Food Chemistry 143 (2014) 440–445

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Chemical, morphological and functional properties of Brazilian jackfruit (*Artocarpus heterophyllus* L.) seeds starch

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ARTICLE INFO

Article history:
Received 28 January 2013
Received in revised form 30 July 2013
Accepted 1 August 2013
Available online 9 August 2013

Keywords:
Jackfruit seed
Starch
Functional properties

ABSTRACT

Starches used in food industry are extracted from roots, tubers and cereals. Seeds of jackfruit are abundant and contain high amounts of starch. They are discarded during the fruit processing or consumption and can be an alternative source of starch. The starch was extract from the jackfruit seeds and characterised to chemical, morphological and functional properties. Soft and hard jackfruit seeds showed starch content of 92.8% and 94.5%, respectively. Starch granules showed round and bell shape and some irregular cuts on their surface with type-A crystallinity pattern, similar to cereals starches. The swelling power and solubility of jackfruit starch increased with increasing temperature, showing opaque pastes. The soft seeds starch showed initial and final gelatinisation temperature of 36 °C and 56 °C, respectively; while hard seeds starch presented initial gelatinisation at 40 °C and final at 61 °C. These results suggest that the Brazilian jackfruit seeds starches could be used in food products.

1. Introduction

Jackfruit (*Artocarpus heterophyllus* L.) is a shrub belonging to the family Moraceae and is widely distributed in tropical countries such as Brazil, Thailand, Indonesia, India, the Philippines and Malaysia (Chowdhury, Raman, & Mian, 1997). Due to its spontaneous proliferation in warmer regions (specifically Brazil), it is now cultivated throughout the Amazon region and tropical coast of Brazil, from the states of Para down to Rio de Janeiro (Souza et al., 2009). Jackfruit are composed of several berries of yellow pulp and brown seeds encased in a hard shell and are rich in carbohydrates, complex B vitamins and minerals. However, only 15–20% of the fruit is used as food, which can be cooked, baked or roasted on coals (Silva, Jordão Filho, Ribeiro, & Silva, 2007).

The berries are eaten fresh or processed in the form of jams, compotes, frozen fruit pulps, juices and soft drinks. Their consistency can be slightly hard or completely soft, hence the distinction of two varieties popularly known as "soft jackfruit" and "hard jackfruit" (Silva et al., 2007). Jackfruit seeds are from 2 to 4 cm long, and a fruit can contain from 100 to 500 seeds, which represent 8–15% of the total fruit weight. The seeds usually are consumed roasted, boiled, steamed, and are eaten as a snack. However, fresh seeds have short shelf-life. The addition of jackfruit seed flour in the preparation of biscuits, sweets and breads has been investigated as an alternative use of this by-product (Aldana et al., 2011; Bobbio, El-Dash, Bobbio, & Rodrigues, 1978; Mukprasit & Sajjaanantakul, 2004).

Starch is widely distributed in various plant species as a reserve carbohydrate and is abundant in cereal grains, legumes, tubers and immature fruits (Lajolo & Menezes, 2006). It consists of two macromolecules: amylose (20–30%) and amylopectin (70–80%), which are associated with each other by hydrogen bonds (Singh, Singh, Kaur, Sodhi, & Gill, 2003). The proportions in which these structures appear differ in relation to their botanical sources, varieties of the same species and even within the same variety, and according to the plant maturity level (Tester, Karkalas, & Qi, 2004). According to Vandeputte and Delcour (2004), shape (round, ovoid, or polyhedral), particle size (2–100 μm), and particle size distribution (unimodal, bimodal, or trimodal) of granules are characteristic of biological origin and are responsible for the technological properties and industrial applications (e.g., use as a thickener, stabiliser, or gelling agent) of starch in the food industry.

The functional properties of starch depend on the molecular structure composition of amylose and amylopectin and how they are arranged in starch granules, which plays an important role in food formulations. The consistency of starch paste depends on the gelatinisation degree and swelling power of the starch granule; the paste texture is determined by the viscoelastic deformation and depends on the strength of molecular bonds and amount of broken granules. The clarity of the paste or gel can vary from clear to opaque, and this property is related to light dispersion resulting

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0308-8146/$ - see front matter
http://dx.doi.org/10.1016/j.foodchem.2013.08.003

from the association of amylose and other components present in the starch (Karam, 2003).

The increasing demand for new products has imposed to food industry the use of starches with characteristics such as absence of syneresis, transparency, stability and solubility to cold, which added to the restrictions on the use of chemically modified starches have directed researches for new sources of native starches with characteristics physico-chemical differentiated.

The literature provides little information about the isolation and properties of starches from unconventional sources such as fruit seeds. Studies on the functional properties of starch extracted from these seeds, including jackfruit seeds, have been conducted to verify its applicability in food, pharmaceutics and other uses and to replace with less costs commercial sources of starch (Aldana et al., 2011; Bello-Perez et al., 2006; Lawal & Adebowale, 2005; Mukprasit & Sajjaanantakul, 2004). However, the jackfruit seeds could be found in soft and hard varieties, which have direct influences in properties of their starch. Still, climate and soil conditions, where the jackfruit is grown, could result in different chemical composition, consequently, have influence in functional properties (Aldana et al., 2011; Bello-Perez et al., 2006). The present study characterised for the first time starch extracted from two Brazilian jackfruit seeds varieties (hard and soft), focusing in the physicochemical, morphological and functional properties to determine its applicability in the food industry.

2. Materials and methods

2.1. Obtaining starch from hard and soft jackfruit seeds

Jackfruit seeds (*A. heterophyllus* L.) (soft and hard) were extracted from mature fruits purchased from a local market in João Pessoa city, Paraíba State, Brazil. The brown spermoderm covering the cotyledons was removed by immersing jackfruit seeds in a 5% sodium hydroxide solution, followed by washing with running water. The starch was extracted from the cotyledons.

The extraction of starch from hard and soft jackfruit seeds was conducted according to the slightly modified methodology of Loos, Hood, and Graham (1981). First, seeds were removed from pulp, peeled, cut into small pieces and allowed to soak for 24 h in a sodium metabisulphite solution (0.2%). Starch was extracted by grinding the raw material with sodium metabisulphite in a regular blender at low speed for 30 min. After homogenisation, the mixture was processed through a 200 mesh sieve (0.074 mm).

The samples were then decanted twice for 24 h, with resuspension in sodium metabisulphite and centrifugation at 5000 rpm/15 min between each decanting; the supernatants of both were discarded. The white starch residue was spread on a tray and dried in lyophiliser – Terroni Equipmentos LTDA (LS 3000, São Paulo, Brazil) at a temperature of −45 °C. The dried starch was sprayed and stored in a plastic container under refrigeration until use.

2.2. Chemical quality of seeds and starch from hard and soft jackfruit seeds

The chemical composition of seeds and starch from hard and soft jackfruit seeds was determined according to the methodology described in the AOAC (2012). Analyses of moisture were conducted by desiccation in an oven at 105 °C until a constant weight was achieved; total lipids by extraction with hexane in Soxhlet; ash by incineration in a muffle furnace at 550 °C; total protein by the Kjeldahl method (N × 6.25); and starch by acid hydrolysis followed by quantification by titration using Fehling reagents A and B.

2.3. Morphological characteristics of hard and soft jackfruit seed starch granules

2.3.1. Shape and size of granules

The shape of starch granules was analysed by a digital scanning electron microscope model LEO-1430. Starch dispersions were placed on double-sided tape and coated with gold (sputtering). The mean particle size was determined using an inverted optical microscope (Axiovert 25 Zeiss). Twenty fields were randomly selected and photographed and 10 granules from each field were measured (for a total of 200 granules).

2.3.2. X-ray diffraction

The X-ray diffraction diffractogram was obtained from starch in the powder form containing approximately 11% moisture. The interval of 2θ angles ranged from 4 ° to 60 ° in the X-ray Diffractometer (Model D5000, São Paulo, Brazil), at a rate of 1.2 °/min and operating at a power of 40 kV/20 mA. The diffractogram patterns were evaluated according to Zobel (1964).

2.4. Functional properties of hard and soft jackfruit seed starch

2.4.1. Swelling power and solubility

Swelling power and solubility were determined according to the method described by Leach, Mc Cowen, and Schoch (1959) by weighing 0.1 g of starch in previously weighed centrifuge tubes and adding 10 ml of distilled water. The suspension was stirred and placed in a water bath for 30 min at temperatures ranging from 55 °C to 95 °C, increasing 10 ° from time to time and centrifuging for 15 min at 3400*g*. A 5 ml aliquot was removed from the supernatant, placed in petri dishes and placed on the stove at 105 °C for 24 h to determine the weight of the solubilised starch. After the outer walls of the tubes were dried, the tubes were carefully weighed, and the swelling power and solubility were determined as follows:

$$\begin{aligned}\text{Swelling power} = {} & (\text{weight of tube} + \text{residue after centrifugation}) \\ & - (\text{weight of tube plus sample on dry basis})/\text{weight of sample}\end{aligned}$$

$$\begin{aligned}\text{Solubility\%} = {} & (\text{weight of plate with sample after evaporation}) \\ & - (\text{weight of plate}) \times 100\end{aligned}$$

2.4.2. Paste transparency

The paste transparency was determined as described by Craig, Maningat, Seib, and Hoseney (1989). The paste transparency was determined by placing the starch suspension (3% w.v. $^{-1}$) in deionised water. Transmittance (% T) was determined at 650 nm using a spectrophotometer (Coleman 33D Spectrometer). Samples were stored at 4 °C for 8 days, and transmittance readings were conducted every 24 h to monitor retrogradation.

2.4.3. Viscosity

Viscosity was determined using a rapid viscosity analyser RVA-4, with the aid of the Thermocline for Windows software version 2.3, Newport Scientific Pty, Ltd, and according to the N° 162 methodology proposed by the ICC using the *Standard* 1 profile (Lawal & Adebowale, 2005).

2.4.4. Gelatinisation

The gelatinisation parameters were determined by Differential Scanning Calorimetry (DSC) using a differential exploratory calorimeter (Shimadzu, model DSC 50, coupled to computer software) in a nitrogen atmosphere at a flow rate of 50 ml min^{-1}. For the preparation of the samples, 6 μL of distilled water were added to 2 mg of starch, sealed in tubes and weighed again; to provide the uniform distribution of water in starch the samples were maintained 24 h at room temperature before analysis. The scanning temperature ranged from 30 °C to 150 °C, and the heating rate was 10 °C min^{-1} (Lawal & Adebowale, 2005).

3. Results and discussion

3.1. Chemical quality of seeds and hard and soft jackfruit seed starch

The chemical composition of the starch content in the seeds showed protein (7.98% soft and 5.56% hard) and lipids levels (0.59% soft and 0.24% hard) similar to those reported by Silveira (2002) for protein (5.07% soft and 5.50% hard) and lipids (0.52% soft and 0.23% hard) in jackfruit preparation containing seeds and residue. The starch isolated from jackfruit seeds showed for soft and hard varieties, respectively, 2.75 ± 0.10 and 2.86 ± 0.10 of moisture, 0.37% of lipids (for both), 1.53% and 0.62% of protein and 0.16% and 0.07% of ash. The starch content in soft and hard jackfruit seeds were 92.8% and 94.5%, respectively, higher than the 81% first describes to jackfruit seeds starch (Aldana et al., 2011). These results are in accordance with minimum specifications required by Brazilian Legislation for commercial starches used in food industry, which allows up to 14% moisture and 0.5% ash and requires at least 80% starch (Brazil, 1987). Considering the higher starch content and low content of protein, fat and ash founded in two varieties of jackfruit seeds studied here, it could be hypothesised that the starch of Brazilian jackfruit seeds could be employed in foods formulations, since these are characteristics of the starches of great quality (Franco et al., 2001).

Early study (Aldana et al. 2011) conducted with jackfruit seeds grown in México, reported high protein content (ca. 22%) and less amounts of starch to seeds at different stages of fruit maturity and ripeness, when compared to amounts detected in the present study. However, variations in chemical constitution of seeds could be related to soil and climate conditions from the region where the fruit was grown and the higher content of starch could be a marker of the jackfruit seeds cultivated in Northeast of Brazil.

3.2. Morphological characterisation of hard and soft jackfruit seed starch granules

The scanning electron microscopy analysis showed granules with round and bell shapes and some irregular shapes showing cuts in their surface, which appear to be characteristic of these starches (Fig. 1). The results shown here are consistent with those observed by Tulyathan, Tananuwong, Songjinda, and Jaiboon (2002) for native starch from jackfruit seeds grown in Asia. The average size of starch granules analysed by the optical microscope were 6–11 µm for the soft and 6–13 µm for the hard variety, do not show differences related to size between the seeds. Bobbio et al. (1978) reported that jackfruit seed starch has round or bell shapes, ranging in size from 7 to 11 µm, similar to results of the present study. Tongdang (2008) studied certain properties of starch extracted from three fruit seeds grown in Thailand and found the following results: Durian seed starch (*Durio zibethinus L*/Murr) showed polygonal shapes similar to rice starch granules with an average size of 4.43; Chempedak seed starch (*Artocarpus integer*) and jackfruit seed starch (*A. heterophyllus* L.) showed similar semi-oval or bell shapes but differed in size; Chempedak starch showed an average granule size of 6.47 µm and, in jackfruit seeds, granules with a mean size of 7.75 µm. These results suggest that the average size and shapes observed for starches in the present study are typical of the jackfruit seeds, growing around the world.

Jackfruit seed starch of both varieties analysed (soft and hard seeds) showed similar XRD patterns. Due to the partial crystallinity

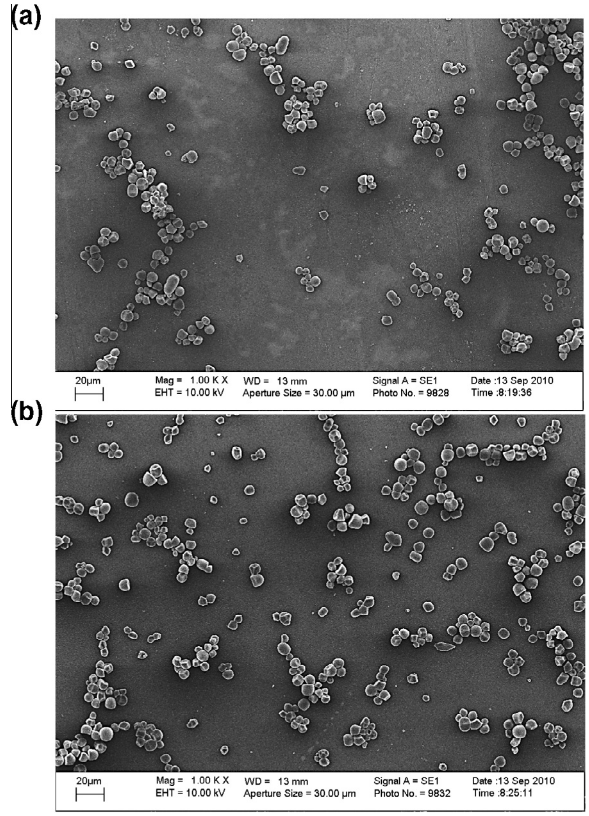

Fig. 1. Micrographs obtained through SEM in 1000× resolution of jackfruit seed starch for soft (a) and hard (b) varieties.

of starch granules, they provide specific X-ray diffraction patterns, which vary according to the vegetal source. Pattern A is characteristic of cereals, pattern B of tubers, fruit, corn with high amylose content and retrograded starches, and pattern C is regarded as a mixture of patterns A and B, which is characteristic of starch from legumes (Bello-Perez et al., 2006; Biliaderis, 1992). The X-ray diffractogram shown in Fig. 2 indicates a type-A crystallinity pattern, with peaks of higher intensity in 2θ at approximately 15.1 °, 17.18 ° and 23.64 ° and no peak in 2θ at 5 °. According to Zobel (1964), type-A starches show strong signals in 2θ equal to 15.3 °, 17.1 °, 18.2 ° and 23.5 °, while for type-B starches, strong bands appear in 5.6 °, 14.4 °, 17.2 °, 22.2 ° and 24 ° and for type-C starches, the signals are stronger in 5.6 °, 15.3 °, 17.3 ° and 23.5 °. Tulyathan et al. (2002) also reported the absence of a peak in angle 2θ (equal to 5 °) and characterises jackfruit seed starch as type-A, which has in structure less space to water molecules.

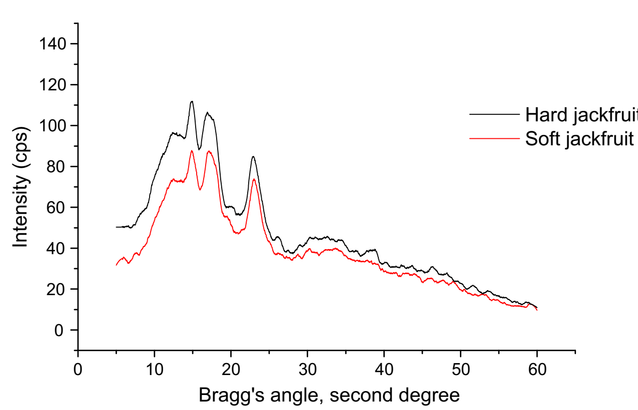

Fig. 2. X-ray diffractogram of soft and hard jackfruit seed starch.

3.3. Functional properties of hard and soft jackfruit seed starch

The swelling power (SP) and solubility index (SI) were directly correlated with increasing temperature (Figs. 3 and 4). The starch from the jackfruit varieties studied did not show large variations in SP and SI until reaching temperatures of 75 °C; however, above this temperature, a significant increase in swelling and solubility index values was observed. The increase in temperature causes rupture of intermolecular bonding (hydrogen interactions) and the opening of the chains allows the entry of water molecules; over the temperature range of gelatinisation, the starch granule has only limited swelling which a quantity of carbohydrate is solubilized, but as the temperature increases above the temperature gelatinisation, there is an increase power swelling (Agunbiade & Long, 1999).

The profiles observed here are consistent with those related in previous studies involving jackfruit seed starches, which showed the increase in swelling and solubility index values above 75 °C (Aldana et al., 2011; Tongdang, 2008).

The swelling of granules occurs simultaneously with the loss of birefringence and before solubilisation. The SP is generally influenced by the bond strength between molecules and by the molecular structure of amylopectin. Low SP can be attributed to the presence of various crystals formed by the association between long chains of amylopectin. Increased crystallisation results in higher stability of granules, which reduces the swelling capacity (Singh et al., 2003).

The gel of jackfruit seed starch showed lower transmittance with opaque pastes. In starches from both varieties, transmittance (%) decreased throughout the storage period. The tendency of transparency reduction of starch pastes stored under refrigeration is mainly related to their retrogradation. In general, starches with increased retrogradation resistance do not reduce the clarity of their pastes (Stahl, 2003). According to Craig et al. (1989), opaque pastes show more organised granular structure, with greater association between chains, which hinders the passage of light. Starches with higher amylose content and high retrogradation show opaque and firmer gels (Silva et al., 2006). The characteristics observed for the pastes formed revealed that the jackfruit seeds starches may be interesting to use in formulation which do not require transparency, such as soups, sauces and creams.

Viscosity is one of the most important properties of starchy materials. The viscosity curve represents the behaviour of the starch during heating and allows evaluation of the characteristics of the paste formed by structural modifications of starch molecules and the tendency for retrogradation to occur during cooling

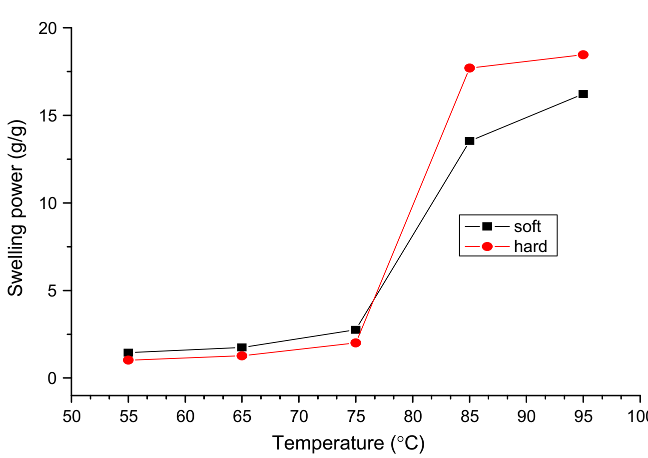

Fig. 3. Swelling power (g/g) of soft and hard jackfruit seed starch.

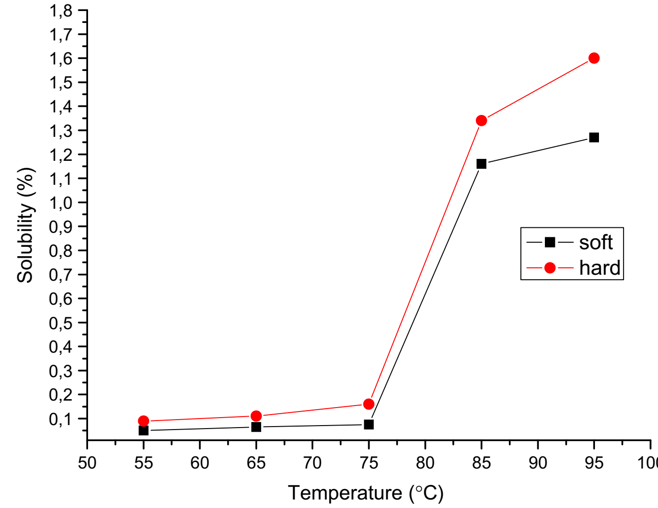

Fig. 4. Solubility (%) of soft and hard jackfruit seed starch.

(Lustosa, Leonel, Leite, Franco, & Mischan, 2009). The viscoamylograph curves obtained from a Rapid Visco Analyser (RVA) of soft and hard jackfruit seed starch showed that increasing temperatures lead to starch gelatinisation, which increased viscosity due to the swelling of starch granules. The temperature at which granules begin to swell is called the pasting temperature (i.e., the initial gelatinisation temperature when the viscosity curve starts), which was higher for soft jackfruit seed starch (83.15 °C) than hard jackfruit (81.60 °C). Rengsutthi and Charoenrein (2011) studied jackfruit seed starch and found a pasting temperature of 81.58 °C, which was similar to that obtained in this study for hard jackfruit seed starch. The maximum viscosity achieved for hard jackfruit seed starch was higher (2616 cP) than that for soft jackfruit (1716 cP). This result could be related to the higher protein content observed in soft jackfruit seed starch, when compared to the hard variety, which is negatively correlated with maximum viscosity (El-Saied, Ahmed, Roushdi, & El-Attar, 1979).

During the period of constant temperature (95 °C) while stirring, the granules begin to dissociate, and the solubilisation of amylose molecules causes a decrease in viscosity. The difference between maximum and minimum viscosity is called the Breakdown, which represents the resistance of starch to mechanical agitation. During this resistance period, it is possible to evaluate the starch stability at high temperatures, whose granules are broken under mechanical stirring (Thomas & Atwell, 2008). Soft jackfruit seed starch showed the lowest breakdown value (672 cP); therefore, this starch can be considered more stable (resistant to heating) with reduced breakdown when compared to hard jackfruit seed starch (1383 cP).

The final viscosity of the starch under study was 1998 cP (soft variety) and 3236 cP (hard variety), which is considered low when compared to results reported by Muccilo (2009) for native pinion starch (5072.5 cP) and native corn starch (4534.5 cps). Tongdang (2008) studied the functional properties of starches extracted from fruit seeds and found the following final viscosity results: chempedak (4088.19 cP); Jackfruit (3853.11 cP), Durian (4114.76 cP) and Mung bean (4232.05 cP). Considering these aspects a product made with starch from Brazilian jackfruit seeds is a product less viscous than one formulated with the starches mentioned above.

The setback (tendency for retrogradation) for soft jackfruit seed starch was significantly lower (954 cP) compared to hard jackfruit (2002 cP). Muccilo (2009) studied native pinion starch and found a setback (2.275 cP) higher than that reported in this study. Yuan, Zhan, Daí, and Yu (2007) reported that higher setback values are

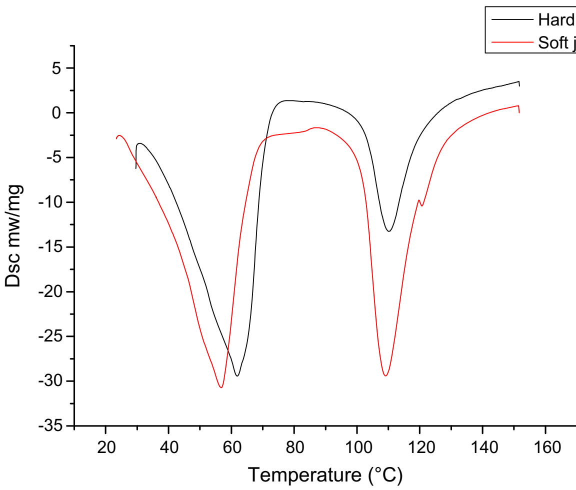

Fig. 5. Thermograms obtained through DSC of soft and hard jackfruit seed starches.

found in starches whose granules have larger diameter due to the increased fragility found in larger granules, which agrees with the results observed in the analysis of the size of granules, indicating lower values for soft jackfruit (6–13 μm).

3.4. Functional properties of hard and soft jackfruit seed starch

Fig. 5 shows the thermogram obtained by the differential scanning calorimetry analysis (DSC) for soft and hard jackfruit seed starch. The parameters were initial gelatinisation temperature (T_o = 36.0 °C and 40.0 °C), endothermic peak temperature (T_p = 56.0 °C and 61.0 °C), final temperature (T_c = 65.0 °C and 70.0 °C), gelatinisation range (T_c–T_o = 29.0 °C and 30.0 °C) and gelatinisation enthalpy (ΔH_{gel} = 462.84 J g $^{-1}$ and 480.05 J g $^{-1}$).

The endothermic peak temperature of soft jackfruit seed starch was lower (56 °C) than the hard variety (61 °C). Mukprasit and Sajjaanantakul (2004) reported a peak temperature value of 66.8 °C for jackfruit seed starch showed that this characteristic, closely related to the functional properties of the starch varying between the seeds of the jackfruit varieties.

Comparing the initial gelatinisation temperature (T_0) obtained through DSC with paste temperatures using RVA, observed lower values by DSC for the formation of starch pastes than the RVA. However, the same was observed by Peroni (2003) for starch obtained from cassava and other plant species, which had paste temperatures higher than those obtained by DSC. According to Perez, Breene, and Bahnassey (1998), the paste temperature obtained using RVA was higher due to the reduced sensitivity in detecting the first increases in the viscosity of starch pastes, unlike the initial gelatinisation temperature, which is detected when the first granules begin to become disorganised. DSC values are more accurate, while RVA values have a greater temperature range. In the thermogram of hard and soft jackfruit seed starches, two peaks were observed; the first relates to gelatinisation, which was analysed to obtain data; the second relates to the gelatinisation of amylose, which was complexed with lipids within the starch and during liquid evaporation.

4. Conclusions

Jackfruit seeds (*A. heterophyllus* L.), both soft and hard varieties, have shown high amounts of starch with the potential to be used in the food industry as a raw material. The starch granules are similar to those present in cereals (in terms of a crystallinity standard), and the results of functional property analyses indicate that the use of these non-common starches in food systems could be an interesting alternative to use the jackfruit seeds, which are generally discarded as waste.

Acknowledgements

We would like to thank CAPES and CNPQ for financial support and the scholarships awarded.

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