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Brazilian *Dioscoreaceas* starches

Thermal, structural and rheological properties compared to commercial starches

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Abstract Determination of the characteristics of native starches is crucial in order to select their best application in various industrial fields. Thus, two different types of non-traditional native starches from the *Dioscoreaceas* species (*Dioscorea* sp. and *Dioscorea piperifolia* Humb. var. Wild) were studied regarding their thermal, structural and rheological properties. The results were contrasted with traditional commercial starch sources (potato, cassava and corn). From the thermogravimetric results (TG/DTG), *D. piperifolia* starch obtained the highest thermal stability of the samples, except for potato starch. Furthermore, using differential scanning calorimetry and viscoamylograph profiles (RVA), it was found that the *Dioscoreaceas* starches presented a higher onset (T_o) temperature and susceptibility to retrogradation. They also showed lower values in relation to relative crystallinity, which was calculated from their X-ray patterns and tendency to white (L^*) colour. The shapes of the *Discoreaceas* starch granules were determined using electron microscopy; it was found that as the potato starch the *Dioscoreaceas* starches showed a wide range of particle size.

Keywords Yam native starch · Unusual starch source · Rheological behaviour · Thermogravimetric profile

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Introduction

In general, the food industry is the largest consumer of starch. However, starch is also used to process various products in the paper, chemicals, pharmaceutical and textile industries [1, 2]. Due to restrictions that have been mainly imposed by the food industry, and also because of the concerns about environmental issues, industries are increasingly interested in novel and underutilised sources of starch [3].

Starch is the most abundant carbohydrate reserve in plants and is also the main source of calories in the human diet. It is a non-toxic and renewable material. Products derived from starch have established applications in many fields. Starch granules are composed of two glucose polymers: amylose and amylopectin, which represent approximately 98–99 % of the dry total mass. Amylose is predominantly a linear polymer of *a* 1–4 linked glucose, whereas amylopectin is a highly branched polysaccharide consisting of *a* 1–4 linked glucose with *a* 1–6 linkages at the branch points [1].

The starch derived from cereal crops is the main form that is extracted and commercialised nowadays. Even though plants widely used by humans (root tubers, rhizomes, bulbs) are rich in starch, they are still underused. Brazil and others tropical countries have a large variety of tubers cultivars due to their mild climate [4]. Tubers from the *Dioscoreaceae* family are estimated to number approximately 600 species [5]. *Dioscorea* tuber crops are cultivated in many parts of Africa and South East Asia. In Brazil, the cultivation is mainly performed by traditional farmers for their own consumption and commercialised in the north-east of the country [5, 6]. In these tubers, the starch is the most abundant constituent, making up to 20–60 % of total content; it serves as a

Fig. 1 Photography of **a** *Dioscorea* sp. tuber, **b** *D. piperifolia* Humb. var. Wild. tuber

sustainable source of starch for food and other industrial uses [7–9].

Thermal analysis can be used to show the behaviour of starch granules when they are heated. For example, the differential scanning calorimetry (DSC) technique can be used to investigate the phase transitions of starch/water systems (gelatinisation process) and to estimate transition enthalpies [10]. Furthermore, the structural configuration, paste and colour properties of starch play an important role in the development of food processing and new products [11].

In this context, there is a need to investigate non-traditional starch sources which may be suitable for industrial use as additives to fulfil other specific purposes. Thus, the objective of this research was to study the thermal, rheological and structural properties of starch from *Dioscoreaceae* family tubers (*Dioscorea* sp. and *Dioscorea piperifolia* Humb. var. Wild) and compare them with commercialised starches.

Materials and methods

The starches (500 g), from cassava (*Manihot esculenta*), corn (*Zea mays*) and potato (*Solanum tuberosum*) were purchased commercially in the city of Curitiba, Paraná,

Brazil. The tubers of white yam (*Dioscorea* sp.) and aerial yam (*D. piperifolia* Humb. var. Wild.), which are presented in Fig. 1, were harvested from a crop area located in Aripuanã, Mato Grosso, Brazil. The tuber starches were obtained using the method described in a previous study [12]. The codification of the samples was as follows: samples of the native starches [white yam *D.* sp.—(a); aerial yam *D. piperifolia*—(b)]; commercial starches [potato—(c); cassava—(d) and corn—(e)].

To obtain the thermal profile of the samples, in terms of mass loss under a controlled programmed temperature, the analysis was performed using TGA-50 thermal analysis system equipment (Shimadzu, Japan). All the mass loss percentages and the derivative thermogravimetric results were determined using TA-60 WS data analysis software [1, 2, 13].

The DSC analysis was performed using a thermal analysis system (TA-Instruments, DSC-Q200 model, USA). The system was previously calibrated with 99.99 % purity indium, with a melting point of $T_p = 156.6$ °C, $\Delta H = 28.56$ J g^{-1}. The thermal profiles were recorded under an air flow of 50 mL min^{-1} with a heating rate of 10 °C min^{-1} and the samples, which weighed about 2.5 mg, were placed in sealed aluminium crucibles. A water:starch w/w (4:1) mixture was prepared and

Fig. 2 TG and DTG curves: *native* starches. **a** *Dioscorea* sp., **b** *D. piperifolia*; commercial starches, **c** potato, **d** cassava, **e** corn, respectively
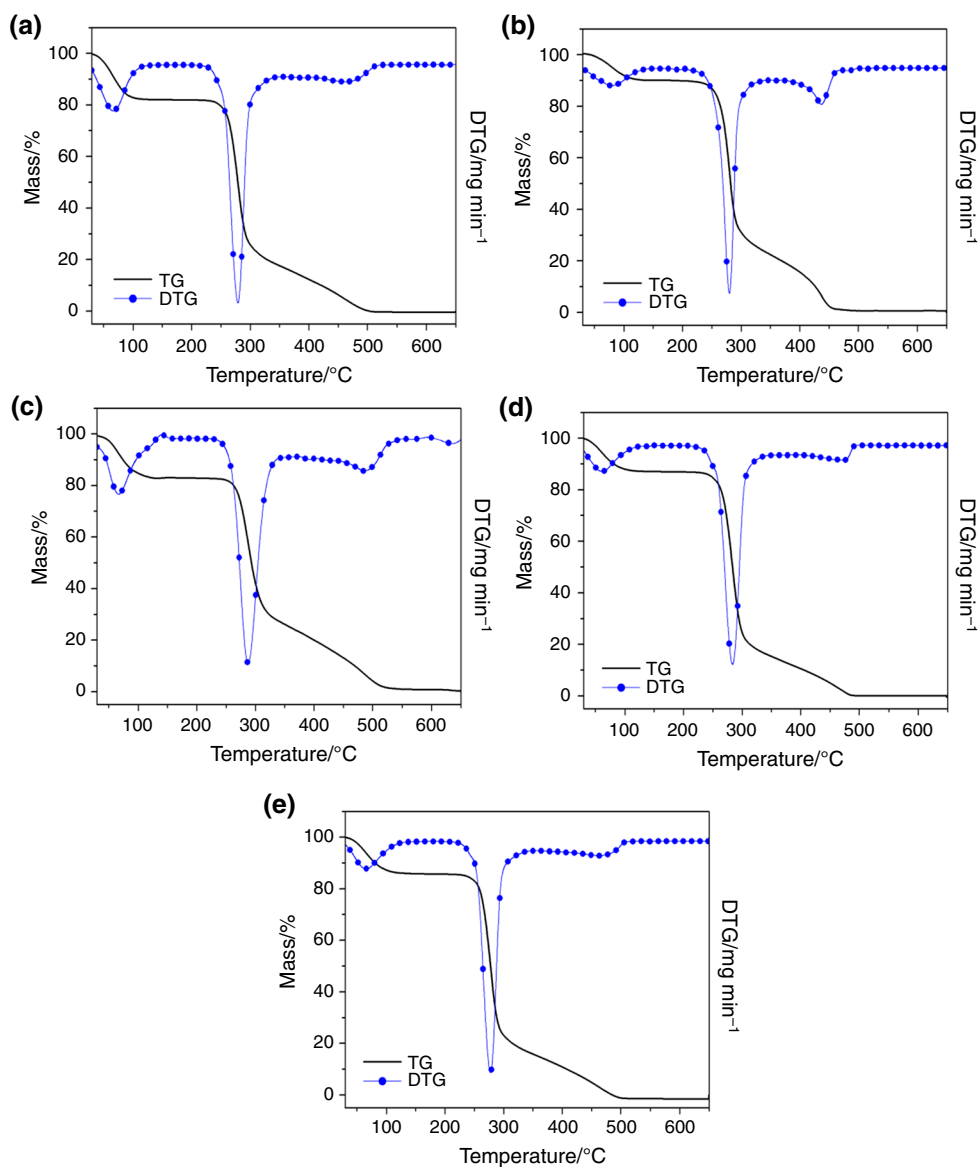
maintained for 60 min in order to equilibrate the moisture content [1, 2, 13].

In order to study the pasting properties of the samples, the viscoamylograph profiles were obtained using a rapid viscometer (Newport Sci., RVA-4 model, Australia). A suspension of 2.24 g of starch in 25.76 g of distilled water underwent a controlled heating and cooling cycle following the methodology described in the literature [14, 15].

The morphology of the starch granules and their surface area were examined using a scanning electron microscope (Tescan, VEGA 3, Kohoutovice, Czech Republic) under an acceleration voltage of 15 kV and magnification of 1000× after the samples had been coated with plasma of gold and palladium [1, 2, 16]. The average size of the starch granules was calculated using ImageJ free software (version 1.49v).

The X-ray diffraction powder patterns (XRD) of the samples were investigated using an X-ray diffractometer (Rigaku, Ultima 4 model, Japan), and the degree of relative crystallinity was quantitatively estimated using Eq. 1. The method followed was as described in the literature [14, 17–19]

$$X_c = \frac{A_p}{(A_p + A_b)} \times 100 \qquad (1)$$

where X_c relative crystallinity, A_p peak area, A_b base area.

A MiniScan EZ 4500L spectrophotometer (Hunter Inc., USA) was used to determine the colour parameters of the starch samples (L^*, brightness, which ranges from 0 (black) to 100 (white); a^*, positive values (red) and negative (green) and b^*, positive (yellow) to negative (blue)) [1, 2, 14, 20].

Table 1 TG and DTG results: *native* starches

Samples	Step	TG results	DTG results	
		$\Delta m/\%$	$\Delta T/°C$	$T_p/°C$
a	1st	17.95	30–103	66.27
	Stability	–	103–227	–
	2nd	65.42	227–336	279.05
	3rd	15.99	336–516	457.67
b	1st	9.85	30–108	79.13
	Stability	–	108–235	–
	2nd	67.79	235–372	280.06
	3rd	21.47	372–458	453.07
c	1st	15.89	30–105	72.54
	Stability	–	105–242	–
	2nd	63.41	242–415	288.32
	3rd	19.87	415–532	479.87
d	1st	12.87	30–102	65.36
	Stability	–	102–216	–
	2nd	72.46	216–392	283.61
	3rd	14.39	392–498	481.55
e	1st	12.11	30–101	65.67
	Stability	–	101–232	–
	2nd	72.37	232–360	280.60
	3rd	14.13	360–506	481.82

a *Dioscorea* sp., b *D. piperifolia*; commercial starches, c potato, d cassava, e corn, respectively

Δm mass loss (%), ΔT temperature range, T_p peak temperature
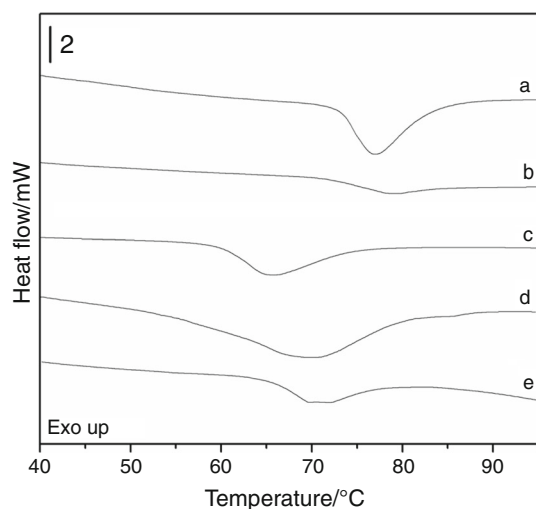

Fig. 3 DSC curves *native* starches. *a Dioscorea* sp., *b D. piperifolia*; commercial starches, *c* potato, *d* cassava, *e* corn, respectively

All the results were analysed for variance (ANOVA). Duncan's test was used to compare the means of the samples at 95 % confidence level ($p < 0.05$) utilising STATISTICA 7.0 software (StatSoft Inc., Tulsa, OK, USA).

Results and discussion

The thermal decomposition of the starch samples occurred in three main steps, as can be observed in the curves shown in Fig. 2. The first step of mass loss, which was associated with the first peak followed by a stability step, was attributed to dehydration due to the water loss of the starch granules. The decomposition and oxidation of the organic matter and consecutive reactions were associated with the second and third steps, respectively [1, 13, 20, 21]. Similar results were observed in previous studies [1, 16, 20–25].

Differences were observed regarding the derivative thermogravimetric profiles (DTG curves) of the studied samples. In relation to the first step (dehydration), it could be seen that the samples of white yam starch (a) and potato starch (c) presented a well-defined peak compared to the other samples; this can be attributed to the higher values for moisture content (a—17.95 %; c—15.89 %), Table 1. The moisture content of the samples, b, d and e, was from 9 to 12 %. Low moisture content can increase the stability of starches, and it also facilitates transport and storage [26]. Furthermore, the third peak (oxidation) in the DTG curve (Fig. 2) of sample b (aerial yam) was different from the others. This could have been related to the slightly high depolymerisation (which occurs above 300 °C under an oxidative atmosphere) of the starch molecules [21, 22, 27] compared with the other samples. Table 1 shows the third event for sample b, which presented the highest mass loss (21.47 %) of all samples that were analysed.

The aerial yam starch (sample b), as listed in Table 1, presented a slightly higher range of stability step (108–235 °C) when compared to the other samples, except for the potato starch. Thus, sample b could be considered a starch more stable to thermal degradation when compared to cassava and corn starches.

The results calculated from the TG/DTG curves are listed in Table 1. The residue corresponding to the mineral content of the samples was 0.64, 0.89, 0.83, 0.98 and 1.09 % for samples a, b, c, d and e, respectively.

The DSC technique was used to study the gelatinisation process of the different starch samples. The profiles of the curves are depicted in Fig. 3, and Table 2 shows all the calculated results, which were carried out in triplicate.

Figure 3 shows the very different endothermic events (calorimetric curves) that occurred for the analysed samples. The initial temperatures, which were attributed to the change of the baseline, were quite similar for samples a and b (*Dioscoreaceas* starches) and also for samples c and d (potato and cassava).

The gelatinisation process leads to irreversible changes in the structure of starch molecules, such as granules

Table 2 DSC, XRD, SEM results: *native* starches

Sample	DSC results				XRD results	SEM results
	T_o/°C	T_p/°C	T_c/°C	ΔH/J g^{-1}	Degree of relative crystallinity (%)	Average size (μm)
a	73.35 ± 0.01[c]	76.92 ± 0.11[d]	80.99 ± 0.07[c]	15.56 ± 0.20[c]	16.78 ± 0.02[b]	29.57 ± 4.33[c]
b	72.12 ± 0.01[c]	78.88 ± 0.03[d]	82.61 ± 0.07[c]	10.05 ± 0.08[b]	11.87 ± 0.05[a]	31.14 ± 7.31[d]
c	60.19 ± 0.02[a]	65.49 ± 0.01[a]	74.27 ± 0.03[a]	12.07 ± 0.10[c]	20.58 ± 0.02[c]	35.55 ± 16.80[e]
d	63.71 ± 0.05[a]	70.51 ± 0.31[b]	79.40 ± 1.03[b]	9.63 ± 0.63[a]	24.38 ± 0.03[d]	13.83 ± 2.67[b]
e	67.44 ± 0.03[b]	73.23 ± 0.10[c]	78.81 ± 0.46[b]	8.49 ± 0.25[a]	26.81 ± 0.03[d]	12.95 ± 2.89[a]

a *Dioscorea* sp., b *D. piperifolia*; commercial starches, c potato, d cassava, e corn, respectively

T_o "onset" initial temperature, T_p peak temperature, T_c "endset" final temperature, ΔH_{gel} gelatinization enthalpy

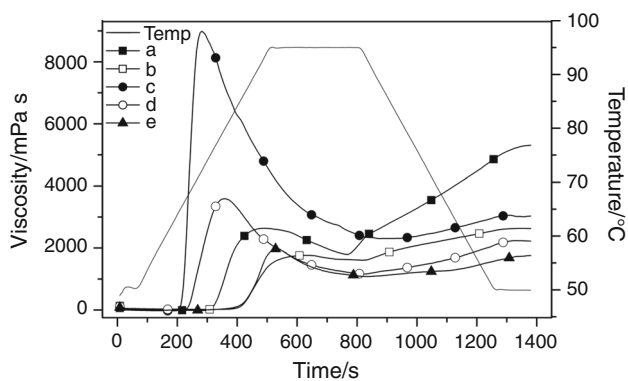Means followed by the same letter do not differ significantly by the Tukey test ($p < 0.05$)

Fig. 4 RVA profiles: *native* starches. **a** *Dioscorea* sp., **b** *D. piperifolia*; commercial starches, **c** potato, **d** cassava, **e** corn, respectively

swelling and crystallite melting. As can be seen from the results in Table 2, the native starches from the *Dioscorea* species presented the highest values for the evolved temperatures of the gelatinisation process (T_o, T_p, T_c). These values were expected, due to the botanical sources of the yam starches which contain approximately 15–30 % of amylose of its total starch (60–85 %) in dry basis [23]. When starch contains a high amylose content, it requires higher temperatures and a greater amount of energy for gelatinisation to occur. In solution, the linear structure of

amylose assists in rapidly aligning with itself, resulting in more extensive hydrogen bonding and high gel strength. Thus, a high amount of energy is required to break these bonds and gelatinise [28]. Similar results were obtained in previous studies of different species of yam starches (*Dioscoreaceas*) [25, 29–31].

In the present study, the *Discorea* starch from the white yam cultivar (sample a) showed the highest ΔH value (15.56). A previous study obtained even higher ΔH values (17.32–18.98 J g^{-1}) for seven types of *Discorea* starches from Sri Lanka. Campbell et al. [32] studied the influence of the variation of the size of starch granules on its functional properties. They found that starches with larger sized diameter particles can result in higher temperatures for DSC values. The *Dioscoreaceas* starches (samples a and b) had shown an average granule size of 29.57, 31.14 μm, respectively (Table 2), as well as higher ΔH values, as related above. Regarding the ΔH values obtained for sample b (*D. piperifolia*), this native starch consumed less energy to form the starch gel in comparison with the potato sample (35.55 μm).

One of the most important characteristics when recommending the technological application of amylaceous materials is viscosity. Viscosity curves can show the behaviour of the starch paste during heating and cooling

**Table 3** RVA results: *native* starches

Sample	Pasting temperature/°C	Viscosity peak/mPa s	Peak time/s	Setback/mPa s	Breakdown/mPa s	Final viscosity/mPa s
a	76.70 ± 0.28[c]	2383.50 ± 0.71[c]	420.05 ± 0.07[c]	3943.00 ± 2.83[a]	1007.50 ± 6.36[c]	5324.50 ± 4.95[a]
b	74.43 ± 0.04[c]	1762.60 ± 0.85[e]	628.28 ± 0.11[a]	1024.00 ± 1.41[b]	161.50 ± 2.12[e]	2628.50 ± 2.12[c]
c	64.87 ± 0.01[a]	8982.30 ± 0.42[a]	280.00 ± 1.41[e]	723.73 ± 0.18[d]	6675.05 ± 0.21[a]	3031.12 ± 0.16[b]
d	66.75 ± 0.07[a]	3597.50 ± 2.12[b]	348.55 ± 0.78[d]	833.75 ± 2.47[c]	2331.15 ± 1.63[b]	2054.00 ± 5.66[d]
e	70.58 ± 0.67[b]	1972.40 ± 0.57[d]	531.25 ± 1.06[b]	681.10 ± 0.14[e]	895.15 ± 0.21[d]	1758.05 ± 0.07[e]

a *Dioscorea* sp., b *D. piperifolia*; commercial starches, c potato, d cassava, e corn, respectively

mPa s millipascal-second, *s* seconds

Averages followed by the same letters in the same column do not differ statistically by Tukey test ($p < 0.05$)

Fig. 5 SEM images (with magnifying ×1000): *native* starches. **a** *Dioscorea* sp., **b** *D. piperifolia*; commercial starches, **c** potato, **d** cassava, **e** corn, respectively
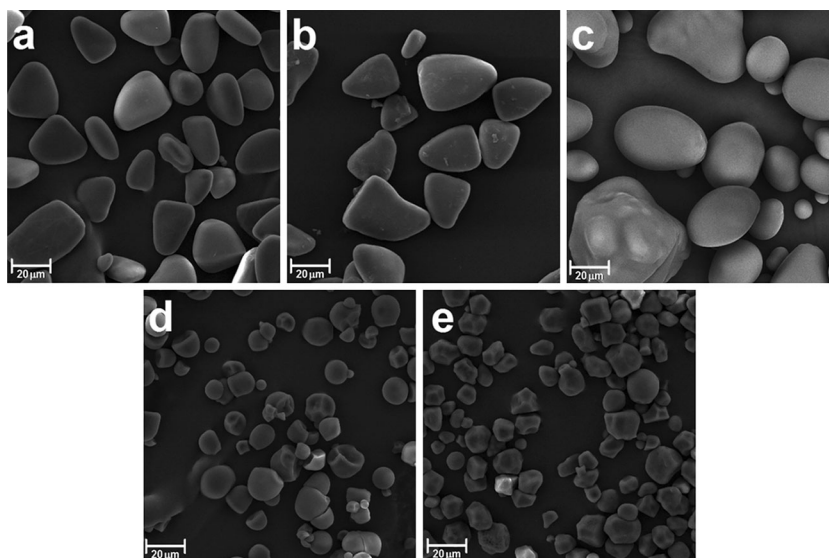

[33]. The pasting properties for the starch samples obtained by the RVA profiles are presented in Fig. 4, and the main results are listed in Table 3.

The pasting temperatures obtained by RVA corroborated the onset temperatures of gelatinisation that were registered by the DSC analysis. The higher values recorded by RVA are due to the fact that this technique is less sensitive in detecting when starch granules begin to disrupt their configuration [33]. Compared to the commercial samples, the higher pasting temperatures presented by the yam starches (samples a and b) may have been related to a high resistance to the swelling and rupture of the starch granules. All the results for peak viscosity, peak time, setback, breakdown and final viscosity were significantly different between the samples. In spite of the fact that sample c (potato) showed a high value for peak viscosity (8982.30 ± 0.42 mPa s), the highest value in terms of final viscosity was achieved for the white yam starch (sample a—5324.50 ± 4.95 mPa s). This result obtained for the white starch suggests that the native starch would be a better alternative than potato starch to be used in the food industry as a basis for soups, gravies, sauces, baby foods, etc. [34]. Highly viscous starch presents a heavy body, shorter texture and a lower level of breakdown in cooking, thereby functioning as a better food system stabiliser [34]. The setback parameter can provide an idea of the proportion of the partial association with the amylose and amylopectin macromolecules after the gelatinisation process [35]. This association expels the water molecules from the gel, forming hard pastes in a process known as retrogradation [35]. Starches that have a higher tendency to retrogradation (setback values), such as those presented by the

Dioscoreaceas starches (samples a and b), are considered to be resistant starches [36]. Starches that present such results can be applied as a basis for bioplastic material due to their ability to form cohesive films [37]. Different results for setback are found in the literature regarding *Dioscorea* starch sources [7, 23, 38]. These differences can be attributed to the botanical origin, chemical composition, the method used and the conditions of analysis [33].

Figure 5 shows the morphological images of the studied samples acquired by SEM. The micrographs showed that the shapes and sizes of the starches granules for the analysed samples differed significantly between themselves. The starch granules from samples a, b and c range from large to medium size, and samples a and b were predominantly triangular in shape. The results obtained for all the samples were similar to those in previous studies [3, 7–9, 21, 23, 25]. Based on the micrograph images of the starch granules, the average size was calculated and the results are listed in Table 2. In terms of these results, all samples analysed presented significant differences. Sample a showed a range between 24 and 40 μm with an average size of 29.57 μm. Sample b ranged between 24 and 41 μm with an average size of 31.14. Determining the shape and size of starch particles is important in order to understand their technological behaviour [39]. For example, in the pharmaceutical industry the shape of starch granules influences their density during packing in a tableting machine or in filling gelatine capsule shells [39]. The pharmaceutical industry is interested in starch granules that show a wide range of particle size, such as the ones obtained for samples a, b and c (*Dioscoreaceas* and potato starches), which implies better technological performance

Fig. 6 XRD diffractograms: *native* starches. **a** *Dioscorea* sp., **b** *D. piperifolia*; commercial starches, **c** potato, **d** cassava, **e** corn, respectively
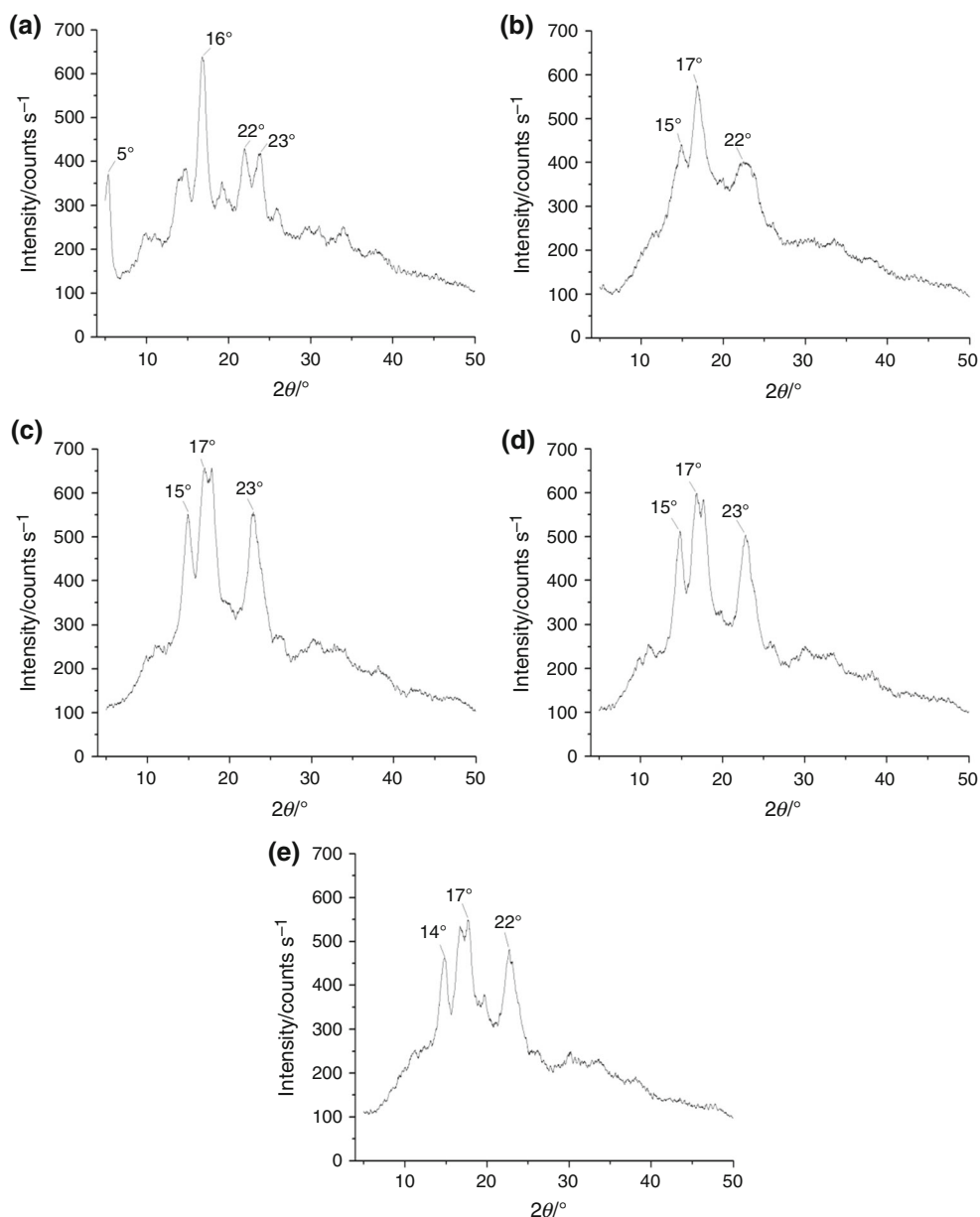
[39]. The range of particles size for potato, cassava and corn starch were 11–49, 10–13 and 9–17 μm, respectively.

Observing the diffraction patterns (Fig. 6), regarding the main peaks the samples of the *Dioscoreaceas* species, were different from the commercial starch samples. The diffraction patterns of samples a and b were very similar to the ones obtained in previous studies of *Dioscorea permsimilis* and *Dioscorea opposita* [8, 40]. However, sample b showed a different pattern from a sample of the same species presented in the literature [7]. These differences can be attributed to the origin of the cultivars (climate, region, period of collection), the methodology used and the data analysis. The patterns can be better analysed by the results as presented in Table 2. Depending on the strongest

diffraction peaks that are registered, starch can be categorised as follows: type A (main peaks at 15°, 17°, 18° and 20° into 2θ); type B (5°, 6°, 15°, 17°, 18° and 23°) or type C (a mixture of types A and B = 5.5°, 15°, 17°, 22° and 23°), the last one is normally attributed to rhizome starches [1, 3]. Therefore, in this study the main peaks into 2θ presented for samples a (5°, 16°, 22° and 23°) and b (15°, 17° and 22°) can be classified as type C starches as well as the ones obtained for the commercial samples (Fig. 6).

The results for the degree of relative crystallinity showed that samples a and b were significantly different from the commercial starch samples. Furthermore, these *Dioscorea* starch samples showed the lowest values for relative crystallinity, which were 16.78 and 11.87 %,

Table 4 Colourimetry results: *native* starches

Sample	Colour parameters results		
	L^*	a^*	b^*
a	85.88 ± 0.42^b	-0.20 ± 0.07^a	1.57 ± 0.30^a
b	72.36 ± 0.18^a	10.10 ± 0.03^c	35.65 ± 0.14^d
c	93.16 ± 0.59^d	-0.07 ± 0.03^b	3.85 ± 0.09^b
d	88.84 ± 0.33^c	0.27 ± 0.21^d	2.08 ± 0.31^a
e	93.41 ± 0.56^d	0.84 ± 0.04^e	5.51 ± 0.05^c

a *Dioscorea* sp., b *D. piperifolia*; commercial starches, c potato, d cassava, e corn, respectively

Averages followed by the same letters in the same column do not differ statistically by Tukey test ($p < 0.05$)

respectively. These results are different from the data obtained in the literature [7, 41] for *D.* sp. (35 %) and for *D. piperifolia* (32.06 %). These differences were mainly due to the composition of the starch, the moisture content and the botanical source [8]. These low values can be attributed to high amylose content [23]. Starches composed of large amounts of amylose molecules have great potential for the development of resistant edible films [31], and high amylose starches are considered to be healthier for human consumption because of their resistance to digestion [36, 42]. Considering the results for low relative crystallinity and high setback for samples a and b, the amount of amylose could contribute for the differences between those starches and the commercial samples. It is also relevant to mention that a low degree of relative crystallinity of a starch can increase the enthalpy (ΔH) of the gelatinisation process [43], as shown by sample a (*D.* sp.—white yam) (Table 2).

The results of the colour properties are presented in Table 4. Visually, all the starch samples were white, with the exception of the starch from sample b (*D. piperifolia*), which was yellow. The visual colour observation of sample b was confirmed by the results for the b^* parameter, which registered a positive value of $+35.65$ (yellow) and the lowest value for brightness (L^*) of $+72.36$. Sample a (*D.* sp.) also presented a lower value for the L^* parameter when compared to the commercial starch samples although it showed the least tendency to yellow ($b^* = 1.57$) of all the samples. Similar results were found for these cultivars in previous studies [7–9, 23]. Regarding sample (b) from the *D. piperifolia* starch, the presence of pigments like carotene in its tuber [44] carried over to the starch, which may have been responsible for its colour properties. Starches that bring pigmentation from their source can be applied in product formulations without a negative impact on their colour characteristics because the crucial factor is the amount that is applied [45].

Conclusions

The TG/DTG curves showed that the yam starches obtained a higher stability step compared to the commercial starch samples of cassava and corn. The DSC and RVA results verified the initial temperature of the gelatinisation process. It was possible to observe that the *Dioscoreaceas* samples showed a strong tendency to retrogradation (RVA—setback) and that the starch sample from *D.* sp. had the higher value for gelatinisation enthalpy. The XRD crystallinity patterns of type C starches were observed for the *Dioscoreaceas* samples, which also presented the lowest values for relative crystallinity. All the colour results for the samples were significantly different, and the aerial yam was the most coloured sample, tending to yellow. From the characteristics displayed in this study, *Dioscoreaceas* starches could be useful as a raw material for different commercial applications.

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