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The effect of microwave radiation on some thermal, rheological and structural properties of cassava starch

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Abstract Three samples of native or untreated cassava starch were exposed to microwave radiation for periods of 5, 10 or 15 min. The temperature of each sample was measured immediately after each exposure time and the temperature of the samples was around 135 °C. The samples were cooled to room temperature and maintained in a desiccator with anhydrous calcium chloride. All the samples were analysed by thermogravimetry-derivative thermogravimetry, differential scanning calorimetry (DSC), rapid viscoamylographic analysis (RVA), X-ray diffraction powder patterns, non-contact atomic force microscopy and colour characteristics by reflectance spectrophotometry. The thermal behaviour, gelatinisation temperatures, enthalpy and pasting properties were determined. Relative to the time of microwave exposure, the peak viscosity and gelatinisation (RVA and DSC) increased slightly after 5 min, and, after 10 and 15 min, it decreased considerably. The degree of relative crystallinity (%) decreased, while the average roughness increased. The reflectance spectrophotometry showed that microwave action occured quickly and progressively, causing colour changes (mainly with trends to yellow) and very small differences to the starch samples that were heated at controlled temperature in a conventional oven.

Keywords Microwave · Thermal analysis · Gelatinisation · Modified starch · Enthalpy

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Introduction

Starch is the most important storage reserve of carbohydrates in plants. Starch granules are made up of glucose polymers, named amylose and amylopectin, and they are found inside vegetable cells, from where they are extracted and can be treated for several industrial applications. These glucose polymers that constitute starch come in two molecular forms, linear and branched. The first is referred to as amylose and the latter as amylopectin. Amylose is mostly a linear chain and typically consists of up to 3,000 anhydroglucose units (AGU) primarily interconnected by α-1,4 glycosidic linkages and, reportedly, a few branched networks. Amylopectin is a large-branched polymer with α-1.4 linkages that serve as the backbone and α-1.6 bridges at the branching points [1–3]. Starches are of great value for the food industry, but have some limitations in their native form. Some of these constraints are their insolubility in cold water, low stability to freeze-thawing, and synaeresis, that in some cases makes them difficult to use [4].

Microwaves comprise electromagnetic radiation in the frequency range of 300 MHz–300 GHz. On exposure to microwaves, the charged or polar particles tend to align themselves with the electric field components of the microwaves, which rapidly reverse their direction. As the polar or charged particles in a reaction medium fail to align themselves as fast as the direction of the electric field of microwaves changes, friction is created to heat the medium [5].

The microwave creates heat deep inside the materials being processed as a result of rapid alterations of the electromagnetic field at high frequency. This property results in a much shorter process time, higher yield and better quality of products, than that obtained by conventional processing techniques [6].

According to the literature, microwave irradiation seems applicable to starch processing, but, thus far, it has not been used on a commercial scale. The suggestion of using microwave ovens for this purpose seems promising, but requires a detailed study of the effect of microwave processing on starch. Several observations in the literature on the modification of tuber and cereal starches by microwave irradiation are similar to observations on the effects of heat-moisture treatment, which is the treatment that typically employs prolonged heating of starch at a temperature range of 90–100 °C, with limited quantities of moisture that are insufficient to gelatinise the starch [7, 8].

Microwave heating of foods is a thermal treatment that offers many advantages in processing, including less startup time, faster heating, energy efficiency, space savings, precise process control, selective heating and final products with improved nutritive quality [9].

However, baking using microwave energy has been limited, due to poor final product quality compared to products baked using conventional energy sources. The causes for these differences in quality are not fully understood as yet, but the specific changes to starch granules are generally thought to contribute to the poor texture of the baked product [10].

The objective of this work was to apply microwave heating at different times (5, 10 and 15 min) to untreated cassava starch and to comprehensively investigate the changes, using the following thermoanalytical techniques: thermogravimetry-derivative thermogravimetry (TG/DTG), differential scanning calorimetry (DSC), rheological technique (rapid viscoamylographic analysis RVA), X-ray diffraction powder patterns (XRD), non-contact atomic force microscopy (NC-AFM) and colour characteristics by reflectance spectrophotometry.

Materials and methods

All the starch samples used in this study were of commercial grade (Yoki – lot. MT-55; moisture content until 18 %). The thermogravimetric curves (TG) were obtained with the thermal analysis system TGA-50 (Shimadzu, Japan), where the samples were heated from 35 to 600 °C using open alumina crucible with approximately 7.0 mg of the sample under a synthetic air flow of 150 mL min^{-1} at a heating rate of 10 °C min^{-1}. The instrument was preliminarily calibrated with standard weight and with standard calcium oxalate monohydrate. All mass loss percentages were determined using TA-60 WS data analyses software. The derivative thermogravimetric curves (DTG) were the first derivative of TG curve that were calculated. At the same time, as the microwave treatment, a portion of untreated sample was heated in a conventional oven at 150 °C for 2 hours.

The DSC curves were obtained using the thermal analysis system model DSC-Q200 (TA-Instruments, USA), in two different temperature programmes. The first was carried out to follow the thermogravimetric process, according to the instrumental conditions: the samples were heated from 35 to 600 °C using aluminium crucible with perforated cover and approximately 7.0 mg of the sample under a synthetic air flow of 150 mL min^{-1} at a heating rate of 10 °C min^{-1}.

In the second temperature programme, the DSC curves were carried out with the objective of studying the gelatinisation process: the curves were recorded under an air flow of 50 mL min^{-1}, heating rate of 5.0 °C min^{-1} and samples weighing about 2.5 mg. A 4:1 (water:starch w/w) mixture was prepared and maintained for 60 min to equilibrate the moisture content. The aluminium crucibles were hermetically sealed and this was carried out to study the gelatinisation process. The instrument was previously calibrated using Indium 99.99 % purity, $T_p = 156.6$ °C, $\Delta H = 28.56$ J g^{-1}.

The pasting properties of the samples were determined using RVA-4 (Newport Sci., Australia). A suspension of 3 g (6 % moisture) of starch in exactly 25 g of distiled water underwent a controlled heating and cooling cycle under constant shear where it was held at 50 °C for 2 min, heated from 50 to 95 °C at 6 °C min^{-1}, and held at 95 °C for 5 min, cooled to 50 at 6 °C min^{-1} and held at 50 °C

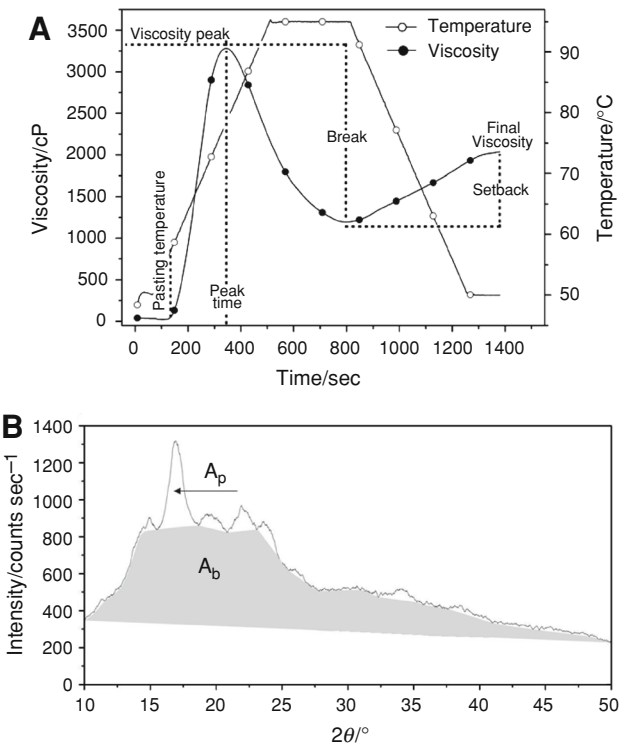

Fig. 1 **a** Parameters obtained through the RVA curves, **b** parameters for calculating the relative degree of crystallinity

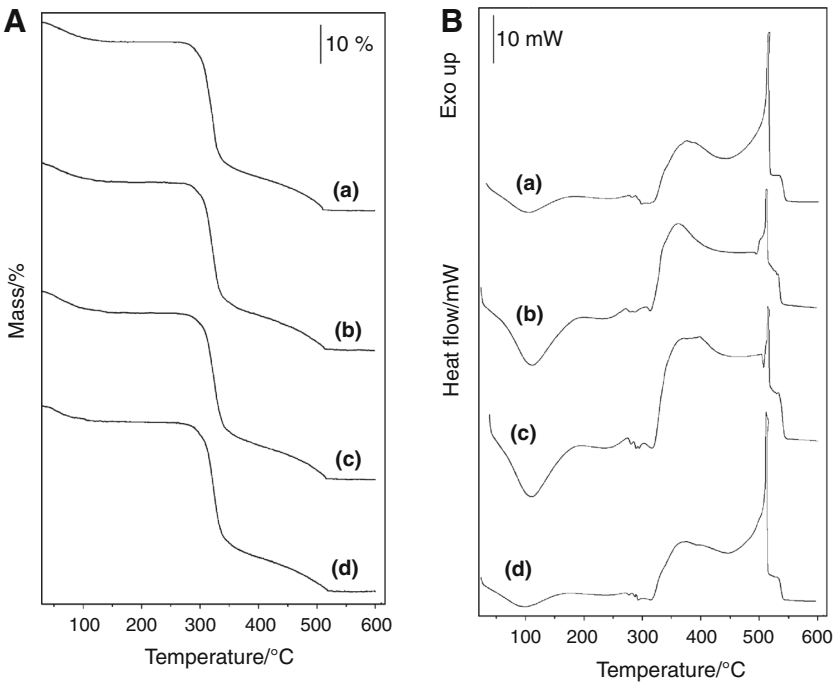

Fig. 2 **A** TG curves of **a** native cassava starch, 7.26 mg; **b** cassava starch exposed 5 min to microwave radiation, 7.24 mg; **c** cassava starch exposed 10 min to microwave radiation, 8.81 mg; and **d** cassava starch exposed 15 min to microwave radiation, 6.47 mg; and **B** DSC curves of **a** native cassava starch, 7.09 mg; **b** cassava starch exposed 5 min to microwave radiation, 7.28 mg; **c** cassava starch exposed 10 min to microwave radiation, 6.92 mg; and **d** cassava starch exposed 15 min to microwave radiation, 6.85 mg

Table 1 TG and DTG results of: (a) native cassava starch, (b) cassava starch exposed 5 min to microwave radiation, (c) cassava starch exposed 10 min to microwave radiation, and (d) cassava starch exposed 15 min to microwave radiation

Samples	TG results		DTG results	
	Step	Δm/%	ΔT/°C	T_p/°C
(a)	1st	10.60	30–146	56.68
	Stability	–	146–274	–
	2nd	72.88	274–413	341.69
	3rd	16.25	413–541	513.63
(b)	1st	10.65	30–145	58.59
	Stability	–	145–276	–
	2nd	73.80	279–425	342.05
	3rd	15.26	425–543	513.50
(c)	1st	10.07	30–149	59.21
	Stability	–	149–270	–
	2nd	73.37	270–420	342.40
	3rd	16.21	420–545	514.24
(d)	1st	10.91	30–144	66.71
	Stability	–	144–262	–
	2nd	72.77	262–423	340.08
	3rd	15.52	423–553	511.87

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

for 2 min. Figure 1a shows a representative RVA analysis of starch.

At the beginning of heating, swelling of the starch granules occurs, which causes an increase in the viscosity slurry (time and temperature pasting) when the polymers with low molecular weight (amylose molecules) begin to be leached from the granules. The viscosity peak is observed when the granules are completely swollen, and while maintaining the pasting temperature at 95 °C under constant agitation. The granules begin to break down and solubilisation of the polymers provides a reduction in viscosity (break). The analysis follows, with a cooling cycle that causes a reorganisation of some amylose and amylopectin polymers, increasing opacity and viscosity of the paste in a process called setback [11], which occurs due to the strong tendency to form hydrogen bonds between adjacent molecules.

The micro-images of each sample with high resolution were observed using an atomic force microscope SPM-9600 (Shimadzu, Japan), by the non-contact method (NC-AFM). This technique allowed us to observe the surface of the studied starches and it was possible to calculate the average diameter and the average roughness of the samples [12, 13].

XRD were obtained using an X-ray diffractometer mod. Ultima 4 (Rigaku, Japan), employing Cu Kα radiation ($\lambda = 1.541$ Å) and settings of 40 kV and 20 mA. The scattered radiation was detected in the angular range of 5–50° (2θ), with scanning speed of 8° min^{-1} and a step of 0.06°.

The degree of relative crystallinity was quantitatively estimated, following the method described in the literature [14, 15]. A smooth curve, with connect peak baselines, was computed and plotted on the diffractograms (see Fig. 1b).

The area above the smooth curve was taken as the crystalline portion, and the lower area between the smooth

Table 2 DSC results from decomposition and gelatinisation of: (a) native cassava starch, (b) cassava starch exposed 5 min to microwave radiation, (c) cassava starch exposed 10 min to microwave radiation, and (d) cassava starch exposed 15 min to microwave radiation

Samples				
	(a)	(b)	(c)	(d)
Thermal decomposition				
1st Peak				
T_o/°C	33.3 ± 1.53^b	52.3 ± 2.52^a	36.6 ± 2.08^b	23.3 ± 3.05^c
T_p/°C	101 ± 2.66^b	112.3 ± 3.51^a	103 ± 2.65^b	91.12 ± 0.83^c
T_c/°C	111 ± 3.60^b	118 ± 2.64^b	163 ± 2.65^a	112.3. ± 2.52^b
ΔH/J g^{-1}	302.3 ± 2.52^d	312.2 ± 2.99^c	444.3 ± 4.51^a	414.7 ± 4.51^b
–	(endo)	(endo)	(endo)	(endo)
2nd Peak				
T_o/°C	318.3 ± 3.05^b	319.6 ± 2.51^{ab}	326.6 ± 2.08^a	322.6 ± 2.52^{ab}
T_p/°C	370.3 ± 4.04^a	352.3 ± 2.52^b	355.6 ± 4.04^b	361.6 ± 2.52^{ab}
T_c/°C	384 ± 3.60^b	405.3 ± 4.73^a	393.3 ± 3.05^b	392.6 ± 2.52^b
ΔH/J g^{-1}	544.6 ± 4.51^a	448.3 ± 4.04^b	406.3 ± 5.51^c	556 ± 5.57^a
–	(exo)	(exo)	(exo)	(exo)
3rd Peak				
T_o/°C	488.7 ± 3.51^b	490.3 ± 4.51^b	507 ± 4.93^a	509.7 ± 4.93^a
T_p/°C	571.7 ± 7.23^a	513 ± 3.00^a	518 ± 3.61^a	519 ± 3.61^a
T_c/°C	518.7 ± 6.63^b	519.7 ± 4.16^b	539.3 ± 2.52^a	511.7 ± 1.53^b
ΔH/J g^{-1}	217.3 ± 2.52^b	130.3 ± 5.51^c	59.7 ± 7.02^d	554.7 ± 5.51^a
–	(exo)	(exo)	(exo)	(exo)
Gelatinisation				
T_o/°C	58.77 ± 0.29^a	55.78 ± 1.16^b	52.20 ± 0.73^c	51.36 ± 1.28^c
T_p/°C	64.57 ± 0.02^a	64.63 ± 0.09^a	62.26 ± 0.98^b	59.45 ± 0.03^c
T_c/°C	69.56 ± 4.41^a	66.98 ± 0.03^a	66.67 ± 0.02^a	64.46 ± 0.06^a
ΔH/J g^{-1}	10.88 ± 1.87^{ab}	11.45 ± 1.57^{ab}	13.25 ± 3.40^a	9.14 ± 2.85^b

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

T_o 'onset' initial temperature, T_p peak temperature, T_c 'endset' final temperature, ΔH_{gel} gelatinisation enthalpy

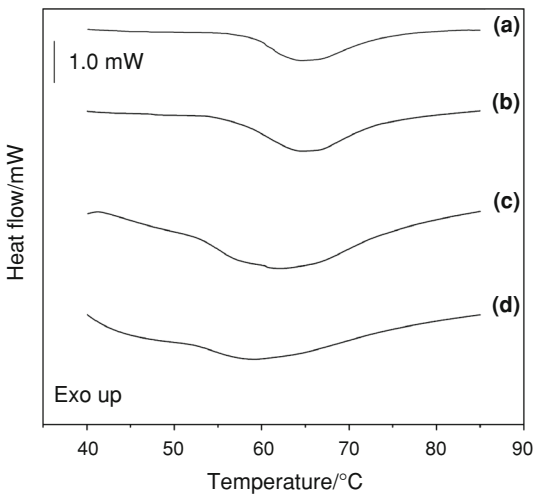

Fig. 3 DSC gelatinisation curves of (*a*) native cassava starch, 3.19 mg; (*b*) cassava starch exposed 5 min to microwave radiation, 3.12 mg; (*c*) cassava starch exposed 10 min to microwave radiation, 2.93 mg; and (*d*) cassava starch exposed 15 min to microwave radiation, 3.34 mg

curve and the linear baseline which covered the 2θ range from 5° to 50° was taken as the amorphous section. The upper diffraction peak area and the total diffraction area over the diffraction angle 5–50° were integrated. The ratio of upper area to total diffraction was used as the degree of relative crystallinity. The equation for calculating the degree of relative crystallinity was as follows:

$$X_c = A_p/A_p + A_b \quad (1)$$

where X_c refers to the relative crystallinity degree, A_p refers to the crystalised area on the X-ray diffractogram and A_b refers to the amorphous area on the X-ray diffractogram.

For determining the colour parameters of the starch, before and after treatment with microwaves, the MiniScan XE reflectance spectrophotometer 45/0-L Plus (Hunter Inc., USA) was used, which consists of three colour components: L^*, a^* and b^*. The colour of untreated and treated cassava starch samples was evaluated by the parameters L^*, brightness ranging from 0 (black) to 100 (white); a^*

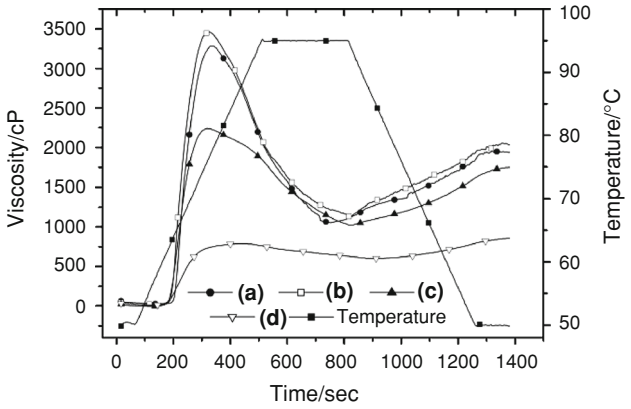

Fig. 4 RVA curves of (*a*) native cassava starch, (*b*) cassava starch exposed 5 min to microwave radiation, (*c*) cassava starch exposed 10 min to microwave radiation, (*d*) cassava starch exposed 15 min to microwave radiation

ranging from positive (red) and negative (green); and b^*, which varies from positive (yellow) to negative (blue).

All the analyses were made in triplicate. Analysis of variance (ANOVA) and Tukey test were used to compare sample means at 95 % confidence level ($p < 0.05$) using STATISTICA 7.0 software (StatSoft, Inc., Tulsa, OK, USA).

Results and discussion

One portion of the untreated sample (a) and three portions of samples subjected to microwave action for 5, 10 and 15 min (b), (c) and (d) were kept in a desiccator with calcium chloride until constant mass. After the period of microwave action, the temperature of samples (b), (c) and (d) were 135 °C, respectively. Visually, only the samples treated for 10 and 15 min with microwave irradiation

Table 3 RVA results of: (a) native cassava starch, (b) cassava starch exposed 5 min to microwave radiation, (c) cassava starch exposed 10 min to microwave radiation, and (d) cassava starch exposed 15 min to microwave radiation

Samples	Pasting temperature/°C	Viscosity peak/cP	Peak time/sec	Setback/cP	Break/cP	Final viscosity/cP
(a)	63.5 ± 0.05^{a}	3283.7 ± 3.21^{b}	335.3 ± 5.03^{b}	948 ± 6.93^{a}	$2{,}292 \pm 2.65^{b}$	$1{,}934.3 \pm 4.04^{b}$
(b)	62.2 ± 0.15^{bc}	3460.3 ± 2.08^{a}	323.3 ± 3.06^{c}	904 ± 4.00^{b}	$2{,}324 \pm 3.61^{a}$	$2{,}034.7 \pm 4.16^{a}$
(c)	61.5 ± 0.56^{c}	2234.7 ± 4.51^{c}	317 ± 2.65^{c}	724 ± 3.61^{c}	$1{,}214 \pm 3.61^{c}$	$1{,}748.3 \pm 2.08^{c}$
(d)	62.4 ± 0.35^{b}	784.7 ± 4.51^{d}	446 ± 5.29^{a}	254 ± 3.46^{d}	184.7 ± 4.16^{d}	854.33 ± 3.79^{d}

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

cP centipoises, *sec* seconds

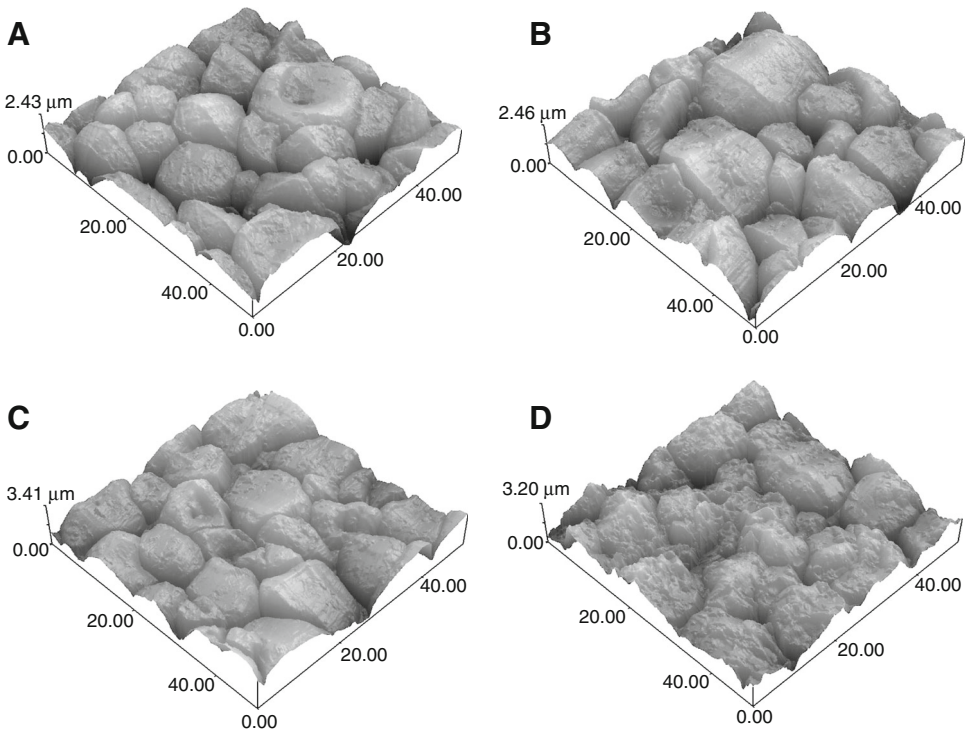

Fig. 5 NC-AFM micro-images of **a** native cassava starch, **b** cassava starch exposed 5 min to microwave radiation, **c** cassava starch exposed 10 min to microwave radiation, **d** cassava starch exposed 15 min to microwave radiation

Table 4 NC-AFM, XRD and colour parameters results of: (a) native cassava starch, (b) cassava starch exposed 5 min to microwave radiation, (c) cassava starch exposed 10 min to microwave radiation, and (d) cassava starch exposed 15 min to microwave radiation

Sample	NC-AFM		XRD					Colour parameters		
			Peaks (2θ)				Degree of relative crystallinity			
	d_a/μm	r_a/μm	1st	2nd	3rd	4th		L^*	a^*	b^*
(a)	11.22 ± 3.04^{a}	327.15 ± 1.55^{d}	15.18 ± 0.03^{a}	17.03 ± 0.03^{a}	18.87 ± 0.02^{a}	22.97 ± 0.15^{a}	28.10 ± 0.10^{a}	96.02 ± 0.41^{a}	0.43 ± 0.02^{d}	3.22 ± 0.01^{e}
(b)	12.36 ± 3.97^{a}	349.15 ± 3.79^{c}	15.02 ± 0.02^{c}	17.04 ± 0.04^{a}	17.96 ± 0.04^{b}	22.87 ± 0.03^{a}	28.17 ± 0.15^{a}	96.37 ± 0.32^{a}	0.50 ± 0.02^{d}	4.23 ± 0.07^{d}
(c)	11.49 ± 1.68^{a}	382.03 ± 2.04^{b}	15.13 ± 0.02^{ab}	17.03 ± 0.03^{a}	17.86 ± 0.04^{c}	22.89 ± 0.04^{a}	20.33 ± 0.08^{b}	93.13 ± 0.12^{b}	1.45 ± 0.01^{b}	8.63 ± 0.01^{b}
(d)	10.33 ± 2.97^{a}	404.96 ± 1.93^{a}	15.11 ± 0.01^{b}	17.03 ± 0.03^{a}	17.84 ± 0.04^{c}	22.63 ± 0.03^{b}	18.47 ± 0.06^{c}	87.56 ± 0.34^{c}	2.78 ± 0.02^{a}	14.09 ± 0.06^{a}

The degree of crystallinity was calculated as a percentage, peaks are determined in 2θ. Averages followed by the same letters in the same column do not differ statistically by Tukey test ($p < 0.05$)

d_a average diameter, r_a average roughness

shown colour alterations changing from white to light yellow.

The profile of TG curves of the studied compounds (Fig. 2A a–d, left) were similar, with three mass losses. The first mass loss was due to the dehydration process, which after microwave treatment for 5, 10 or 15 min, did not show significant changes. After this, the anhydrous compounds were stable until around 262 °C when the second and third mass losses occurred in two consecutive steps that were attributed to the decomposition of organic matter. The initial mass of each sample were: (a) 7.26 mg, (b) 7.24 mg, (c) 8.81 mg and (d) 6.47 mg. The final residue of decomposition was attributed to the formation of ash, with 0.27, 0.29, 0.35 and 0.80 % of initial mass, respectively. DTG curves were used in the determination of the temperatures and mass losses in TG curves that show the three main mass losses, and the results are presented in Table 1. The heating of a portion of untreated sample in a conventional oven at 150 °C for 2 hours was carried out and the results showed similarity with the sample treated for 5 min in a microwave.

The DSC curves (Fig. 2B a–d, right) were carried out to monitor the thermal decomposition process of the samples, and display endothermic (dehydration) or exothermic (decomposition of organic matter) events, results of which are shown in Table 2. The onset temperature (T_o), peak temperature (T_p) and conclusion temperature (T_c) as well as the enthalpy (ΔH) of main peaks were calculated, and all the experiments were performed in triplicate.

Figure 3 shows the DSC curves of untreated cassava starch (a) and after microwave treatment for 5, 10 and 15 min (b–d). Each sample was prepared in 4:1 (water:-starch w/w) mixture and maintained for 60 min to equilibrate the moisture content, with the aim of verifying the gelatinisation parameters.

The obtained results are depicted in Table 2. In relation to the untreated cassava starch (a), the gelatinisation enthalpy increased for samples (b) and (c), and decreased for sample (d).

At the same time, as the samples of untreated starch were microwaved for 5, 10 and 15 min, a new sample of untreated starch was maintained for 2 hours in a conventional oven at 150 °C. After cooling, this sample was subjected to the same parameters and the results were: $T_o = 57.63$, $T_p = 64.83$ and $T_c = 73.57$ °C as well as $\Delta H_{gel} = 10.95$ J g^{-1}; results similar to those from untreated (a) samples and those microwaved for 5 min (b).

According to the RVA results (see Fig. 4), the untreated cassava starch (a) showed a viscosity peak at 3,283.7 ± 3.21 cP; the sample treated for 5 min by microwave (b) had increased viscosity and decreased gradually from the samples at 10 (c) and 15 min (d) of exposure. The final viscosity showed the same behaviour, and the setback decreased gradually. The results are shown in Table 3.

In Figure 5, with the NC-AFM technique, slight visual differences between the untreated and treated starch granules can be observed, especially when depressions and protrusions were observed on the surface of each sample and it became possible to determine the average particle diameter (d_a), which showed no significant difference. These results are in agreement with the literature [6], which reports that microwave treatment does not alter the shape and size of starch granules.

However, this technique allowed us to calculate the average roughness (r_a) of the surface of the granules, which was higher, depending on the time of microwave exposure. The results are shown in Table 4.

X-ray diffraction powder patterns were used to verify the main peaks and degree of relative crystallinity of the samples.

No displacement of the main peaks was observed, but the relative crystallinity was calculated according to Eq. 1. The relative crystallinity decreased according to the exposure time to microwaves. According to the literature [16], the decrease in relative crystallinity occurs due to the shearing of amylopectin, which causes a decrease in the pasting viscosity. The obtained results are shown in Table 4.

Visually, a slight difference in colour was observed in the samples treated for 10 and 15 min of exposure to microwaves. The final temperature was measured, and after 5, 10 or 15 min they were all 135 °C.

Three parameters of colour were evaluated: L^*, a^* and b^*. The value of L^* gives us brightness, ranging from white ($L^* = 100$) to black ($L^* = 0$). The a^* value characterises the value of colour in the region of red ($+a^*$) to green ($-a^*$). The b^* value indicates the colour range from yellow ($+b^*$) to blue ($-b^*$).

In Table 4, it can be observed that no difference in the L^* value (trend to white) occured between the untreated sample and that exposed for 5 min to microwaves. Likewise, the b* value (trend to yellow) increased considerably from the untreated sample to those treated for 5, 10 and 15 min of microwave exposure.

For the sample that was left in a conventional oven for 2 hours at 150 °C, the verified values were: $L^* = 92.71 \pm 0.68$, $a^* = 0.80 \pm 0.05$ and $b^* = 5.45 \pm 0.07$. It can be observed that heating in a conventional oven caused similar alterations to those caused by 5 min exposure in a microwave oven.

Conclusions

The TG/DTG curves of the studied samples showed similarity with thermal decomposition in three steps, allowing the calculation of hydration, steps of decomposition and ash degree. All the samples showed a level of stability for each starch after dehydration.

The time of microwave exposure for all the samples initially (after 5 min) caused a slight increase in the viscosity peak, as observed by DSC and RVA, followed by a considerable decrease.

XRD and NC-AFM techniques showed values that were inversely proportional, i.e. the degree of relative crystallinity decreased, while the average roughness increased.

The colour characteristics, observed and analysed by reflectance spectrophotometry, showed that microwave action causes rapid and progressive colour changes, mainly with trends to yellow.

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