

# 1 Properties of different varieties of *Maranta arundinacea*'s starch

2  
3 Amanda Logato Cunha<sup>a</sup>, Luan Alberto Andrade<sup>b\*</sup>, Ana Caroline da Silva<sup>c</sup>, Joelma  
4 Pereira<sup>d</sup>

5  
6 **ABSTRACT:** The objective of this study was to analyze and compare arrowroot  
7 starch in the "seta" (AS) and "redonda" (AR) varieties by assessing their chemical,  
8 morphological, rheological, structural and thermal characteristics, and to suggest  
9 possible applications in the food industry. The analyzes of proximal composition,  
10 infrared spectroscopy, scanning electron microscopy, determination of paste clarity  
11 and properties, X-ray diffraction, and thermal properties have been conducted. The  
12 extracted starches present high purity, with the carbohydrate level above 97%. The  
13 two types of arrowroot starches have exhibited vulnerability to high temperatures,  
14 lower resistance to dissociation of hydrogen bonds, and high final viscosity (357.63  
15 RVU to AR and 432.46 RVU to AS). The characteristic size of the starch granules is  
16 in the range of 8 µm to 45 µm. In this study, there was a predominance of granules of  
17 approximately 30 µm, for both AR and AS, which can be considered large for  
18 granules. The granules predominant geometric shapes are oval and ellipsoid.  
19 Starches exhibited type-C crystallinity and initial, peak and final temperature values  
20 for gelatinization well below the temperatures found in literature. The two starches  
21 exhibited potential for use in the industry of instant foods, breakfast cereals, infant  
22 food, meat products, and bakery products. However, studies applying these starches  
23 in the aforementioned matrices need to be carried out to prove its use.

24 **KEYWORDS:** Arrowroot; Gelatinization; Paste properties; Paste clarity; Thermal  
25 properties.

## 26 27 1. INTRODUCTION

28 Starch is the main energy reserve carbohydrate in plants, and it is found in  
29 great amounts, especially in roots, tubercles and cereals. Beyond being a complement  
30 in man's diet, starch is also used as an ingredient or additive (Gutiérrez, Hernion-  
31 Julien, Álvarez & Álvarez, 2018) to enhance product production, presentation, and  
32 conservation in several industrial areas. In the food industry, starch is used to modify  
33 or control product characteristics through its use as texturizer, thickener, stabilizer,  
34 moisture retainer, and gelatinizer (Agama-Acevedo, Flores-Silva & Bello-Perez, 2019;  
35 Mendes-Ferreira, & Ribeiro, 2012).

36 The main conventional and commercial starch sources with several uses are  
37 maize (52%), cassava (34%), potatoes (7%), and wheat (5%), as well as other  
38 conventional and unconventional sources(2%) (Agama-Acevedo et al., 2019; Felipe,  
39 Alves & Vieira, 2013). However, the use of starch in its native form is limited by some  
40 of its natural characteristics such as its low solubility, thermal resistance, and its high  
41 tendency to retrograde (Haq et al., 2019). Thus, over the years, there have been  
42 attempts to modify these properties of starch, and methods to make them better  
43 suitable for industrial food processes have been studied.

44 There are several methods to carry out the modification of starch. Among

<sup>a</sup> Federal University of Lavras, Department of Food Science, E-mail:  
mandinha\_159@hotmail.com;

<sup>b</sup> Federal University of Lavras, Department of Chemistry, Brazil, Caixa Postal 3037,  
37200-900 Lavras, Minas Gerais, Brazil. E-mail: luanandrade@ufla.br; Tel.: +55 35  
3829 1823; ORCID: <https://orcid.org/0000-0001-5357-3521>

<sup>c</sup> Federal University of Lavras, Department of Food Science, E-mail:  
ana.caroline@acad.ima.edu.br

<sup>d</sup> Federal University of Lavras, Department of Food Science, E-mail: joper@ufla.br,  
ORCID: <https://orcid.org/0000-0002-6110-3914>

\*Corresponding author

45 them, the chemical, physical, and enzymatic techniques stand out. They can be  
46 applied individually or combined. The type of modification and the agent to be used is  
47 chosen accordingly to the application given to the modified product obtained since  
48 the different types of modification reflect in the obtaining of starches with different  
49 properties (Almeida et al., 2019; Palavecino; Penci; Ribotta, 2019).

50 Countries located in tropical regions, such as Brazil, possess a great variety in  
51 starch-rich vegetables, such as taro (*Colocasia esculenta*), yam (*Dioscorea* sp.),  
52 "mangarito" (*Xanthosoma riedelianum*) and arrowroot or maranta (*Maranta*  
53 *arundinacea* L.), which could be used in their native form, dismissing modifications  
54 (Andrade, Barbosa & Pereira, 2017; Martins, Souza, Botrel, Resende & Pereira,  
55 2020; Nogueira, Fakhouri & Oliveira, 2018; Souza et al., 2019).

56 The arrowroot (*Maranta arundinaceae* L.) belongs to Marantaceae family, and  
57 it produces rhizomes that store starch with unparalleled characteristics. This  
58 vegetable is a plant originated in Latin America that has aroused interest in the food  
59 industry due to its high starch content with differentiated characteristics and high  
60 commercial value. (Souza et al., 2019). Despite the affordable commercial price  
61 reached by its starch, and its widespread use in Brazilian cuisine, the arrowroot is not  
62 among the traditionally cultivated vegetables, simply because its consumption is  
63 limited to just a few regions of the country. Its world production is also low, and there  
64 are only a few studies on this species, with scarce production data.

65 According to Valencia et al. (2015) the arrowroot starch (without specifying  
66 the variety) has granules with spherical and elongated geometries with an average  
67 size of 56.60  $\mu\text{m}$ . Its granules also have type, crystallinity, and a gelatinization  
68 temperature of 65.5  $^{\circ}\text{C}$ . This type of starch is very digestible, has high gelling  
69 capacity, and presents special physicochemical characteristics such as high amylose  
70 content, in addition to being gluten-free (Chang et al., 2016). In general, the  
71 maranta starch could have numerous applications in the food industry, with  
72 promising potential for the formation of strong polymeric matrices for the preparation of  
73 edible coating and film applications, due to the high amylose content (Valencia et al.  
74 2015).

75 In view of the potential use of this polysaccharide, studies that further  
76 investigate its properties are necessary to assess its capacity for industrial  
77 application. In addition to the study of starch as an unconventional vegetable, it is  
78 interesting to investigate different varieties, especially the arrowroot starches in the  
79 varieties "seta" and "redonda". Thus, the objective of this study was to analyze and  
80 compare arrowroot starch in the "seta" (AS) and "redonda" (AR) varieties by  
81 assessing their chemical, morphological, rheological, structural, and thermal  
82 characteristics as well as verifying their possible applicability in the food industry.

## 83 84 **2. MATERIAL AND METHODS**

### 85 **2.1 Starch extraction**

86 Redonda and Seta arrowroot rhizomes were supplied by the Brazilian  
87 Agricultural Research Corporation (Embrapa). The planting of both types of arrowroot  
88 was simultaneous, carried out in mid-October 2013, and harvested in early June  
89 2014. The work was conducted at Embrapa Hortaliças, Brasília, DF (15°56'S,  
90 48°08'W, altitude of 997 m), in an open field. No pesticides were used due to its  
91 rusticity.

92 The redonda (AR) and seta (AS) arrowroot varieties rhizomes were selected  
93 according to the lack of injuries and deformities. Afterwards, they were weighted with  
94 their peels, and washed in running water. After washing, they were immersed in

95 sodium hypochlorite solution ( $0.150 \text{ mL L}^{-1}$ ), for a 15 minutes period, for sanitation.  
96 Next, they were manually peeled with a stainless steel knife, and, once again,  
97 washed and weighted. The rhizomes were cut into 0.5 cm thick slices, and immersed  
98 in sodium metabisulphite solution ( $2 \text{ mg kg}^{-1}$ ) for 15 minutes to avoid darkening.

99 To extract the starch, the arrowroot rhizomes were crushed in a industrial  
100 blender (Lucre, model C4, Brazil) in the proportion of 1:1 with distilled water, and,  
101 then, filtered in polyester mesh. The suspension was left to rest ( $\pm 16$  hours) in a  
102 refrigerated environment ( $\pm 5 \text{ }^\circ\text{C}$ ). The supernatant was discarded, and the  
103 precipitated starch was resuspend with distilled water in order to be decanted again.  
104 This procedure of starch suspension and decantation was repeated until the product  
105 presented color and texture that are characteristic for starch. The material was then  
106 dried in forced air circulation oven at  $45 \text{ }^\circ\text{C}$  until reaching constant weight, cooled at  
107 room temperature, then reduced to powder using mortar and pestle, and sieved  
108 through 0.350 mm sieve, and, finally, conditioned in bottles until its later use,  
109 according to methodology quoted by Daiuto and Cereda (2003). Subsequently,  
110 characterization analyzes were carried out on AR and AS starches.

## 112 2.2 Chemical analysis and infrared spectrum

113 The moisture, ether extract, crude protein, ash and carbohydrate fraction  
114 contents, were determined according to the AOAC (2010). Crude fiber content was  
115 determined using the method of Van de Kumer and Van Ginke (1952). This method  
116 is gravimetric, and it uses reflux with acetic, trichloroacetic, and nitric acids as well as  
117 subsequent filtration and drying in an oven.

118 The ART-FTIR (Infrared Analyser with Attenuated Total Reflectance) specter  
119 was collected through a spectrometer (Affinity-1) equipped with an accessory for  
120 Attenuated Total Reflectance (ATR) with ZnSe crystal. The specter was acquired  
121 with 64 scans and resolution of  $4 \text{ cm}^{-1}$ , in the range of  $4,400 \text{ cm}^{-1}$  to  $600 \text{ cm}^{-1}$   
122 according to the methodology proposed by Chen et al. (2018), with modifications.

## 124 2.3 Morphological properties - scanning electron microscope (SEM)

125 After drying and grinding, the powder samples of AR and AS were deposited  
126 on a double-sided carbon tape, placed on racks covered with aluminum foil, and  
127 sputter-coated with gold (Balzers Sputter Coater SCD 050). At the end of this  
128 procedure, the samples were examined under a scanning electron microscope. The  
129 generated images were scanned at varying magnifications at 20.00 kV, and work  
130 distance between 3.0 and 9.0 mm (Andrade, Barbosa & Pereira, 2017).  
131 Approximately 10 starch scanning electromicrographs of each arrowroot variety were  
132 taken at 200x magnification and  $20 \text{ }\mu\text{m}$  scale.

## 134 2.4 Determination of paste clarity and properties

135 The paste clarity was determined as described by Craig, Maningat, Seib, and  
136 Hosney (1989) using starch suspensions ( $1\% \text{ w v}^{-1}$ ) in deionized water. The  
137 suspension was gelatinized, and placed in a water bath of boiling water for 30  
138 minutes, with agitation for 30 seconds every 5 minutes. The solution was agitated  
139 and cooled down to room temperature, and the transmittance (percentage) was  
140 determined at 650 nm (wavelength) using a spectrophotometer (Varian Indústria e  
141 Comércio Ltda., Inc., model Cary 50, Brazil) and a computer system.

142  
143 Paste viscosity analyzes were conducted using the fast viscosity analyzer  
144 (Rapid Visco Analyser – RVA; Newport Scientific) in order to set the starches

viscosity profile, following the methodology proposed by Diniz (2006). At first, the paste viscosity was set at 50°C for four minutes. Later, the gel was heated to a temperature of 95 °C, for 3 minutes. Cooling was carried out to 50°C, and then the gels were kept at this temperature for another 4 minutes. In this analysis, the values of paste temperature, as well as maximum, minimum and final viscosities, viscosity drop (difference between maximum and minimum viscosity), and tendency to retrograde (difference between final and minimum viscosity) were obtained.

## 2.5 X-Ray diffraction (XRD)

The starches were also characterized using the X-Ray diffraction (XRD) powder method with PANalytical equipment model X'Pert Pro, a 2 $\theta$  angular variation of 4° to 70°, CoK $\alpha$  radiation and a scanning rate of 5° min<sup>-1</sup>. The spacing  $d$  was calculated using the Bragg equations ( $n\lambda = 2d\sin\theta$ , where  $d$  is the spacing,  $n = 1$ , and  $\lambda = 1.7889 \text{ \AA}$ ). From the recorded diffractogram, the crystallinity index (CI) was calculated (Andrade, Barbosa & Pereira, 2017).

## 2.6 Thermal properties – TGA, DTGA, DTA and DSC

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were performed according to the methodology proposed by Pan et al. (2019), with modifications, in a DTG-60H thermogravimetric analyzer (Shimadzu) at temperatures ranging from 30 °C to 600 °C and scanning rates of 10 °C min<sup>-1</sup>. The analyzes were also run under N<sub>2</sub> atmosphere with a flow rate of 50 mL min<sup>-1</sup>. Derived thermogravimetric (DTGA) is a mathematical source that provides the first derivative of the TGA curve as a function of time or temperature. The peak area under the DTGA curve is proportional to the loss of mass during a thermal event. The calculation peak area was performed in the present work.

The differential scanning calorimetry (DSC) analysis was conducted in a DSC Q200 differential scanning calorimeter (TA Instruments, New Castle, USA), according to the methodology (Farkirov et al., 1997). The sample was prepared in suspension with distilled water, in the proportion of 3:1, and kept at rest for 30 minutes at 5 °C. The instrument was calibrated using indium as a standard. In order to determine the starch gelatinization temperature, approximately 5.0 mg of a known moisture sample were placed in a hermetically sealed aluminum crucible. Heating up to 120 °C was used as the scanning protocol with a heating rate of 10 °C min<sup>-1</sup>, under a flow of 50 mL of nitrogen per minute. The gelatinization enthalpy was calculated using the Universal Analysis software version 4.3A.

## 3. RESULTS AND DISCUSSION

### 3.1 Chemical composition and infrared spectrum (ATR-FTIR)

Table 1 shows the values for the proximal composition (moisture, ether extract, crude protein, crude fiber, ashes, and carbohydrate fraction) from starches AR and AS.

The moisture for AR and AS starches were  $10.56 \pm 0,03 \text{ g } 100\text{g}^{-1}$  and  $10.12 \pm 0,02 \text{ g } 100\text{g}^{-1}$ , respectively. The level of arrowroot starch present in the work of Nogueira et al. (2018) was higher, approximately  $15 \text{ g } 100\text{g}^{-1}$ . The moisture contents were less than  $14 \text{ g } 100 \text{ g}^{-1}$ , which is generally acceptable for dry products and, consequently, a longer life cycle for the product (Andrade et al., 2017).

195 Table 1 Chemical components and paste properties of AR and AS starches

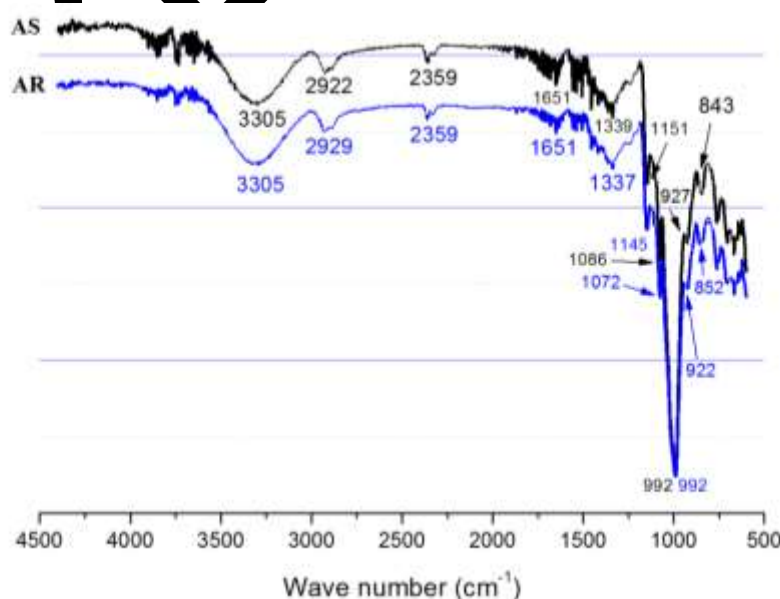
Chemical components (g 100g <sup>-1</sup> )	Starches	
	AR	AS
Moisture	10,56 ± 0,03	10,12 ± 0,02
Ether Extract*	0,35 ± 0,03	0,48 ± 0,08
Crude Protein*	0,85 ± 0,01	0,85 ± 0,01
Crude Fiber*	1,63 ± 0,18	1,06 ± 0,07
Ashes*	0,03 ± 0,00	0,12 ± 0,01
Carbohydrate Fraction*	97,14 ± 0,00	97,49 ± 0,00
Parameters (RVA)**	AR	AS
Temperature initial paste (°C)	69.85	68.95
Maximum viscosity (RVU)	563.15	481.55
Maximum viscosity temperature (°C)	78.05	84.60
Minimum viscosity (RVU)	184.35	221.98
Minimum viscosity temperature (°C)	89.90	91.50
Final viscosity (RVU)	357.63	432.46
Breakdown (RVU)	378.79	259.57
Set back (RVU)	173.28	210.47

\*dry basis; \*\*Each value represents the average of two determinations; AR = Arrowroot Redonda and AS = Arrowroot Seta.

196 It can be observed that the level of ether extract, proteins, ash, and crude  
 197 fiber were low, therefore having a high purity level in the starches obtained. Also,  
 198 high levels of carbohydrate fraction were obtained. Souza et al. (2019) also obtained  
 199 a high content of glycidic fraction (greater than 93.98 g 100g<sup>-1</sup>) in the extraction of  
 200 arrowroot starch grown under different agronomic managements.

201 Gordillo, Valencia, Zapata and Benao (2014) also found low values for the  
 202 components of ash, crude protein, lipids, and crude fiber in arrowroot starch used to  
 203 produce glycerol membranes, showing efficiency in extraction, similar to the present  
 204 work.

205 Starches were evaluated by ATR-FTIR spectra to determine the presence of  
 206 functional groups by bands, which explain their structure (Figure 1).



221 Figure 1 ATR-FTIR spectra for the arrowroot redonda (AR) and arrowroot seta (AS)  
 222 starches  
 225

226  
227 The ATR-FTIR specters represent a typical starch behavior, as described in  
228 the literature. Thus, it is not possible to observe many differences between the  
229 samples in absorption bands, with only small changes in their transmittances.

230 The specters show a broad band between  $3,400\text{ cm}^{-1}$  and  $3,200\text{ cm}^{-1}$  which  
231 corresponds to the axis deformation of hydroxyl groups ( $-\text{OH}$ ) in the inter-molecular  
232 hydrogen bonds of alcohols commonly found in polysaccharides, confirming the  
233 presence of carbohydrates (Andrade, Silva, Nundes & Pereira, 2020; Saikia &  
234 Konwar, 2012), in the present case, starch.

235 The bands at  $2,929\text{ cm}^{-1}$  and  $2922\text{ cm}^{-1}$  are attributed to the axis deformation  
236 of the C-H bond found in the region between  $3,000\text{ cm}^{-1}$  and  $2,840\text{ cm}^{-1}$  (Zeng, Liu &  
237 Liu, 2014). Meanwhile, the bands at  $2,359\text{ cm}^{-1}$  are attributed to the  $\text{CO}_2$  absorbed  
238 from the environment (Andrade et al., 2020).

239 Absorption bands at  $1,651\text{ cm}^{-1}$  correspond to bound water in the amorphous  
240 region in starch granules. As this band is related to crystallinity of each starch, its  
241 variations have the potential of affecting this band (Kizil, Irudayaraj & Seearaman,  
242 2002). In the present study, the bands at  $1,651\text{ cm}^{-1}$  for arrowroot starches do not  
243 present different intensities, which may indicate that the crystalline patterns of AS  
244 and AR starches do not differ significantly (Andrade et al., 2017). The absorptions in  
245  $1,339\text{--}1,337\text{ cm}^{-1}$  have been related to deformations of C-O-C groups or vibrational  
246 bands (bending and deformation) related to C and H atoms (Andrade et al., 2020;  
247 Andrade et al., 2017).

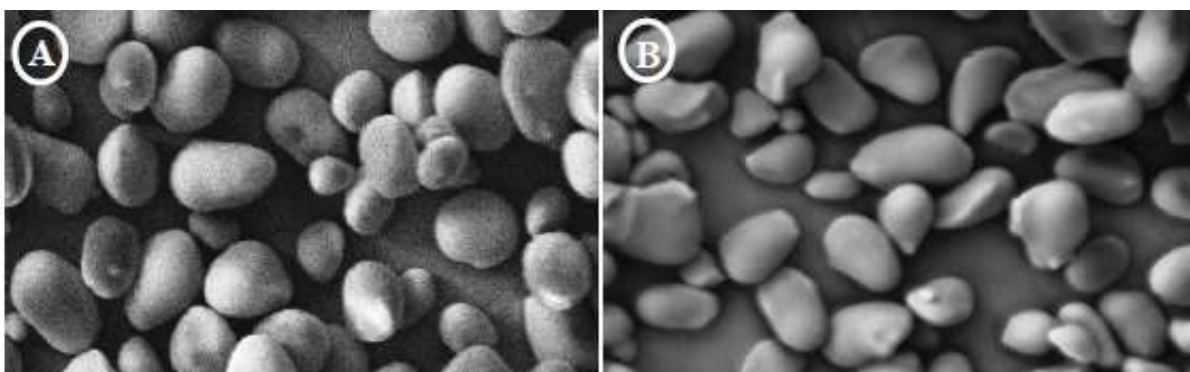
248 The bands at  $1,151\text{ cm}^{-1}$  and  $1,145\text{ cm}^{-1}$  correspond to the coupling of the C-O  
249 and C-C stretching mode (Andrade et al., 2017). Thus, the infrared spectra proves  
250 the structure of a polysaccharide with C-O-C bonds, characteristic to carbohydrates  
251 (like starch) between  $1,200\text{ cm}^{-1}$  and  $900\text{ cm}^{-1}$ , confirming the bond between the  
252 polymer forming monomers, as described by Andrade et al. (2020). According to  
253 Zeng et al. (2014), who studied the taro starch cultivated in China, the bands at  
254  $1,081\text{ cm}^{-1}$  and  $1,019\text{ cm}^{-1}$  are characteristic of the O-C stretching associated with  
255 anhydrous glucose, which can be observed at  $1,072\text{ cm}^{-1}$  for AR and at  $1086\text{ cm}^{-1}$  for  
256 AS.

257 The bands at  $977\text{ cm}^{-1}$  and  $922\text{ cm}^{-1}$  correspond to the water sensitivity, and  
258 indicates the presence of this molecule in the starch structure (Deepika, Jayaram  
259 Kumar & Animesh, 2013; Andrade et al., 2017), as evidenced by the moisture analysis  
260 (Table 1).

### 261 262 **3.2 Morphological properties**

263 The size and shape of the arrowroot starch granules are shown in the Figure 2.  
264 It is possible to see that the predominant geometric shapes of the starch granules of  
265 the two arrowroot varieties are oval and ellipsoid. The characteristic size is in the  
266 range of  $8\text{ }\mu\text{m}$  to  $45\text{ }\mu\text{m}$ , with a predominance of granules of approximately  $30\text{ }\mu\text{m}$ ,  
267 for both AR and AS. Therefore, they can be considered large granules (ten granules  
268 were measured in order to best represent the average of their size). It can also be  
269 observed that the surface of the granules is quite smooth, without irregularities or  
270 superficial porosity, which shows intact and undamaged granules, similar to the ones  
271 in work of Nogueira et al. (2018).

272 The average size found for arrowroot in the work of Valencia et al. (2015) was  
 273 approximately 56.60  $\mu\text{m}$ , a higher value than those found in the present study. This  
 274 difference may be due to the difference in the variety of arrowroot used for the  
 275 extraction of starch. However, the work cited does not specify the variety used.



276 Figure 2 Scanning electromicrographs of AR starch (A) and AS starch (B), with 830x  
 277 magnification and 20  $\mu\text{m}$  scale.

### 279 3.3 Paste clarity and properties

280 AR and AS starches showed a low percentage of transmittance, 6.99% and  
 281 3.92%, respectively, thus forming opaque pastes. According to Craig et al. (1989),  
 282 the clarity of the paste and the tendency to retrograde influence the technological  
 283 quality of the starch. Starches used as a thickener, pie fillings, in food topping, or as  
 284 edible films should preferably be transparent, while starches used in salad dressings,  
 285 and in the preparation of puddings and ready-made desserts must be opaque, like  
 286 the starch pastes analyzed in the present work.

287 The rheological profile of AR and AS starches was analyzed by RVA and their  
 288 results are shown in Table 1. It is verified that the AR starch presented paste  
 289 temperature of 69.85  $^{\circ}\text{C}$ , and maximum viscosity of 563.15 RVU. The values for  
 290 paste temperature and maximum viscosity for the AS starch were, respectively,  
 291 68.95  $^{\circ}\text{C}$  and 487.75 RVU. The two temperatures are low, which may indicate a  
 292 smaller resistance to dissociation of intramolecular hydrogen bonds, and greater  
 293 starch ability for expansion. Because they produce viscous pastes more rapidly,  
 294 these starches may be potentially useful for their use in instant-preparation food,  
 295 such as soups and puddings, and, due to their high viscosity, they can be used as  
 296 thickeners.

297 Paste temperatures for the starches of the two arrowroot varieties were close  
 298 to the one found by Barroso and Mastro (2019), which were of 67.2 $^{\circ}\text{C}$  for arrowroot  
 299 starch. As for the viscosity peaks, they were higher than all the starches from  
 300 different vegetables (sweet potato, peruvian carrot, cassava, maize, and waxy maize)  
 301 analyzed by Shirai et al. (2007), except the potato starch.

302 The AR starch breakdown presented itself to be higher (378.79 RVU) than AS'  
 303 (259.57 RVU), which characterizes greater fragility when cooked in water, and lower  
 304 resistance to hot agitation. When compared to the values observed by Shirai et al.  
 305 (2007), the potato starch, once again, obtained greater values while the peruvian  
 306 carrot starch presented a breakdown value that is only higher than the arrowroot seta  
 307 starch.

308 The AS starch setback was 210.47 RVU, higher than the setback for AR  
 309 starch this was 173.28 RVU. This higher tendency to starch setback for the two  
 310 arrowroot varieties indicates lower granule stability to mechanical work and higher

311 capability of amylose recrystallization. When compared to studies in which the  
312 arrowroot starch was analyzed, these values are higher (Barroso & Mastro, 2019,  
313 Leonel, Sarmiento & Cereda, 2002; Ferrari, Leonel & Sarmiento, 2005), as well as for  
314 the starches in potato, sweet potato, Peruvian carrot, cassava, maize, and waxy  
315 maize, in the study by Shirai et al. (2007).

316 Both the AR starch (357.63 RVU) and the AS starch (432.46 RVU) presented  
317 high final viscosities. In the study of Barroso and Mastro (2019), the final viscosity  
318 value in the starch arrowroot was 57.2 RVU, which is different from the values found  
319 in the present study. Final viscosity is an important parameter in starch use in food  
320 because it corresponds to the product's viscosity after cooling, in this case, at 50 °C.  
321 Pie fillings demand higher viscosity, which would avoid overflowing during  
322 transportation.

323 The AR sample presented higher paste temperature, as well as maximum  
324 viscosity and breakdown while the AS sample presented higher minimum viscosity,  
325 higher final viscosity, and higher setback. All the differences among the analyzed  
326 results for arrowroot starches, both among the varieties here analyzed and among  
327 other authors' starches, can occur, partially, due to variations observed in the  
328 composition (amylose and lipid content), in the starch granule size (Ferrari et al.,  
329 2005), and in the physiological maturity stage of rhizomes. The phosphor and lipid  
330 contents, in high amounts, could influence the paste characteristics.

331 The elevated values for viscosity, peak viscosity breaking, and setback  
332 tendency for the arrowroot starch samples enable their use in fortified breakfast  
333 cereals, infant food, or as ingredients for their formulation, as well as meat products  
334 and bakery products (Maia, Wang, Caserri, Corral, & Fernandes, 1999).

### 336 3.4 X-Ray diffraction (XRD)

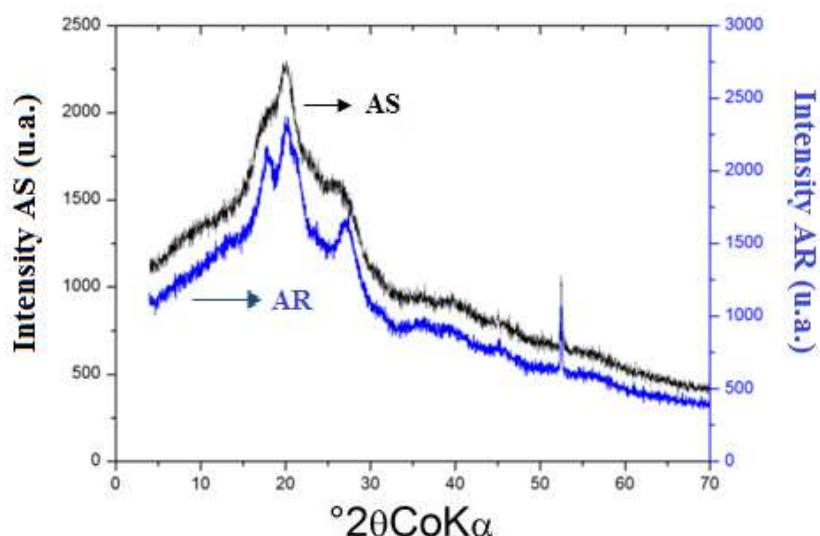
337 X-ray diffractometry reveals the characteristics of crystalline structure of starch  
338 granules (Figure 3). It is possible to observe in Figure 3 that the starches in AR and  
339 AS presented pattern type-C, confirming the result observed in work of Nogueira et al.  
340 (2018). It was observed in the work of Andrade et al. (2017) type-C crystallinity for  
341 the yam tuber, a vegetable from underground parts of the plant, such as  
342 arrowroot rhizomes.

343 The crystallinity index (CI) determined from the total areas and peaks of the  
344 X-ray diffractograms, is an important parameter that influences the physical,  
345 mechanical, and technological properties of starch (Martins et al., 2020). The CI  
346 found for AR and AS starches was of 40.79% and 39.17%, respectively, considered  
347 high, but within the range suggested by Miranda, Carvalho, Vieira and Castro (2019),  
348 between 35 and 45% for native starches. As observed by the infrared spectrum in the  
349 band intensity at  $1651\text{ cm}^{-1}$ , there is no great difference in the crystallinity of the two  
350 starches studied.

351 According to Pepe (2011), the X-ray diffractogram of the native arrowroot  
352 starch showed a very mild peak, around  $5.6^\circ$ , peaks at  $17^\circ$ ,  $18^\circ$ , and  $23^\circ$ , indicating  
353 that these starches had a type C crystalline pattern. In the work of Salgado et al.  
354 (2005), there is the influence of the maturation stage, with green bean starch within  
355 pattern C and mature beans within pattern A, showing differences in the three-  
356 dimensional organization of structures, which may be related to the size of the  
357 amylopectin chain (Freitas, 2002). Therefore, the advanced maturation of arrowroots  
358 studied in this work may have altered their crystalline structure since they do not a  
359 peak at  $5.6^\circ$ , for example.

360





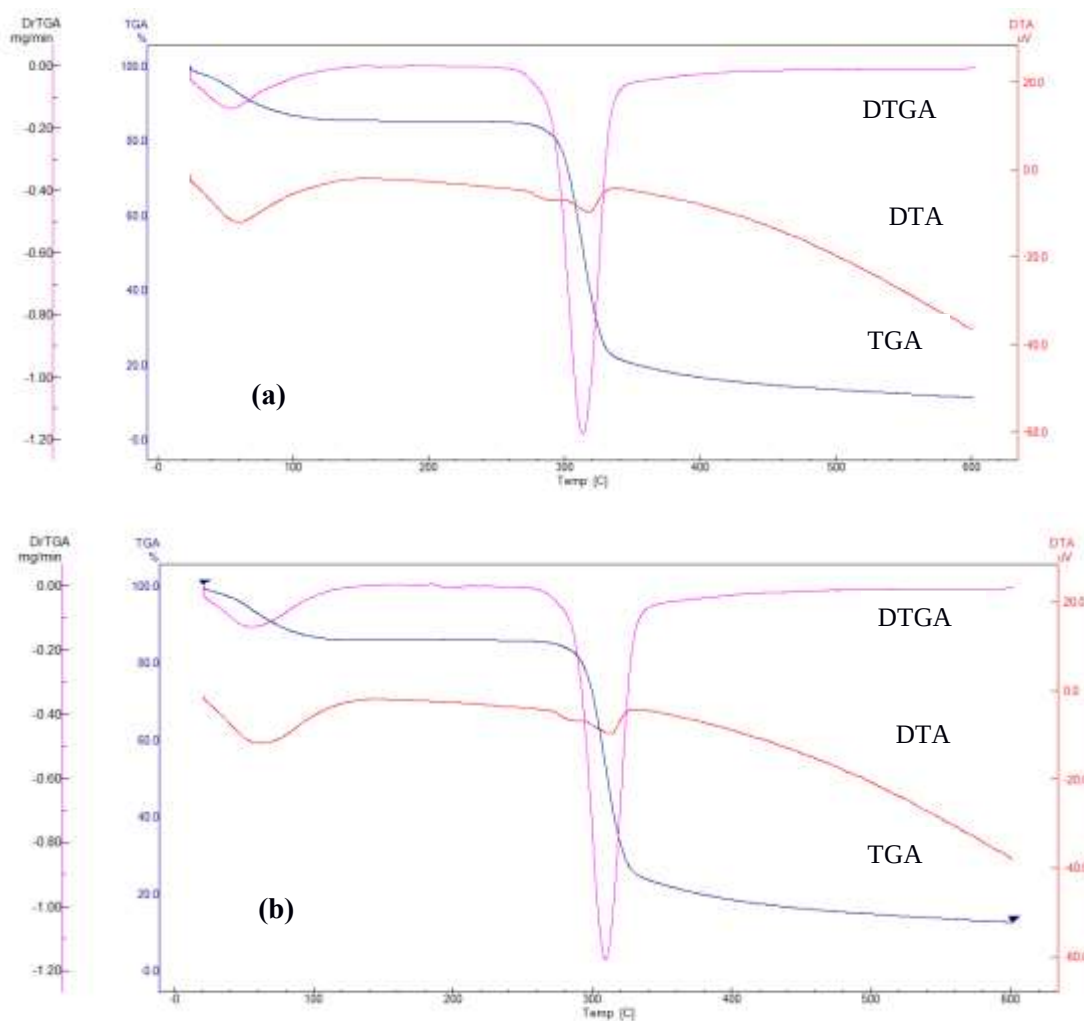
361  
362 Figure 3 X-Ray diffractograms for starch of arrowroot redonda (AR) and starch of  
363 arrowroot seta (AS)  
364

365 As for the degree of crystallinity being studied, it is possible to observe that the  
366 intensity values for the peaks in the diffractograms were similar, suggesting  
367 approximate values for the internal bonding forces in the molecules as well as  
368 association degrees among the starch chains.  
369

### 370 3.5 Thermal properties

371 Through the thermogravimetric technique, it is possible to measure the mass  
372 variation of a substance in function of the temperature, and the results are obtained  
373 through curves that show the conditions regarding stoichiometry, thermal stability,  
374 and composition of the analyzed samples. In the starch, the thermogravimetry  
375 (TGA/DTGA) is used to assist in the studies of thermal degradation of starch material  
376 whereas it is important for the material. However, it should not be used in heating  
377 conditions under which degradation or any undesirable alteration might happen to its  
378 properties. Therefore, knowing the temperature and the degradation parameters for  
379 the starch material is essential in its application. Figures 4a and 4b show the  
380 TGA/DTG and DTA curves for AR and AS starches, respectively.

381 In the AR starch (Figure 4a) two thermal events of weight loss were observed  
382 through the TGA curve. The first, which corresponds to weight loss of 14%, was  
383 attributed to the evaporation of volatile materials (majorly, the water absorbed by the  
384 starch material), and occurred between 24 °C and 112 °C. The second event is  
385 related to the stage of thermal degradation for major starch constituents, and minor  
386 constituents as well, such as proteins and lipids, with initial degradation temperature  
387 ( $T_{\text{onset}}$ ) at 299°C and final ( $T_{\text{endset}}$ ) at 326°C, and weight loss at 49%.  
388



389

390  
391 Figure 4 TGA/DTGA/DTA curves for starch in arrowroot redonda (a) and for starch in  
392 arrowroot seta (b)  
393

394 In the AS starch sample (figure 4b), the two thermal events were also  
395 observed. The first, weight loss of 13%, happened between 21°C and 107 °C while,  
396 in the second thermal event, there was a weight loss of 52%, and  $T_{onset}$  at 293 °C and  
397  $T_{endset}$  at 323 °C. These results were similar between the two samples.

398 Percentage values of the first mass loss in TGA by both starches are slightly  
399 higher than the moisture content (Table 1). It can be concluded that in addition to  
400 water, there may be, in small quantities, other volatile substances.

401 From the obtainment of the derived thermogram curve (DTGA), it was possible  
402 to determine the temperatures at which the degradation velocity is maximum to  
403 each of the samples in the two weight loss events, which are 53.12 °C, when the  
404 maximum volatiles loss happens, and 313.51 °C, which is when the maximum  
405 degradation happens for the AR starch, and, respectively, 55.02 °C and 309.41°C for  
406 the AS starch.

407 From the thermogravimetric analysis, it is possible to determine the content of  
408 inorganic substances in the samples (Araújo et al., 2006). However, the analysis  
409 carried out under a controlled atmosphere of  $N_2$  did not provide data compatible with  
410 the ash content values obtained in the chemical composition (Table 1) as the  
411 impurities present remain as residue after sample volatilization, and decompose  
412 differently in atmosphere of air or nitrogen (BERNAL et al., 2002). For the arrowroot

413 redonda, this value was 11%, while for the arrowhead seta it was 12.29%, a  
 414 difference that was already expected due to the fact that the ash content in the  
 415 arrowroot redonda is higher than in the arrowroot seta.

416 The second well-defined peak for DTGA for both studied starches suggests  
 417 the possibility of a simple degradation mechanism involving amylose and  
 418 amylopectin as observed by Franklin et al. (2017) for the *Curcuma angustifolia* starch.  
 419 The same occurs in the work of Nogueira et al. (2018) with arrowroot starch. Given  
 420 these results, it is possible to affirm that the arrowroot starch is thermally stable, and  
 421 has desirable characteristics for the production of biodegradable, and edible  
 422 packaging or films (Nogueira et al., 2018).

423 For all thermal events, the DTA curves showed endothermic processes, that is,  
 424 they absorb energy for mass losses to occur. In the study of yam and taro starches  
 425 by Andrade et al. (2017), all mass loss processes were endothermic, being similar to  
 426 the present work even though they are starches from different plant sources.

427 To better understand arrowroot starches, we investigated the heat flow signals  
 428 of DSC starches gelatinization that revealed significant variations in their gelatinization  
 429 transition. The parameters of gelatinization temperature [start ( $T_o$ ), peak temperature  
 430 ( $T_p$ ), conclusion ( $T_c$ )], gelatinization temperature range ( $\Delta T = T_c - T_o$ ), and  
 431 gelatinization enthalpy ( $\Delta H$ ) on a dry starch basis are presented in Table 2. It can be  
 432 verified that the AR starch gelatinization occurred at the temperature of 57.61 °C  
 433 while AS starch gelatinized occurred at a slightly lower temperature, at 55.39 °C,  
 434 demonstrating the same behavior observed in the pasta properties analysis.

435

436 Table 2 Thermal properties for gelatinization of AR and AS starches

Starches	$T_o$ (°C)	$T_p$ (°C)	$T_c$ (°C)	$\Delta T$ (°C)	$\Delta H$ (J g <sup>-1</sup> )
Arrowroot redonda (AR)	45.87	57.61	61.37	15.50	12.56
Arrowroot seta (AS)	46.83	55.39	62.10	15.27	15.73

437  $T_o$ ,  $T_p$ ,  $T_c$  = initial temperature or onset, peak temperature and final or conclusion  
 438 temperature, respectively;  $\Delta T$  = temperature variation;  $\Delta H$  = enthalpy variation.

439

440 The results obtained in the gelatinization enthalpy determination demonstrated  
 441 that the AR starch demands less energy in order to make the tumescence happen  
 442 through water absorption and temperature elevation (gelatinization) than the AS  
 443 starch.

444 The initial, peak, and final temperature values for gelatinization were well  
 445 below the temperatures found in the literature. In the study of Barroso and Mastro  
 446 (2019), the value found for initial temperature was 63.9 °C, and 81.3 °C for final  
 447 temperature. In the work of Charles et al. (2016), the values of  $T_o$ ,  $T_c$ , and  $T_p$  for  
 448 arrowroot starch were 76 °C, 86 °C, and 80 °C, values also higher than the ones in  
 449 the present study. The enthalpy of the work mentioned above was closer to the AR  
 450 and AS starches, 11.0 J g<sup>-1</sup>, but different from the work of Barroso and Mastro (2019),  
 451 which has an enthalpy of only 4.2 J g<sup>-1</sup>. This difference may be due to the possible  
 452 difference in arrowroot cultivars among the vegetables studied.

453 The initial gelatinization temperatures of the samples of the two starches  
 454 obtained by the DSC were lower than those presented by the RVA because,  
 455 according to Jane et al. (1999) and Pérez, Breene and Bahnssey (1998), the paste  
 456 temperature obtained from the RVA is related to the sensitivity of the device in  
 457 detecting the first increases in the paste viscosity of starches, differently from the  
 458 initial gelatinization temperature, which is detected when the starches first granules

459 begin to disorganize. The values obtained in DSC are, therefore, more accurate,  
460 while those obtained in RVA correspond to a specific temperature range. Another  
461 important fact is that the RVA analysis started at a temperature of 50 °C, which is  
462 higher than the  $T_0$  found by the DSC.

463 The size of the granules can also influence gelatinization temperatures ( $T_0$   
464 and  $T_p$ ), and smaller granules provide higher gelatinization temperatures (initial,  
465 peak and final). Gelatinization and enthalpy temperatures are altered by the shape  
466 and composition of the granule (crystalline and amorphous zones), distribution of  
467 small and large granules, and by the sources of starches that may contain different  
468 amounts of amylose, amylopectin, and phosphorus (Yonemoto; Calori- Domingues;  
469 Franco, 2008). The crystallinity indices of the studied starches were close  
470 (approximately 40%). Therefore, it was not the factor that caused the difference  
471 between the  $\Delta H$  found.

472 According to Charles et al. (2016), flours and/or starches composed of root  
473 and tuber can meet industrial requirements of carbohydrate-based food products  
474 since they gelatinize at relatively low temperatures and exhibit a high viscosity profile  
475 when compared to cereal starches, as occurred in the present study for arrowroot  
476 rhizomes of two varieties.

#### 477 **4. CONCLUSIONS**

478 1. The extracted starches present a high purity level, with low levels of lipids,  
479 crude protein, crude fiber, and ash. The infrared spectra have characteristics of  
480 carbohydrates.

481 2. The predominant geometrical shapes in AR and AS starches are oval and  
482 ellipsoid with sizes ranging from 10  $\mu\text{m}$  to 15  $\mu\text{m}$ .

483 2. Starches (AR and AS) have low paste clarity and can be used in salad  
484 dressings as well as in the preparation of puddings and ready-made desserts.

485 3. The starches presented relatively high maximum viscosity, being the  
486 highest value found in AR, and both were not stable at high temperatures and  
487 agitation, tending to regression, which was higher for AS – these characteristics  
488 suggest the use of these starches in the instant food industry and in fortified  
489 breakfast cereals industry, infant food, meat products, and bakery products.

490 4. The starches of both arrowroot varieties presented type-C crystalline  
491 structure and, according to literature, approximately 40%.

492 5. The characterization through thermogravimetry allowed the determination  
493 of the average initial temperature for degradation, and the DTGA obtainment  
494 provided the values under which the dehydration phenomenon and the  
495 polysaccharide degradation events occur, similarly in both starches.

496 6. The paste temperatures were shown to be lower than the gelatinization  
497 temperatures obtained by the DSC, and were smaller for the arrowroot seta starch,  
498 which presented higher  $\Delta H$ .

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