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Physicochemical, Thermal, Structural and Pasting Properties of Unconventional Starches from Ginger (*Zingiber officinale*) and White Yam (*Dioscorea* sp.)

Cristina Soltovski de Oliveira[1]
https://orcid.org/0000-0002-1051-6225

Radla Zabian Bassetto Bisinella[1]
https://orcid.org/0000-0001-7299-011X

Camila Delinski Bet[1]
https://orcid.org/0000-0001-8800-2024

Cleoci Beninca[1,2]
https://orcid.org/0000-0003-0051-3562

Ivo Mottin Demiate[1]
https://orcid.org/0000-0002-5609-0186

Egon Schnitzler[1*]
https://orcid.org/0000-0003-2696-4441

[1]State University of Ponta Grossa, Departament of Food Engineering, Ponta Grossa, Paraná, Brasil; [2]Federal Institute of Education, Science and Technology of Santa Catarina, Departament of Food Technology, Canoinhas, Santa Catarina, Brasil.

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***Correspondence: egons@uepg.br; Tel.: +55 42 999916617 (E.S.)

HIGHLIGHTS

- Unconventional starches from ginger (Zingiber officinale) and yam (Dioscorea sp.) were investigated.
- Lower thermal stability was found for unconventional starches when compared to maize starch.
- Greater relative crystallinity and enthalpy of gelatinisation was recorded for ginger starch.
- Yam starch had the highest peak viscosity, and corn starch had the highest breakdown.

Abstract: Ginger and white yam starches were investigated and compared with maize starch. Proximal composition, thermogravimetry, differential scanning calorimetry, microscopy, colourimetry, X-ray powder diffractometry and pasting profile were analysed. The unconventional starches presented higher protein and ash contents than the maize starch, that had the highest thermal stability. Higher gelatinisation temperatures were reported for ginger starch, and the enthalpy of the unconventional starches were similar. The maize starch presented the lowest gelatinisation values. For the corn starch the granules were polygonal and smaller than the unconventional starches, and oval shapes and larger diameters were found for the ginger and yam starches. The unconventional starches presented less brightness and a greater tendency to red and yellow. The maize and ginger starches had A-type diffraction patterns, while the white yam starch had a C-type pattern. The highest relative crystallinity was observed for the ginger starch and there were small differences between the yam and maize starches. Higher peak viscosity and final viscosity and lower pasting temperature were observed for the yam starch. Ginger starch showed the highest shear and stability of heating glue, so may be used in products processed under high temperatures; and yam starch can be used in acidic foods that require high viscosities.

Keywords: native starch; gelatinization; thermal analysis; pasting profile.

INTRODUCTION

The main source of carbohydrate reserve in plants and energy source of the human diet is starch. Its technological characteristics besides great versatility contribute to its wide use in the food industry. Starch is a polymer with a high level of industrial applications; it can be extracted from numerous vegetable sources, and maize starch is one of the major commercial starches. Maize is a cereal belonging to the Poaceae family that can be used in cornmeal, grits, flour, tortillas, snacks, and breakfast cereals [1]. It contains about 72-73% starch, which is composed of 75% amylopectin and 25% amylose [2]. Maize starch is generally used to improve texture and stability characteristics; it is widely used for industrial purposes due to its low cost and high production [3]. Unconventional starch sources have been increasingly studied in an attempt to find differentiated characteristics, without needing changes in the structure of starches.

The yam (*Dioscorea* sp.) originates from Africa and Asia: Nigeria is the world's largest producer [4]. In 2008, Brazil produced about 250,000 tons of yam, being the state of Pernambuco the largest producer and consumer. This way, Brazil was the second largest producer in South America and the eleventh in the world ranking [5] Yams are rich in several vitamins (A, B-complex, C), phosphorus, calcium, iron and carbohydrates; the latter are mainly starch [4-6]. The starch content of these tubers ranges from 70 to 80% on a dry basis, making them an inexpensive source of starch for industrial use. Due to the difficulty in extracting its starch, the yam is still not fully utilised for industrial purposes [7]. Dioscorea genus tubers are used as feedstock to produce diosgenin, which is a starting material for oral contraceptives, sex hormones and other steroids; therefore, they are of great economic importance for the pharmaceutical industry [8].

Ginger (*Zingiber officinale*) is a popular spice that is cultivated and used for medicinal purposes, mainly in India; however, it is also highly cultivated in tropical and subtropical areas such as China, Indonesia and Nigeria [8-9-10]. It has organic acids, such as citric, malic, oxalic, succinic and tartaric acids, as well as gingerol, which is responsible for its pungent taste [11]. Ginger rhizomes contain approximately 11.4% starch; the amylose content (18-30%) is similar to that of potato starch and higher than that of cassava starch [9-12].

The objective of this study was to characterise ginger (*Zingiber officinale*) and yam (*Dioscorea* sp.) unconventional starches by physicochemical, thermal, pasting and structural analyses, and to compare these properties with those of maize starch.

MATERIAL AND METHODS

Materials

The ginger and yams were obtained locally in the city of Ponta Grossa (Paraná, Brazil). The maize starch was commercially available.

Starch extraction

The raw materials were washed to remove soil and dirt. They were then peeled and cut manually before the extraction of the starch following the methodology adapted from Costa et al. [13]. Each sample was extracted in aqueous medium. After drying, the starches were ground in an analytical mill and stored in a desiccator with anhydrous calcium chloride at room temperature (25 °C).

Proximal composition of starches

The physicochemical analysis of the starches was performed in accordance with the literature [14] and AOAC methods [15] using the following the protocols: moisture (925.09); ash (923.03); proteins (920.87); crude fibre (993.21) and lipids (968.20). The carbohydrate content was calculated by difference.

Colourimetry

To determine the colour parameters of the starch granules, Mini Scan XE 45/0-L Plus (Hunter Inc., USA) equipment, with a reflectance spectrophotometer, was used. There were three colour components: L*, brightness ranging from 0 (black) to 100 (white); a* ranging from positive (red) to negative (green); and b* which varies from positive (yellow) to negative (blue) [8-16-17]. The sample was placed in a Petri dish and the values of the colour coordinates were recorded.

Thermogravimetry (TG)

The thermogravimetric curves were obtained using a TGA-50 thermal analysis system (Shimadzu, Japan). The instrument was preliminarily calibrated with mass standard and verified with standard calcium oxalate monohydrate. The samples were heated from 30 to 650 ºC using open alumina crucibles, with approximately 10.0 mg of each sample under a synthetic air flow of 150 mL min-1 and a heating rate of 10 °C min-1. The derivative thermogravimetric curves (DTG) were calculated as the first derivative of the TG curves and were used to determine the mass loss points. All the mass loss percentages were determined using TA-60 data analysis software (Shimadzu, Japan) [16].

Differential scanning calorimetry (DSC)

The DSC Q-200 (TA Instruments, USA) equipment was previously calibrated according to the manufacturer’s specifications and checked with indium standard (99.99% purity). A starch–water suspension (1:4, w/v) was prepared in aluminium crucibles, which were subsequently sealed and allowed to stand for 1 h at room temperature prior to the start of the analysis. The results were obtained using Universal Analysis-2000 software (TA Instruments, USA). The analysis was conducted under the following conditions: sample mass about 2.58-2.87 mg, heating rate of 10 ºC min-1 from 30 to 100 ºC, and air flow rate of 50 mL min-1 [17].

Field emission gun-scanning electron microscopy (FEG)

The surface images of the starch granules were obtained using a MIRA 3 field emission gun-scanning electron microscope (Tescan, Czech Rep). The parameters were: 15 kV tension on field emission gun, generated by a lamp with a tungsten filament.

The samples were pulverised over a carbon tape and were metallised with gold and palladium to promote the passage of electrons in the samples; the images were then obtained with the software Mira TC [18].

X-Ray powder diffractometry (XRD)

Each sample was analysed in an Ultima 4 X-ray diffractometer (Rigaku, Japan); the samples were submitted to 20 kV and 20 mA. The following parameters were set: CuKα radiation (λ = 1.542 Å), scattered radiation detected from the angles of $5^{o} \leq 2\theta \leq 50^{o}$, scanning speed of 2 min-1 and a step of 0.02º. The diffractograms were prepared using Origin 6.1 software (OriginLab, USA) and the degree of relative crystallinity of the starches was calculated using Equation 1[19]:

$$Xc = Ap/(Ap+Ab) \times 100 \qquad (1)$$

Where: Xc = relative crystallinity; Ap = peak area; Ab = basis area, which refers to the amorphous area of the diffractograms.

Pasting properties

An aqueous suspension of 8 % starch (dry basis) in 28 g total mass was prepared and subjected to a controlled heating (until 95 ºC) and cooling (until 50 ºC) cycle under constant stirring. The samples were analysed using Rapid ViscoAnalyser Series 4 (RVA-4, Newport Scientific, Australia) equipment, and the results were identified using Thermocline for Windows software [20].

Statistical analysis

The results of the triplicate analyses were evaluated by means of univariate statistical analysis (analysis of variance - ANOVA) and Tukey's test for comparison of the samples, with significance level of 5%, using SASM-AGRi software v.8.2.

RESULTS

Proximal composition of starches

The results of the physico-chemical analyses of the maize, ginger and yam starches are shown in Table 1.

Table 1: Proximal composition and colorimetric data of starches from maize (a), ginger (b) and yam (c)

Sample	Proximal composition (%)						Colorimetric data		
	Moisture	Carboydrate	Protein	Lipids	Fiber	Ash	L*	a*	b*
(a)	8.01 ± 0.18^{c}	90.96 ± 0.4^{a}	0.28 ± 0.06^{b}	$<0.1^{b}$	0.61 ± 0.04^{a}	0.6 ± 0.31^{b}	96.27 ± 0.28^{a}	-2.36 ± 0.05^{c}	4.27 ± 0.24^{c}
(b)	9.34 ± 0.32^{b}	88.67 ± 0.29^{b}	1.01 ± 0.03^{a}	0.19 ± 0.04^{a}	$<0.1^{b}$	0.5 ± 0.08^{b}	90.51 ± 0.05^{c}	-0.32 ± 0.01^{a}	12.75 ± 0.04^{a}
(c)	11.89 ± 0.27^{a}	84.95 ± 0.24^{c}	1.14 ± 0.1^{a}	$<0.1^{b}$	$<0.1^{b}$	1.7 ± 0.07^{a}	92.04 ± 0.58^{b}	-0.42 ± 0.02^{b}	6.77 ± 0.18^{b}

Values that have the same letter in the same column do not present significant difference by Tukey's test (p<0.05).

Ginger starch presented a proximal composition similar to that reported by Sukhija et al. [7] and Madeneni et al. [9]; however, the values of protein and ash were higher in the present study, highlighting the lipid value that was statistically highest than for other two starches. Protein and ash contents in yam starch were higher than those reported by Zhu [21], and lipid levels were also higher than those found by Jayakody et al. [22] and Andrade et al. [23] Variations in protein content between yam varieties may be due to factors such as different methods of analysis, as well as the genetic and physiological state of ripening tubers [21], in this study the protein content of yam starch were highest than corn starch and similar to the ginger starch. Andrade et al. [23] report that the strong binding of starch to the mucilage present in tubers increases the protein content. The genetics of the species and the type of solvent used in the analysis may influence the lipid content. In terms of ash content, values below 0.3% are generally found: higher values may be related to the phosphorus content, which is commonly observed in tuber starches [21].

The maize starch presented the lowest levels of proteins, lipids and ash, and the highest carbohydrate and fibre content. This may be related to the chemical extraction method, which purifies samples and reduces values for proteins, lipids and ash, compared to the yam and ginger samples, which underwent aqueous extraction.

Colourimetry

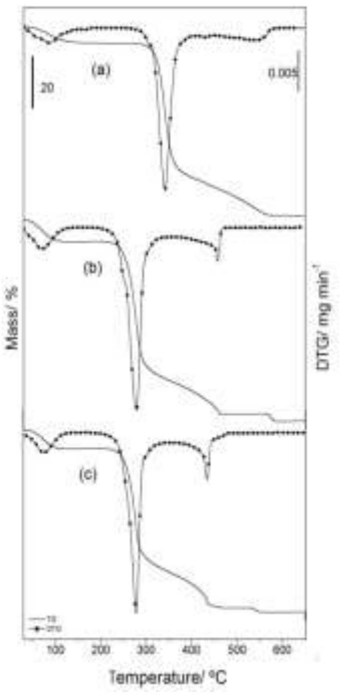
The starch samples showed values with significant differences between all the colorimetric parameters (Table 1). The brightness (L*) values for the maize starch were highest than the ginger and white yam starches, the brightness (L*) values for the maize starch were highest than the ginger and white yam starches, that could be linked to its lower ash content. In addition, a greater tendency to red and yellow was found for the ginger and yam starches.

Only for unconventional starches, white yam starch presented the highest luminosity, with a lower tendency for red and yellow, when compared to ginger starch. This difference was not visually noticeable, although the samples differed significantly by Tukey's test. Sukhija et al. [7] obtained similar results for the brightness of ginger starch, although higher a* values and lower b* values were recorded. Perez et al. [24] obtained similar values for brightness and b*, but lower values for tendency to red.

Thermogravimetry (TG)

When the starches underwent to controlled heating, three mass losses occurred, as shown in Figure 1. The first mass loss was related to sample dehydration, followed by a period of stability. The second loss was related to the decomposition of organic material, and the third loss was attributed to the oxidation of the organic matter [19]. For the ginger and yam starches, the displacement occurred in the last mass loss, observed in the TG curve, was due to the oxidation of the organic matter that was generated by the oxidizing atmosphere (air, with a flow rate of 150 mL min^{-1}) [40].

Figure 1: TG/DTG curves of starches from maize (a), ginger (b) and yam (c)



The TG/DTG results (Table 2) for the first mass loss were similar to those obtained by the gravimetric method for moisture. Thermogravimetric analysis (TG) has an advantage over conventional techniques because it is faster and more accurate, as well as requiring a smaller amount of sample.

Table 2: TG/DTG and DSC results of starches from maize (a), ginger (b) and yam (c)

Samples	TG Results		DTG Results		DSC Results				
	Steps	Δm/%	ΔT/°C	Tp/°C	To (°C)	Tp (°C)	Tc (°C)	Tc-To (°C)	ΔH_{gel} (J g^{-1})
(a)	1st	8.58	30-159	85.13	72.46±0.52 b	75.77±0.19^{b}	79.85±0.38^{c}	7.34±0.20^{c}	11.72±0.42 b
	S	-	159-271	-					
	2nd	71.58	271-401	341.12					
	3rd	18.89	401-598	405.36					
(b)	1st	10.56	30-142	87.98	80.39±0.03 a	87.72±0.29^{a}	95.18±0.13^{a}	15.01±0.17 a	15.75±0.25^{a}
	S	-	142-249	-					
	2nd	65.89	249-377	349.2					
	3rd	23.11	377-516	458.9					
(c)	1st	11.14	30-139	66.4	72.52±0.14 b	75.74±0.16^{b}	83.05±0.20^{b}	10.88±0.37 b	15.02±0.14 a
	S	-	139-248	-					
	2nd	64.71	248-378	346.57					
	3rd	23.23	378-520	492.99					

(*) Values that have different letters in the same column present significant difference by Tukey's test ($p<0.05$). (*)Δm = mass loss (%), ΔT = temperature range (°C), Tp = peak temperature (°C), S = stability. To = ''onset'' initial temperature, Tp = peak temperature, Tc = ''endset'' conclusion temperature, ΔHgel = gelatinization enthalpy.

The thermal stability of starches is related to the ability of a sample to maintain its properties unchanged for a certain period of time under controlled heating conditions [8]. Although the temperature range at which stability occurred was similar between the samples, the maize starch tolerated higher temperatures prior to the start of its decomposition, compared to the ginger and yam starches. Corn starch has an amount of protein in its composition smaller than unconventional starches, this fact possibly gives it a greater thermal stability. The results for thermogravimetric analysis are in agreement with the literature [7-25].

The depolymerization of the starch occurs at temperatures above 300 ° C, where often the breakdown of amylopectin binds contribute to the decomposition of the starch granules at high temperatures [26-27-28].

Differential scanning calorimetry (DSC)

The endothermic events of the starch gelatinisation are shown in the DSC curves (Figure 2). The ginger starch (b) presented the highest gelatinisation temperature, which may have been related to its higher lipid and protein content (Table 2).

Figure 2: DSC curves of starches from maize (a), ginger (b) and yam (c)

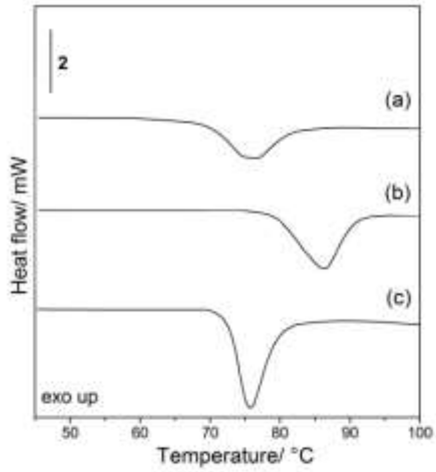


Table 2 presents the gelatinisation temperatures onset (To), peak (Tp) and endset (Tc) temperatures for the starches. Ginger starch (b) having the highest temperatures, followed by the yam (c) and maize starches (a), respectively. The ginger starch temperatures may have been related to its higher lipid and protein content. According to Copeland et al. [29], complexes with lipids can cause changes in the functional properties of starch, such as changes in pasting properties and increased gelatinisation temperatures.

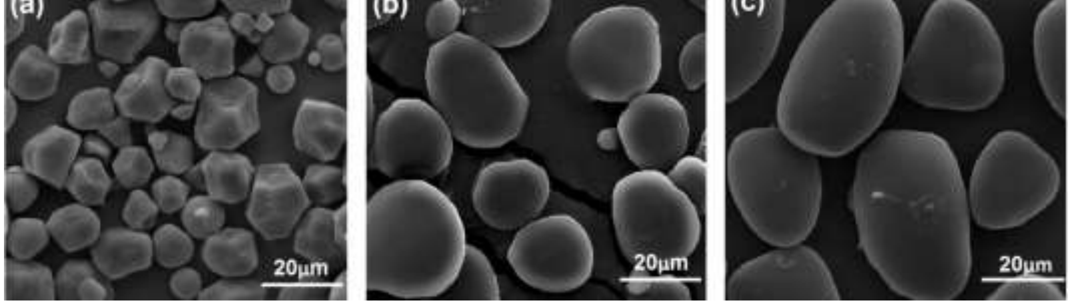
The maize starch showed the lowest gelatinisation enthalpy, while the ginger and white yam starches presented similar values. Jayakody et al. [22] found ΔHgel for yam starch of 18.98 J g^{-1}, while Hornung et al. [8] reported values of 15.6 J g^{-1}. Regarding ginger starch, gelatinisation enthalpy values of 16.6 J g^{-1} and 13.8 J g^{-1} were reported by Tetchi et al. [30] and Sukhija et al. [7], respectively.

According to Alcázar-alay and Meireles [31], the presence of lipids, which can complex to amylose, may hamper or reduce the swelling capacity of starch granules. Thus, high temperatures are required for gelatinisation. Hornung et al. [8] argue that some irreversible changes in the structure of the molecules occur during the formation of the paste; when the starch has a high amylose content the temperatures and enthalpies are consequently high because the resistance to gel formation is increased. In addition, the size of the granules also influences the gelatinisation properties. Therefore, larger granules tend to have higher gelatinisation temperatures and enthalpies. This may have contributed to the results that were found for the ginger starch in the present study.

Field emission gun-scanning electron microscopy (FEG)

The micro images of the maize, ginger and yam starches are presented in Figure 3.

Figure 3: Micro-images of starches from maize (a), ginger (b) and yam (c) (magnification of 5kx)

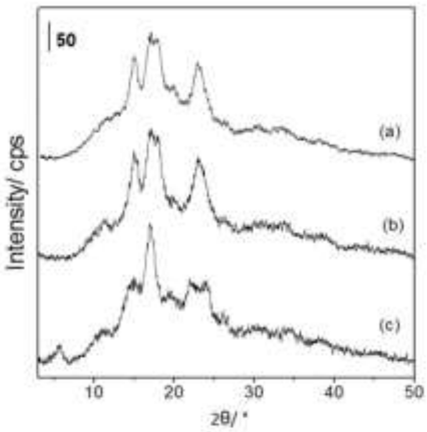


The average length of the ginger and white yam starch granules was 18.47±1.02 μm and 31.53±2.21 μm, respectively. The granules had an oval shape, with a smooth surface and no cracks. Similar results were obtained by other authors for the same vegetables [7-8-9-22-23-32]. The maize starch had the shortest length (14.35 μm) and a polygonal shape and a smooth surface, as reported by Cai et al. [33] and Utrilla-Coello et al. [34], which was similar to waxy maize starch [35]. The differences in the size of the starch granules affect their thermal characteristics and pasting properties, also influencing the quality of the final processed product as for example pasta. Therefore, particle size is a decisive factor in the result of biochemical reactions, as it significantly affects the physicochemical properties by increasing the surface area of the samples [36].

X-ray diffraction patterns and relative crystallinity (RC)

There were no pronounced differences between the diffraction patterns of the maize and ginger starches (Figure 4). Both samples presented diffraction peaks at 15º, 17º, 18º and 23º (2θ) and were classified as an A type diffraction pattern, as reported by Vieira and Sarmento [37].

Figure 4: Diffractograms of starches from maize (a), ginger (b) and yam (c)



The white yam starch exhibited a more intense peak at 17° (2θ) and weaker peaks at 5.8°, 15° and 22° (2θ). Therefore, it was classified as a C type diffraction pattern, similar to that found by Jayakody et al. [22]. The relative crystallinity values of the ginger and yam starch samples reached 31.54±0.57 % and 28.84±0.22 %, respectively, which were higher than those of the maize starch (21.06±0.23 %). Hornung et al. [8] have indicated that such variations may be related to the botanical source of the starch, its composition, moisture levels, and changes in the starch.

Pasting properties

Table 3 shows the pasting properties obtained by Rapid Visco Analyser equipment for the maize, ginger and white yam starches.

Table 3: Pasting properties of starches from maize (a), ginger (b) and yam (c)

Sample	Paste temperature (°C)	Peak Viscosity (cps)	Setback (cps)	Breakdown (cps)	Final Viscosity (cps)	Peak Time (s)
(a)	82.85±0.05^{b}	1970.0±3.42^{b}	699.5±1.23^{c}	766.1±0.08^{a}	1903.5±2.84^{c}	8.93±0.22^{c}
(b)	89.42±0.18^{a}	1287.5±9.19^{c}	77.5±1.41^{b}	1.5±0.71^{c}	2066.5±4.85^{b}	12.79±0.19^{a}
(c)	78.77±0.53^{c}	2272.0±2.83^{a}	2275.5±7.78^{a}	332.4±2.83^{b}	4220.5±0.71^{a}	9.55±0.35^{b}

Values that have different letters in the same column present significant difference by Tukey's test ($p < 0.05$).

The pasting temperatures of the samples corroborated the results obtained in the DSC analysis, where the highest temperatures were also recorded for the ginger starch. Madenine et al. [9] found a similar pasting temperature for ginger starch (88 °C), which is considered to be high when compared with other botanical sources such as potato and cassava [38]. In addition, low breakdown was found for this starch, indicating its low degree of disintegration and high pasting stability when heated and sheared [39]. According to Torres, Leonel and Mischan [38], ginger starch has homogeneous bond strengths, as well as a high degree of association between its components, which favours its high degree of resistance. Madeneni et al. [9] found that the high gelatinisation temperature of ginger starch, together with its mechanical resistance, causes it to resemble modified starches due to cross-linking. This makes it ideal for use as a texture agent in food manufactured at high temperatures, or as a constituent of sterilised products such as infant food and UHT products. On the other hand, in the present study the maize starch had the highest breakdown compared to the unconventional starches.

The yam starch presented the highest peak viscosity. According to Jayakody et al. [22], increases in viscosity during heating cycles are influenced by the leaching of the amylose, granular swelling, and the extent of friction between the swollen granules. Another influential factor is the size of the granules; larger diameters may occupy more volume and thus increase the viscosity. This corroborates with the larger diameters that were observed in the white yam starch and, therefore, the higher viscosity that was found relative to the ginger and maize starches. Zhu [21] concluded that most species of the genus Dioscorea present high viscosity under heat treatment and mechanical shear, suggesting that they may have potential use in some acidic food products that also require thermal processing.

Due to the high retrogradation of the yam starch, its final viscosity increased considerably. The setback value is related to the reorganisation or reassociation of the amylose and amylopectin molecules. The more amylose molecules that are available for the construction of the gel network, the greater the tendency to reassociation of slow enzymatic and the final viscosity. According to Hornung et al. [8], a high level of retrogradation in starch is desirable for breakfast cereals, parboiled rice and dehydrated potato mash because it aids the promotion digestion, with a moderate release of glucose into the bloodstream. Due to its low level of retrogradation, the ginger starch has greater potential to be used in frozen foods compared to maize starch.

Similar values for the pasting profile of the yam starch were found by Hornung et al. [8]. The latter study reported that starches with a greater tendency to retrogradation, such as those extracted from the Dioscoreaceae family, are considered to be resistant starches that can be used as the basis to produce bioplastic material because they have the capacity to form more cohesive films.

CONCLUSION

The properties of starches from unconventional sources differed from those of commercial maize starch. The aqueous extraction process of ginger and white yam starches resulted in a higher protein and ash content when compared to maize starch. Similar values for the thermal stability of ginger and yam starches were recorded, although they were lower than the value of maize starch. The higher transition temperatures and enthalpy of gelatinisation were found for ginger starch, which also had the largest granular diameter and greater relative crystallinity. Corn starch showed the highest brightness and the lowest tendency for red and yellow. The higher peak viscosity was observed for the white yam starch, as well as the lower pasting temperature, and it can be used in foods with a higher acidity, since they need a higher viscosity. The lowest breakdown was exhibited by ginger starch and stability of heating glue, which can applied in products that undergo high processing temperatures.

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