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Original Article

Sorghum starch as depressant in mineral flotation: part 1 – extraction and characterization



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ABSTRACT

The mineral industry is constantly searching for new and sustainable innovations, in order to improve its results and processes. In this context, sorghum (*Sorghum bicolor* (L.) Moench), a widely farmed crop, was characterized and tested as an alternative depressant in mineral flotation. Starch was extracted from sorghum flour produced with grains cultivated in Brazil, resulting in an extraction yield of approximately 26%. Under polarized light microscopy, starch showed Maltese crosses, indicating that the isolation method used produced intact starch granules. SEM images indicated that the starch granules were oval or semi-spherical, with the occasional presence of broken granules and surface pores. Granule size distribution ranged from 5.5 to 30 μm with average size of 15.5 μm . With 25.5% of amylose content and relative crystallinity around 26%, the sorghum starch was classified as medium size A-type starch. FTIR spectroscopy results confirmed the presence of polysaccharide characteristic peaks, as expected. Thermal properties were measured by DCS, with results similar to cornstarch. Rheological characterization indicated a pasting temperature of 67.1 °C and viscosity peak of 2596 cP. The characterization results indicate a high potential for sorghum as depressant in mineral flotation being a feasible option for cornstarch.

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1. Introduction

In Brazil, cornstarch is the most widely used depressant, mainly because its effectiveness in different types of minerals. According to Geller [1], a production of 93 mmt of corn in 2017 is expected to meet an internal demand of 59 mmt. If the exports remain the same as the previous year, Brazil will need to import at least 0.6 mmt of corn. This scenario is not favourable for the mineral industry. In order to reduce production cost, cornstarch usage has been replaced by low quality cornstarch and, in some cases, by coproducts of the cornstarch processing.

Being the fifth most important cereal grain, sorghum (*Sorghum bicolor* (L.) Moench) is cultivated worldwide for animal feed and human food [2]. Sorghum is less demanding on water and soil fertility than other cereals and has a short vegetative cycle (varying from 90 to 130 days), ideal for off-season production associated with other crops, such as sugarcane, corn, or soy. The same planting, cultivating, and harvesting equipment used for other cereals can be used, although the farming can be conducted manually with good adaptation to systems usually used by small producers. Nowadays sorghum is cultivated in Brazil only for animal food, despite the fact of its possible usefulness in different food products. The development of sorghum farming in Brazil occurred primarily in the southeast, south, and central-west regions. The price per bag (60 kg) of corn was sold for US\$ 7.91, while a bag of sorghum was sold for US\$ 6.02 (approximately 24% cheaper) on February 23rd, 2018 (for the producer municipality of Itumbiara, GO, and exchange rate US\$/R\$ of 1/3.2486).

The aim of this study was to evaluate the efficiency of a new depressant for mineral flotation, as an alternative for cornstarch. In this first article, the production and characterization of sorghum flour and starch are presented. Microflotation tests with high purity samples of quartz and hematite were performed with a modified Hallimond tube in order to confirm the sorghum potential as a depressant. The depressant preparation methodology (alkaline gelatinization with NaOH) and the flotation results are presented in the article “Sorghum starch as depressant in mineral flotation: Part 2 – Flotation tests”.

2. Materials and methods

Graniferous sorghum grains were farmed in Ipameri, Goiás, Brazil and kindly provided by the Brazilian company, Agroceres. Straw residues and other impurities were removed by dry sieving. Grains were then oven dried at 35 °C for 30 h to remove residual moisture. Sorghum flour was produced by the comminution of the dried grains using two cereal mills operating in series. The methodology proposed by Rupollo et al. [3] was used to extract sorghum starch from the flour, with minor modifications. A suspension of 500 g of sorghum flour and 2.0 L of sodium bisulphite solution (0.16%) was left in the refrigerator (at 4 °C) for 24 h. The supernatant liquid was drained after the starch decantation. To the precipitate was added 600 mL of distilled water. Using an industrial blender the solution was homogenized for 5 min. Sieves (38, 75 and 355 μm) were used to remove the coarse fraction. Particles under 38 μm

were centrifuged using a micro processed centrifuge FANEM Excelsa II, model 206-BL at 3600 rpm for 5 min. Three segregated phases were produced after the centrifugation. From the top to the bottom: liquid, non-white solids (light brown), and white solids (amylaceous fraction). The liquid fraction was drained and the non-white solids were manually removed using a stainless steel spatula. The amylaceous fraction were rinsed with distilled water. The centrifugation process was repeated until the extinction of the non-white solids phase (between 5 to 8 cycles). The extracted sorghum starch was dried in a forced air oven at 40 °C for 12 h. The starch extraction yield was calculated as the percentage of starch extracted mass in relation to the flour mass (both in dry basis).

Granules morphology was analyzed using a SEM JEOL JSM-6610 at the laboratory Labmic from Federal University of Goiás (UFG). Samples were fixed with carbon tape on aluminium supports and then metallized with a thin gold layer (350 Å). Sorghum granule size distribution was determined by dry measurements using a HELOS laser diffraction particle size analyser from Sympatec coupled with a RODOS dry disperser at TU Clausthal. Amylose content was measured according to the method 61-03.01 and ash content according to the AACCI method n° 08-21.01 [4]. The Bligh and Dyer [5] method was used to measure the lipid quantification and the method proposed by Bradford [6] was used for protein quantification. The swelling power and solubility index were determined as described by Leach et al. [7].

Sorghum starch birefringence was analyzed under polarized light at three different temperatures (25, 50, and 70 °C) using a Carl Zeiss Axioskop MC-80 microscope. FTIR spectrum was acquired using a PerkinElmer spectrometer (resolution of 1 cm^{-1} and range from 450 to 4000 cm^{-1}). Samples were prepared using the KBr pellet method. Relative crystallinity was calculated using the FTIR results and the method proposed by Sun et al. [8].

A Shimadzu XRD-6000 X-ray diffractometer with Cu-K radiation tube (40 kV and 100 mA) was used to obtain the sorghum starch X-ray pattern. The angular scan (2θ) ranged from 5° to 50°. Samples were dried in an oven for 24 h and kept in a desiccator to avoid moisture interference. Starch crystallinity from the XRD results was calculated using two different methodologies: Brückner [9] and Frost et al. [10].

Sorghum starch thermal properties were measured using a Perkin Elmer DSC-7 analyzer and the properties measured were: the gelatinization enthalpy, onset temperature, gelatinization endothermic peak temperature, and final temperature of gelatinization. Samples were weighted directly in aluminium pans (2.0 mg, db). Deionized water (8.0 μL) was added to the pans, which were then hermetically sealed and kept at room temperature for 12 h for thermal equilibrium. The pans were scanned from 25 to 116 °C (10 °C/min). Rheological properties were measured using a Petern RVA 4500 viscometer, based on the methodology proposed by Olayinka et al. [11]. All tests were performed in triplicate.

3. Results and discussion

The extracted sorghum starch was an inodorous white powder. The extraction yield was $25.82 \pm 4.08\%$. Pascoal et al. [12]

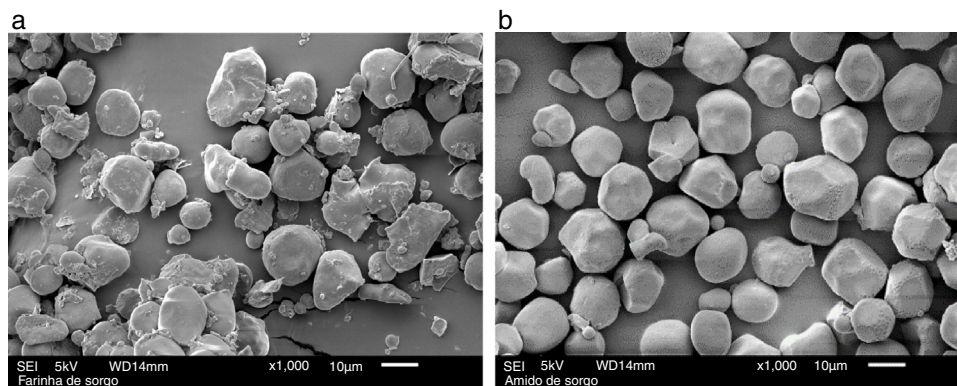


Fig. 1 – SEM images for sorghum (a) flour and (b) starch.

described similar results for cornstarch. SEM results shows the presence of the crushed sorghum's shell in the flour (Fig. 1a) with an irregular polyhedral shape. Since the flour was the milled cereal, the presence of other parts of the cereal was expected. Although the predominant shape of sorghum starch granules was oval or semi-spherical, some grains with varied shapes could be seen (Fig. 1b). BeMiller and Whistler [13] reported similar results for large sorghum granules with polygonal and spherical shapes. Regarding the grains texture, the sorghum starch had smooth surface, with an occasional presence of surface pores. Benmoussa et al. [14] and Olayinka et al. [11] found similar results.

Fig. 2 shows sorghum granule size distribution. Sorghum flour granules ranged from 3.7 to 600 μm (210 μm on average). The sorghum starch was classified as medium size [15] with granules ranging from 5.5 to 30 μm (15.5 μm on average). Wang et al. [16] found similar results for cornstarch, with granules ranging from 6 to 17 μm (11.6 μm on average).

Starch is a polysaccharide carbohydrate mainly composed by amylopectin and amylose, with an average amylose/amylopectin ratio of 0.33 in yellow corn varieties [17]. These two polysaccharides are the primarily responsible for the starch depressant action. Amylose content in sorghum starch was $25.455 \pm 0.787\%$, with an amylose/amylopectin ratio of 0.35, same value found by Zobel [18]. Alves et al. [19] found 16.4% of amylose in sweet sorghum starch. A higher amylose content was found for cornstarch (27.8%) [20] and for

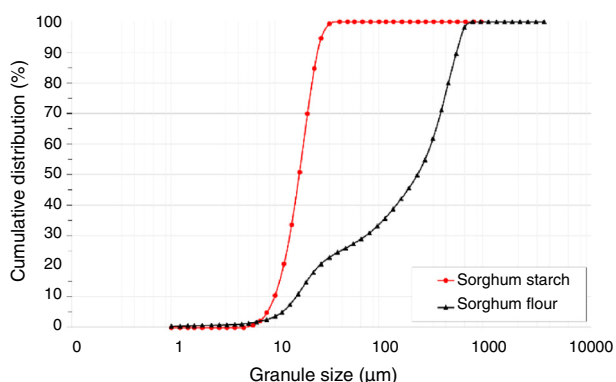


Fig. 2 – Sorghum granules size distribution.

native sorghum starch (37.20%) [21]. Cassava starch, another important starch source for the mineral industry, has 25.97% of amylose with the same amylose/amylopectin ratio of sorghum [22]. As expected, sorghum starch showed a higher amylose content than sorghum flour ($19.182 \pm 0.315\%$ with ratio of 0.26). Amylopectin showed better results in hematite flotation than amylose [23]. For apatite flotation, the same authors showed that pure amylose has lower depressant action, followed by amylopectin and starch.

Lipids present in the starch are harmful in mineral flotation, since they can act as an anti-froth agent. The lipid content in sorghum starch ($0.495 \pm 0.175\%$) was lower than verified for cornstarch (0.79%) and higher than potato starch (0.13%) [24]. Sorghum flour presented the highest lipid content ($3.277 \pm 0.425\%$). According to BeMiller and Whistler [13], the lipid content in sorghum starch should be lower than in cornstarch, since sorghum germ constitutes a smaller kernel proportion than in corn. Lipid content in Indian varieties of sorghum ranged from 2.30 to 2.80% [25], almost 5 times higher than the Brazilian variety tested. According to Guimarães et al. [26], froth quality in the phosphate ore flotation is not affected when the starch oil content is lower than 4% because the collector used in this flotation system, normally ionized fatty acids soap species, is also a frothing agent. However, froth stability parameters, such as half-life time and induction time, cannot be neglected and must be studied for different flotation systems.

The protein amount in sorghum starch ($0.85 \pm 0.05\%$) was almost the same when compared to the cornstarch (0.86%) [27], but lower than in the sorghum flour ($1.86 \pm 0.02\%$). Protein ribbons were seen only in sorghum flour SEM images, corroborating this result. Additional test work is still required to investigate the influence of proteins in the mineral flotation. It appears that different flotation systems react differently to the protein presence. Peres and Correa [17] showed that zein act as an efficient hematite depressant, corroborating industrial observations. On the other hand, phosphate ore plants reported opposite results. Cornstarches commercialized as flotation depressant usually had proteins and oil in their composition [28].

Ash content in the sorghum starch ($0.121 \pm 0.005\%$) was higher than in cornstarch (0.09%) [24], but lower than cassava starch (0.34%) [22], potato starch (0.16%) [24], and sorghum

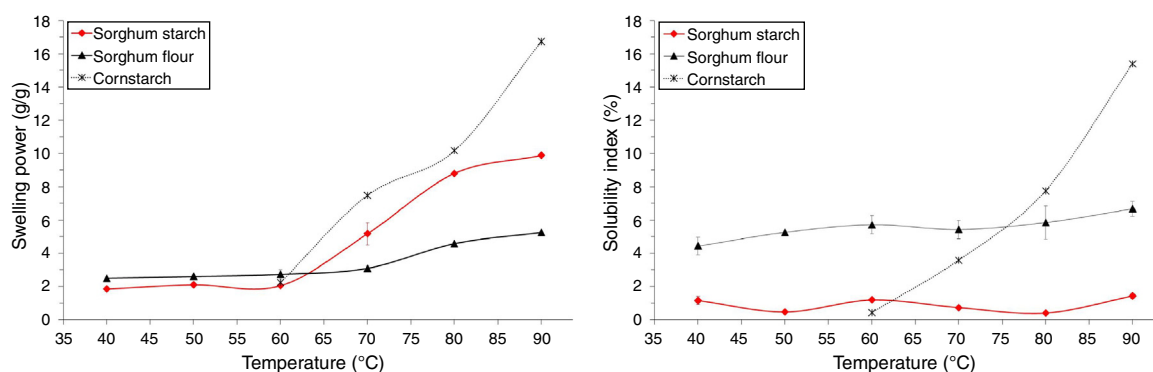


Fig. 3 – Variation of the (a) swelling power and (b) solubility index for sorghum (starch and flour) and cornstarch [29] with the temperature.

flour ($1.489 \pm 0.016\%$). Low ash content and protein amount lead to a high quality starch since the starch's quality is greatly influenced by these two, indicating that the adopted extraction process in laboratory scale was efficient in producing high purity starch.

The swelling power and solubility index of sorghum (flour and starch) are presented in Fig. 3. Cornstarch data were extract from literature [29]. Sorghum starch and flour showed similar results for the swelling power until 60 °C. For high temperatures, the considerable increase in starch swelling power can be explained by the high amount of amylose and amylopectin present in it [30]. Cornstarch showed similar behaviour of sorghum starch, with swelling power increasing with temperature. As expected, cornstarch presented high swelling power, which can be explained due to its higher amylose content. Sorghum flour solubility was higher than sorghum starch for all temperatures analyzed, which could be a direct result of the high amount of protein present in the flour. Claver et al. [31] found a similar behaviour for the swelling power and solubility index for sorghum starches. At high temperatures, cornstarch is more soluble than sorghum starch and flour. However, no difficult or differences were found during the sorghum homogenization in water.

The characteristic Maltese crosses for sorghum starch was observed (Fig. 4) under polarized light, indicating that the extraction method produced intact native starch granules. The starch gelatinization temperature was around 70 °C (Fig. 4c), since at this temperature, it was possible to notice the beginning of the Maltese crosses extinction.

The FTIR spectrum of sorghum starch is shown in Fig. 5a. The presence of O–H stretching vibration for intermolecular

H bonds was confirmed by the peak 3350 cm^{-1} . The peak at 2926 cm^{-1} indicates a stretching vibration of C–H bond from glucose. No alkene C–H was observed, since there were no characteristic peaks above 3000 cm^{-1} . The peak at 1648 cm^{-1} was attributed to H_2O bending vibrations. No C=O stretch was observed, since no strong peak around 1700 cm^{-1} was found. C–H bending vibrations were observed at 1465 (characteristic of CH_2 or CH_3) and 1373 cm^{-1} (characteristic of CH_3). The peak at 1244 cm^{-1} may be attributed to O–H bending vibration. Peaks at 1081 and 1158 cm^{-1} were attributed to C–O bond stretching of C–O–H in starch. The peak around 1016 cm^{-1} was assigned to C–O–H deformation vibration. Fig. 5b shows the crystalline area of the FTIR spectrum. The relative crystallinity of sorghum starch was 26.38%.

Sorghum starch XRD spectrum indicated a crystalline pattern compatible with A-type starches (Fig. 6a). Strong peaks at 15.22° and 23.08° and an unresolved doublet (17.18° and 17.90°) were observed. Similar results were found for sorghum [31] and sweet sorghum starches [19]. Relative crystallinity was 26.07% for the Brückner [9] method and 26.92% for the Frost et al. [10] method (Fig. 6b and c, respectively), which agree with the FTIR result. Several starches show relative crystallinity between 15–45%, with A-type ranging from 33–45%. The relatively high amylose/amylopectin ratio in the sorghum starch could explain its low crystallinity. According to Zobel [18], amylopectin is the main substance responsible for starch crystallinity.

Fig. 7 shows the sorghum starch thermal properties. The endothermic peak gelatinization temperature (or pasting temperature, T_p) for sorghum starch found by DCS (Fig. 7a) was 71.4°C . This result agrees with the Maltese crosses

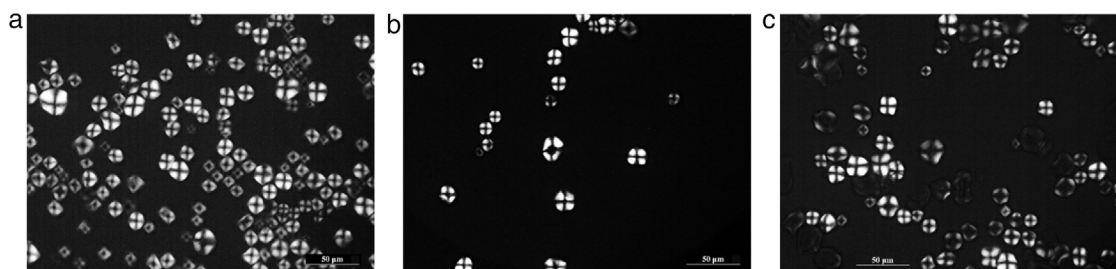


Fig. 4 – Polarized light microscopy of sorghum starch granules at (a) 25, (b) 50, and (c) 70 °C.

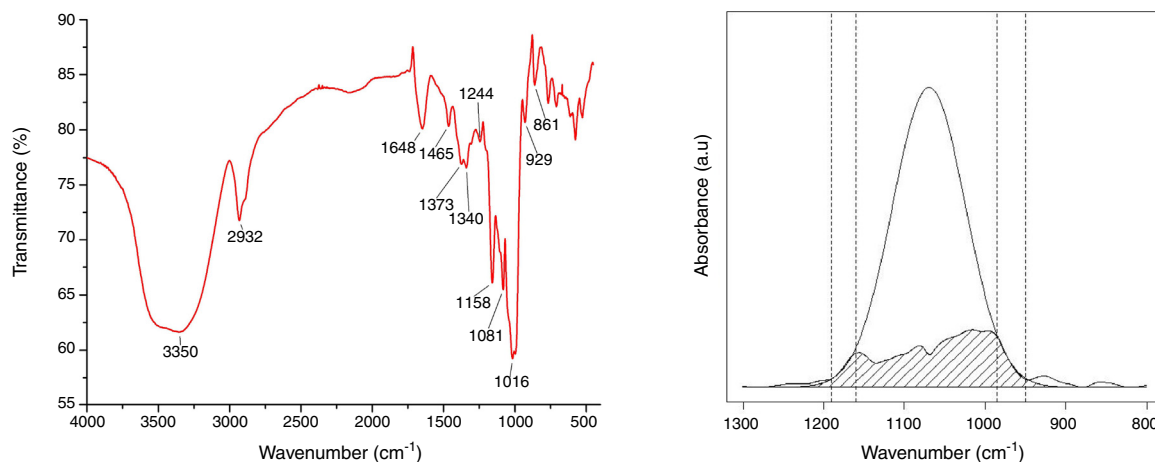


Fig. 5 – (a) Fourier-transform infrared spectrum and (b) crystalline area of sorghum starch.

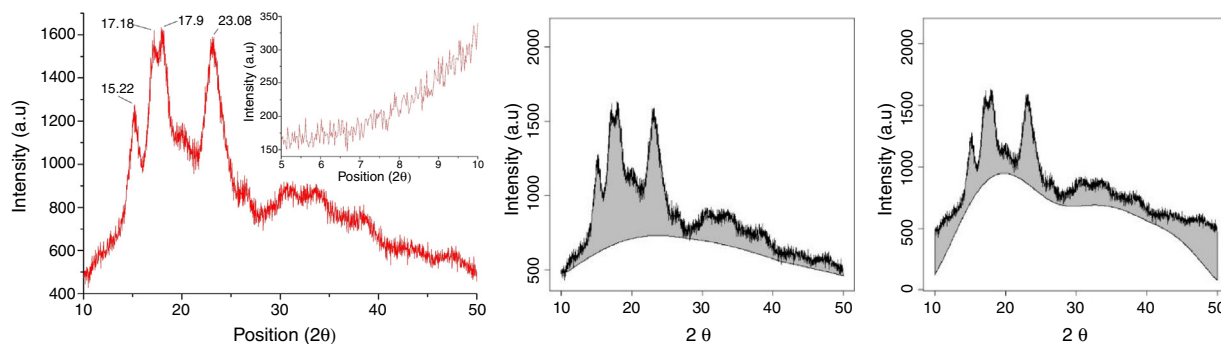


Fig. 6 – (a) Sorghum starch X-ray diffraction spectrum. Crystalline area of the XRD spectrum (b) Brückner and (c) Frost Method.

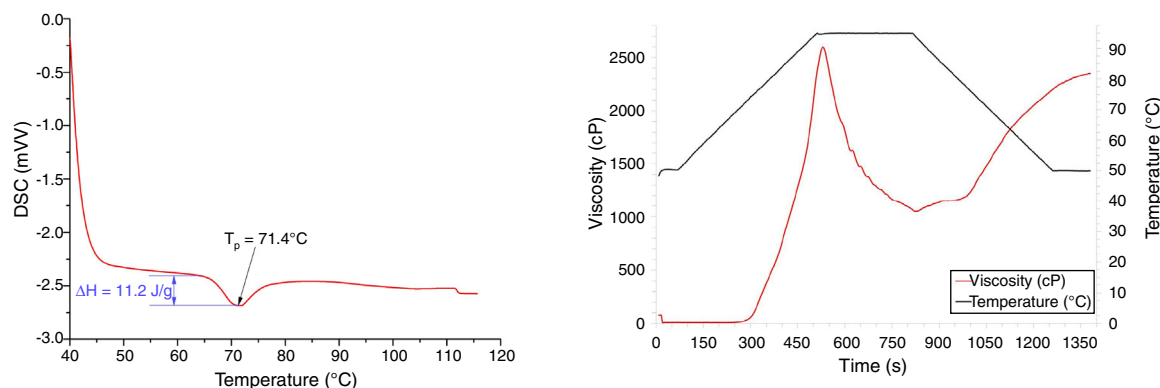


Fig. 7 – Sorghum starch thermal properties: (a) DSC and (b) RVA results.

extinction observed around 70 °C. The gelatinization enthalpy was 11.2 J/g, onset temperature 66.3 °C, and final temperature of gelatinization 82.3 °C. Similar results were obtained for sorghum [32], sweet sorghum [19], and corn [24,33], but different from cassava [34] and potato starch [24,35]. The pasting curve for sorghum starch is shown in Fig. 7b. The pasting temperature was 67.1 °C, confirming the onset temperature found by DSC (66.03 °C). Regarding the rheology, the viscosity peak was 2596 cP, trough viscosity 1053 cP (breakdown of

1543 cP), and the final viscosity 2350 cP (setback of 1297 cP). Sorghum starch presented shear thinning behaviour, since the viscosity breakdown ratio (BDR) was around 0.41. Dhiyal et al. [24] found similar viscosity profiles for cornstarches with BDR of 0.6. However, the authors found a higher pasting temperature (83.6 °C), considerably lower viscosity peak (1463 cP), but similar trough viscosity (956 cP). These results could be explained by the presence of single-helical complexes between amylose and lipid in the cornstarch. For the cassava

starch, Chandanasree et al. [22] found a pasting temperature of 71.43 °C, viscosity peak of 3995 cP, and trough viscosity of 1430 cP (BDR of 0.36). These results shows that sorghum starch gelatinization requires less energy than cornstarch, but results in a more viscous paste. The opposite happens when compared to cassava.

4. Conclusions

Starch was extracted and characterized from the grains of a Brazilian graniferous variety of *Sorghum bicolor* (L.) Moench, with an extraction yield of around 26%. Starch was classified as A-type regarding its XRD pattern, with low relative crystallinity, lipids, protein, and ash content. The relative crystallinity was confirmed by two different tests. As expected, the protein content of sorghum flour was 219% higher when compared with the sorghum starch. This result is due to the fact that the sorghum grain was integrally present in the flour and only comminution stages were performed in order to produce it.

The SEM analysis showed that the starch granules were oval or semi-spherical in shape, free of protein ribbons, and with occasional superficial pores. Sorghum starch was classified as medium size, once the granulometric analysis showed that granules ranged from 5.5 to 30 µm. DSC results show that gelatinization endothermic peak temperature for the sorghum starch was similar to that reported in literature for other cereals.

No major differences were found regarding the sorghum starch paste rheology when compared with corn or cassava starches, which could indicate that no changes regarding pumps or equipment would be required in the case of the adoption of the new depressant in the mineral industry. However, gelatinized starches behaves as non-Newtonian fluids, to whom rheological properties play a critical role in fluid flow. More test work is required in order to investigate how the sorghum starch paste viscosity behaves for a range of shear stress, similar to those applied in industrial mineral circuits.

No restriction was found regarding the sorghum starch lipid content and its use as a depressant. In fact, for more oil sensitive flotation systems, or when there are froth production or stabilization problems, the sorghum starch is more indicated than cornstarch. It is expected that sorghum starch would be a better hematite depressant than cornstarch, once cornstarch has 9.21% more amylose than sorghum starch and, consequently, less amylopectin.

Apart from the lipid and protein content, sorghum flour also presents itself as a possible depressant. Since the flour production cost has to be less than the starch cost, sorghum flour could be feasible in flotation systems such as phosphate rock flotation. The found results make the sorghum a very promising starch source as depressant in the mineral flotation process. The analyzed parameters were similar to cornstarch, the major depressant used nowadays in mineral flotation. The application of these products in flotation will be evaluated and the results will be presented in the next paper of this study – Part 2.

Conflicts of interest

The authors declare no conflicts of interest.

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