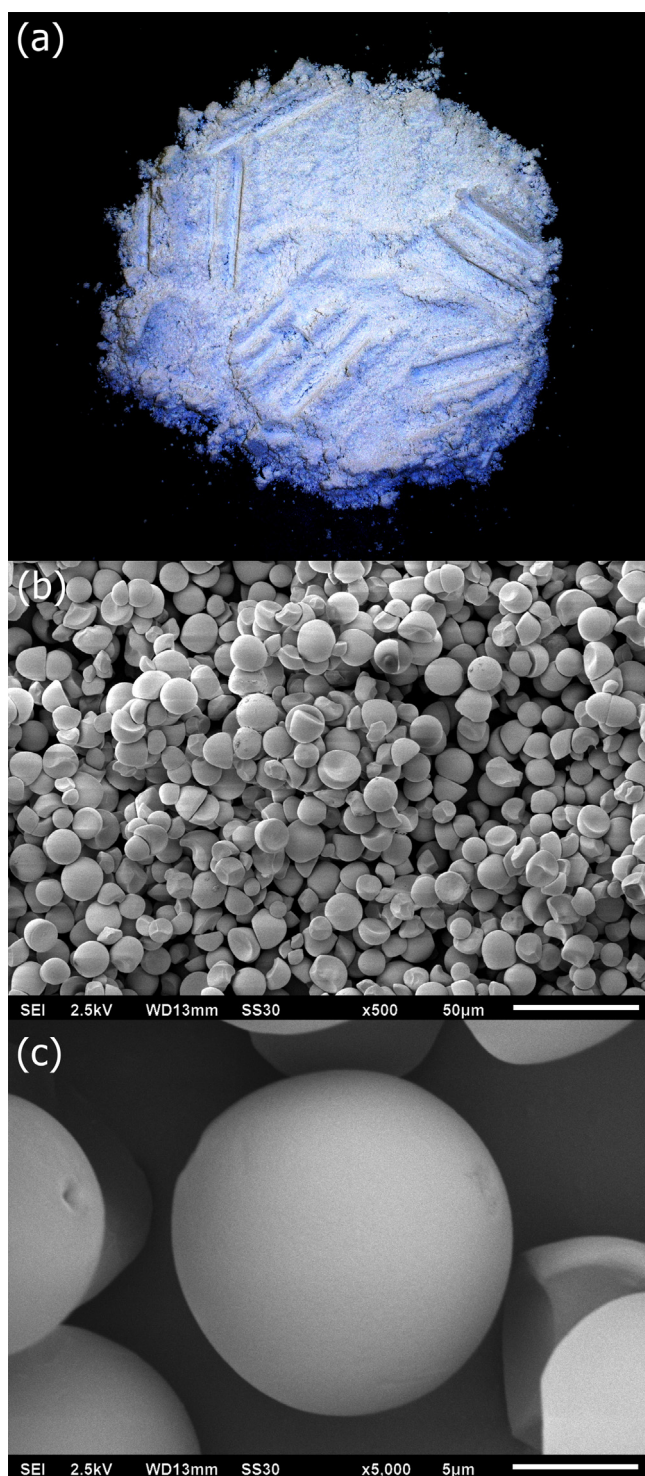


Fig. 1. Photograph (a) and SEM micrographics (b, c) of *Solanum lycocarpum* starch.

data (Cheetham & Tao, 1998; Gong, Wang, & Tu, 2006). The spectra show the signal at high field (99.8 ppm) related to C-1 (Fig. 3a) and a signal at  $\delta$  60.4 ppm (Fig. 3c) that can be assigned to C-6 (Mathew & Abraham, 2007; Wangsakan, McClements, Chinachoti, & Dickinson, 2004). The signals at  $\delta$  71.9,  $\delta$  73.1 and  $\delta$  71.5 ppm are respectively assigned to the C-2, C-3 and C-5 from the  $\alpha$ -anomer of the D-glucose unit characteristic of starch (Fig. 3b). Other important feature observed in the NMR spectra (Fig. 3) was the relative minor signals at  $\delta$  99.9,  $\delta$  70.2,  $\delta$  72.9 and  $\delta$  60.6 ppm which are attributed

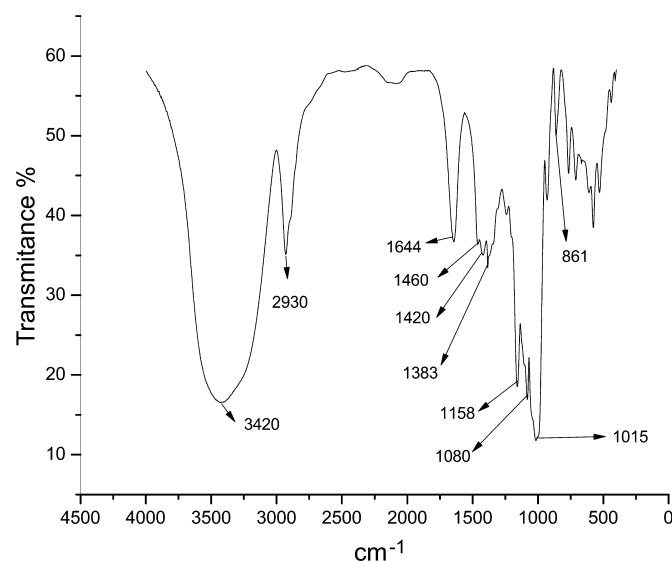


Fig. 2. Fourier transform infrared spectra of *Solanum lycocarpum* starch.

to C-1, C-4, C-5 and C-6 at the non-reducing glucopyranose unit from amylopectin, respectively (Gong et al., 2006).

### 3.2.5. Amylose content

The amylose content differs according to the source, but generally varies from 20 to 30% in cereals and from 15 to 25% in tubers (Joshi et al., 2013; Rolland-Sabaté et al., 2012; Weber, Callares-Queiroz, & Chang, 2009). The amylose content has an important effect on the starch chemical properties and thus it will determine the starch applications (Martinez & Prodolliet, 1996).

The *S. lycocarpum* starch presented 34.66% of amylose in its composition, value higher than those from other sources (Table 1).

The high amylose content of *S. lycocarpum* starch could allow its application in several industrial fields, such as film production with good oxygen barrier properties, water-low permeability biomembranes, food additive or covering and coating materials for pharmaceutical products (Joshi et al., 2013; Liu, Yu, Xie, & Chen, 2006; Mark, Roth, Mehlretter, & Rist, 1966; Silva, Ulhoa, Batista, Yamashita, & Fernandes, 2011; Weber et al., 2009).

In addition, high-amylose starches are of interest because of their potential health benefits. High levels of amylose are positively correlated to high content of resistant starch, which have been related to lowering the glycaemic and insulin responses as well as reducing the risk for developing type II diabetes, obesity and cardiovascular diseases (Falade & Okafor, 2013; Man et al., 2013).

### 3.2.6. Intrinsic viscosity and molecular weight

The intrinsic viscosity of a polymer solution is a measure of the capacity that a polymer molecule has to enhance the viscosity of a fluid. The intrinsic viscosity of *S. lycocarpum* starch was 3515 mPa s, value higher than those reported by Hoover (2001) for cassava (1728 mPa s) and reported by Kaur, Singh, McCarthy, & Singh (2007) for potato starches (2365–3377 mPa s). The viscosity properties of starches can be influenced by the size of the starch granule (Falade & Okafor, 2013; Li, Shoemaker, Ma, Moon, & Zhong, 2008), amylose content (Xie et al., 2009) and length of amylopectin chains (Vandeputte, Derycke, Geeromes, & Delcour, 2003).

Under the conditions used on this study, the molecular weight estimated for the starch from *S. lycocarpum* was 645.69 kDa.

### 3.2.7. X-ray diffractometry

Much of the information about starch granule crystalline properties can be acquired from X-ray diffraction studies. In general,

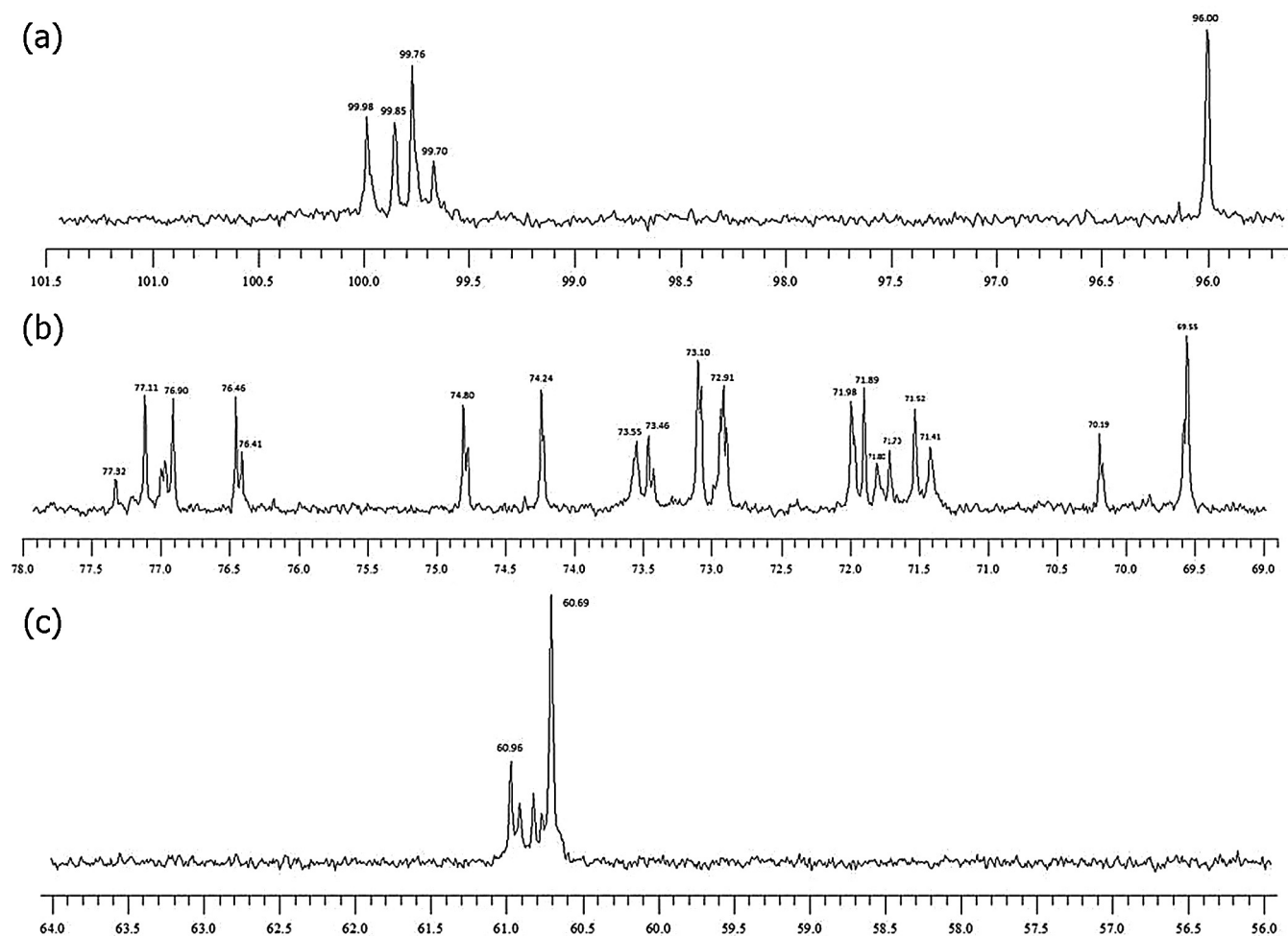


Fig. 3.  $^{13}\text{C}$  NMR spectra of native starches from *Solanum lycocarpum*.

the starches are classified as: (i) type A, associated mainly with cereal starches; (ii) type B, usually found in tuber starches; (iii) type C, a hybrid of A and B forms; and (iv) type V, where amylose is complexed with other non-carbohydrate compounds (Buléon et al., 1997; Cheetham & Tao, 1998; Matveev et al., 2001). Moreover, according to the work of Cheetham & Tao, 1998 and Matveev et al. (2001), the amylose content influences the pattern of starches diffractograms.

The diffractogram of the *S. lycocarpum* starch is compatible with an intermediate amylose content starch (ca. 40%). In spite of the absence of the reflection at  $2\theta$  about  $5^\circ$ , the diffractogram of the *S. lycocarpum* starch presents similarity with C-polymorph type, due to the strong reflection at  $2\theta$  about  $15.3^\circ$ ,  $17.2^\circ$ ,  $19.6^\circ$ ,  $24.4^\circ$  and the broad peak at  $2\theta$  about  $22.2^\circ$  (Fig. 4). A polymorph-C type comprises a mixture of A and B polymorphs and typically presents characteristic signatures at  $5.5^\circ$  (B polymorph) and a doublet at  $2\theta = 17.1^\circ$  and  $18.1^\circ$  (A polymorph) (Mutungi, Passauer, Onyango, Jaros, & Rohm, 2012).

The content of amylose also has an important effect on the crystallinity of the starch. The degree of crystallinity will affect several functional properties and biochemical characteristics (Cheetham & Tao, 1998). In spite of some authors relate the amylose content to crystallinity of starches, other parameters, such as size of the crystals, amount of crystalline regions (influenced by the content and chain length of amylopectin), orientation of the double helices within the crystalline domains and the extent of interaction between double helices are also important to determine the crystallinity degree (Ambigaipalan et al., 2011).

The crystallinity degree calculated for *S. lycocarpum* starch was 38%. This value is higher than those observed for a typical A-polymorph and lower than B-type starches, which gives evidence of the mixed character of the *S. lycocarpum* starch.

### 3.2.8. Differential scanning calorimetry (DSC)

When starch is heated in the presence of enough water, its crystalline organization decomposes. This molecular disordering is called gelatinization, an endothermic phenomenon frequently accessed by using differential scanning calorimetry (DSC) (Beninca

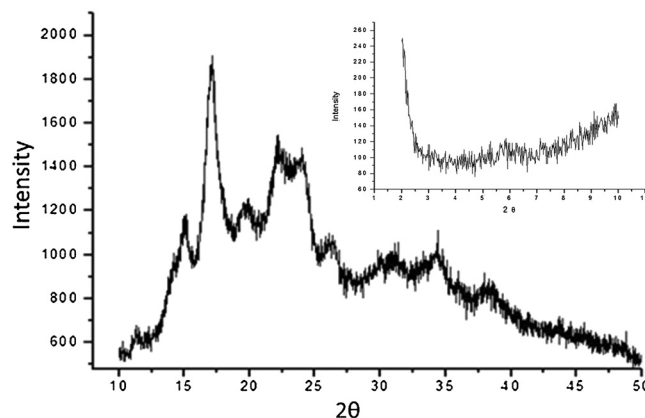
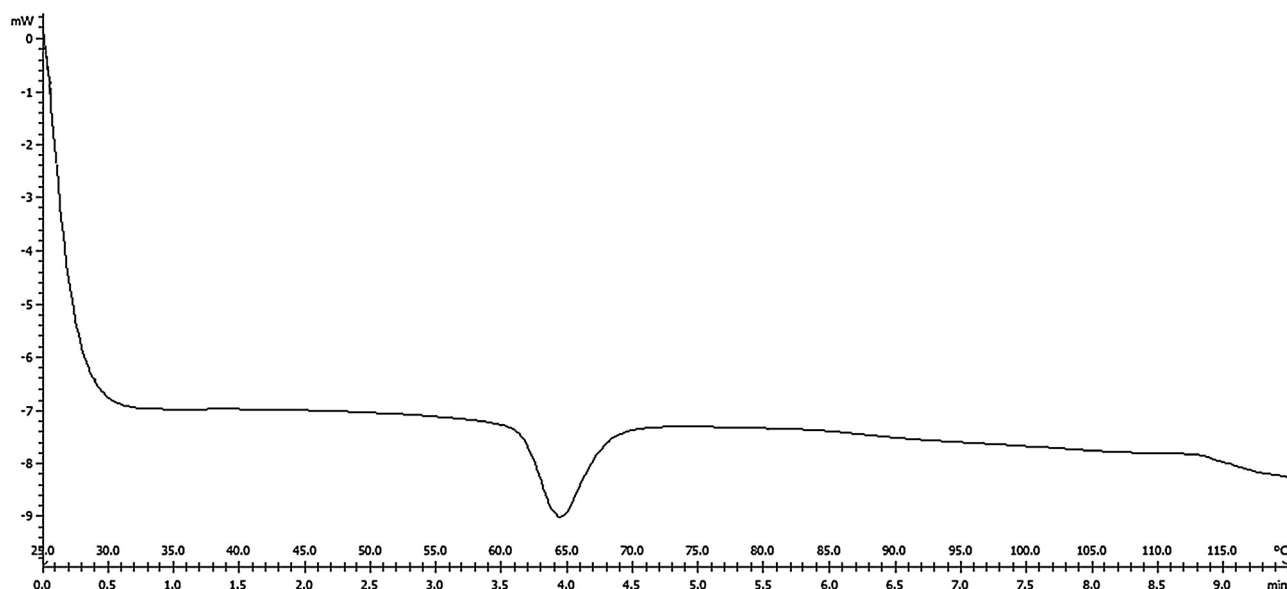


Fig. 4. X-ray diffraction spectra of the *Solanum lycocarpum* starch.

**Table 2**

Amylose content, endothermic transition temperatures ( $T_o$ ,  $T_p$  and  $T_c$ ), gelatinization temperature ranges ( $\Delta T$ ), enthalpy changes ( $\Delta H$ ) and relative crystallinity of starches from different sources.

| Starch source                | Amylose (%) | $T_o$ (°C) | $T_p$ | $T_c$ | $\Delta T$ | $\Delta H$ (J g <sup>-1</sup> ) | Relative crystallinity (%) |
|------------------------------|-------------|------------|-------|-------|------------|---------------------------------|----------------------------|
| <i>S. Lycocarpum</i>         | 38          | 61.25      | 64.5  | 67.55 | 6.3        | 13.21                           | 34.66                      |
| Potato (Yusuph et al., 2003) | 28.7        | 59.1       | 63.2  | 70.3  | 11.1       | 18.6                            | 26                         |
| Cassava (Ma et al., 2010)    | 20.8        | 50.3       | 56.2  | 66.7  | 16.4       | 7.89                            | 38.33                      |
| Cassava (Ma et al., 2010)    | 30.3        | 53.7       | 60.9  | 68.8  | 15.1       | 7.55                            | 43.1                       |
| Cassava (Ma et al., 2010)    | 31.8        | 58.2       | 65.8  | 73.1  | 14.9       | 8.42                            | 36.71                      |
| Barley (Tang et al., 2002)   | 25.9        | 55.6       | n.d.  | 71.7  | 16.1       | 7.4–8.5                         | 20.3–23.9                  |

**Fig. 5.** DSC curve obtained for native starch from *Solanum lycocarpum*.

et al., 2013). The transition from an ordered to a disordered phase occurs over a temperature range characteristic of the starch source. Therefore, DCS is an important measure to understand the chemical composition of starches and, therefore, its potential applications.

The DSC curves of *S. lycocarpum* starch are shown in Fig. 5. The thermogram presents the pattern characteristic of starches with a single endothermic peak of energy around 65 °C.

Table 2 summarizes the endothermic transition temperatures ( $T_o$ ,  $T_p$  and  $T_c$ ), gelatinization temperature ranges ( $\Delta T$ ) and enthalpy changes ( $\Delta H$ ) of starches from different sources (Ma, Chang, Zheng, Yu, & Ma, 2010; Tang, Watanabe, & Mistunaga, 2002; Yusuph, Tester, Ansell, & Snape, 2003). These parameters are influenced by the molecular architecture of the crystalline region mainly composed by short amylopectin chain. According to Noda, Takahata, Sato, Ikoma, & Mochida (1996), low values of  $T_o$ ,  $T_p$  and  $T_c$  and  $\Delta H$  reflects the presence of abundant short amylopectin chains. Observing data showed in Table 2, *S. lycocarpum* starch probably presents relatively high amounts of short amylopectin chain, since  $\Delta H$  value is the second higher, lower than potato only. According to Tester and Morrison (1990),  $\Delta H$  reflects the overall crystallinity of amylopectin defined as the quality and amount of starch crystallites. In this sense, the high  $\Delta H$  value (13.21 J g<sup>-1</sup>) of *S. lycocarpum* starch is in close agreement with its crystallinity degree (38%). In addition, the low  $\Delta T$  value (6.3 °C) for the *S. lycocarpum* starch is an important result, since low  $\Delta T$  values are related to high homogeneity and purity of the extracted material.

#### 4. Conclusion

In this study, some structural and physicochemical properties of *S. lycocarpum* starch were determined. The extraction resulted in

51% yield of a highly pure starch. The starch's granules presented a unique architecture with hemispherical units composed of two or three subunits. The main chemical groups from polysaccharides were present in *S. lycocarpum* starch as evidenced by FT-IR and NMR analysis. *S. lycocarpum* starch presented viscosity of 3515 mPa s, and estimated molecular weight of 645.69 kDa. Finally, the *S. lycocarpum* starch was classified as a B-type crystal with high amylose content (34.66%) and high degree of crystallinity (38%). DSC analysis indicates that *S. lycocarpum* starch has considerable amount of short amylopectin chains and high degree of homogeneity. These results make the *S. lycocarpum* fruit a very promising source of starch for biotechnological applications.

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