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Characterization of starch and flour of Gros Michel banana fruit (*Musa acuminata* AAA)

Caracterización de harina y almidón de frutos de banano Gros Michel (*Musa acuminata* AAA)

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Abstract

The flour and starch were extracted from an unconventional starchy source, Gros Michel bananas (*Musa acuminata*), harvested in farms in from Quindio department, Colombia. The study evaluated their physicochemical, thermal and rheological characteristics. The proximal flour analysis resulted in fiber content 18.82 %; while for starch 1.92 % protein, 5.3 % fat and 2.76 % fiber. The highest temperature of heat absorption was for flour was 68.88° C and the enthalpy of gelatinization was 2.17 J/g, for starch were 48.36° C and 44.62 J/g respectively. While the flour, the thermogravimetric analysis (TGA) temperatures at which the breakdown of carbohydrates (low molecular weight components) and polysaccharides (high molecular weight components) recorded were 284.51° C and 470.42° C respectively and for starch were of 307.51° C and 500.46° C. Banana starch granules are ellipsoidal in shape with an average longitudinal size of 39.39 microns and an average transverse size of 29.47 microns. The viscoamylograms showed that for flour the gelatinization onset temperature (Tg) was 76.3° C, the peak viscosity for starch 1120 cP and the Tg was 70.75° C with maximum viscosity 2087 cP.

Key words: Musaceae, *Musa acuminata*, baking characteristics, DSC, TGA, X-ray diffraction (XRD), RVA.

Resumen

En el estudio se determinaron las características fisicoquímicas, térmicas y reológicas de la harina y el almidón de frutos de banano Gros Michel (*Musa acuminata*) cosechado en fincas del departamento del Quindío, Colombia. En el análisis proximal, la harina presentó un contenido de fibra de 18.82% y el almidón presentó contenidos de proteína de 1.92%, grasa de 5.3% y fibra de 2.76%. La harina presentó la temperatura más alta de absorción de calor (68.88 °C) y su entalpía de gelatinización fue de 2.17 J/g; mientras que para el almidón estos valores fueron de 48.36 °C y 44.62 J/g, respectivamente. El análisis termogravimétrico (TGA) de la harina o temperaturas en las cuales se registra la descomposición de

carbohidratos (componentes de bajo peso molecular) y polisacáridos (componentes de alto peso molecular) fueron, respectivamente, de 284.51 °C y 470.42 °C; y para el almidón fueron de 307.51 °C y 500.46 °C. Los gránulos de almidón de banano tienen forma elipsoidal con un tamaño longitudinal promedio de 39.39 μ m y tamaño transversal promedio de 29.47 μ m. Los difractogramas de rayos X mostraron patrones de difracción tipo B. Los viscoamilogramas mostraron que para la harina la temperatura de inicio de gelatinización (Tg) es de 76.3 °C, la viscosidad máxima de 1120 cP, y para el almidón la Tg fue de 70.75 °C y la viscosidad máxima de 2087 cP.

Palabras clave: Musaceae, *Musa acuminata*, harina, almidón, características de la cocción, DSC, TGA, Rayos X, RVA.

Introduction

Flour and starch, are important food sources in Africa and Central and South America, where the Gros Michel banana (Musa acuminata AAA) is grown in extensive areas (Xu et al., 2014; Glenn et al., 2014; Avérous and Halley, 2014; Laycock and Halley, 2014). These compounds are used as additives to improve the technological properties that characterize several processed food, since they retain water and therefore, they cause changes in the rheology of the final product, depending on the physical and chemical characteristics of the starch granules (De la Torre et al., 2008). The main conventional sources to obtain flour and starch are cereals such as maize, wheat, rice and sorghum and, tuber such as potato and cassava; it is also used legume leaves and seeds. Currently, other non-conventional sources are being explored that show physicochemical, structural and functional characteristics for industrial use (Shrestha and Halley, 2014), as materials for biodegradable packing (Pelissari et al., 2013), production of resistant starches (Zhang and Hamaker, 2012), preparation of food products and as substitutes of wheat and maize starch in bakery (De la Torre et al., 2008).

The banana is a fruit grown in tropical and subtropical regions, with an annual world production of approximately 104 millions of tons for 2010 (Bello *et al.*, 2011). The main producer countries are Brazil, China, Ecuador, Philippines and India and the largest exporters are Ecuador, Colombia, Costa Rica and Philippines. The edible musaceas are classified in the genomic groups AA, AB, BB, AAA, AAB, ABB, AAAA, AAAB and, ABBB, (Aurore *et al.*, 2009; Mohapatra *et al.*, 2009, 2010a,b).

The immature banana fruits contain high concentrations of starch, cellulose and hemicellulose. These compounds, for their high nutritious and nutraceutical potential, are an important source to produce flours for food industry (Langkilde *et al.*, 2002). The objective of this research was to determine the rheologic, thermal and physicochemical properties of the flour and starch of Gros Michel banana (*Musa acuminata* AAA) as alternatives to improve the characteristics of agroindustrial products.

Materials and methods

Sampling processing. Banana fruits were harvested green in farms from Armenia, Colombia and processed in the pilot plant of food at the Universidad de Quindio. Initially, they were weighted and then the peel was removed to weight them again. The bananas were cut in slices and placed on a drying oven (Memmert UL40, Germany) at 40 °C for 48 h, before they were ground in a mill (IKA 2870900 MF 10.1, USA). Finally they were passed through a filter with a 100 μ m membrane in order to get the flour (Lucas *et al.*, 2013).

For starch extraction the traditional method was used, consisting on adding water to the banana slices on a 1:1 rate before blending at 600 rpm for 1 min and, passing the mix through a 100 μ m sieve adding enough distilled water. The mix was centrifuged at 10,000 rpm for 1 min at 25 °C. The precipitated was filtered through a mesh with 100 μ m pores and the filtrate with starch was dried out on a recirculation oven (Memmert UL40, Germany) at 40 °C for 48 h (Dufour *et al.*, 2008).

Chemical composition. The crude protein was determined by micro-Kjeldahl (AOAC, 2000), humidity by the method 925.10 (AOAC, 2000), fat by the method 30–25 (AACC, 2000) and ashes by the method 08– 01 (AACC, 2000). All the determinations were done in triplicates.

Thermogravimetric analysis (TGA). This analysis was done on a TA Instruments TGA Q500, USA equipment under a nitrogen atmosphere with a temperature range from room temperature to 800 °C, with a heating speed of 5 °C/min in samples of 6 ± 0.50 mg (Pineda *et al.*, 2011).

Differential scanning calorimetry (**DSC**). To determine temperature (T_p) and the enthalpy of gelatinization (ΔH_p) it was used a TA Instruments DSC–Q100, USA, in 10 ± 0.50 mg samples with 80% water content, prepared on hermetic aluminum capsules where the starch mass and flour were mixed directly, at a heating temperature of 5 °C/min, from room temperature to 100 °C under a nitrogen atmosphere (Pineda *et al.*, 2011).

Scanning electron microscopy (SEM). It was done with a high vacuum scanning electron microscope, JEOL JSM-6060LV, Japan. The analysis conditions were 20 kV of acceleration tension for electrons and 12 – 20 Pa of pressure at the sample chamber, to obtain images on the fracture surfaces with the secondary electrons signal. Samples were fixed on a cupper sample holder with carbon cap and covered with a golden layer to improve the material conductivity (Londoño *et al.*, 2014). The diameters and morphological characteristics of the granules were obtained with the Image Tool V 2.0 software.

X ray difraction. The samples were reduced to a fine powder and passed through a 150 μ m sieve. Later, they were densely packed on an aluminum holder. The X rays diffraction patterns were obtained using the Siemens D5000, Germany equipment, with a radiation line Cu Ka (l = 1.5418 Å) and difference in potential of 30 kV at a current density of 20 mA. Samples were registered between 5 and 40 degrees (2q) with an angular pitch of 0.050 and 15 s as counting time per pitch. The material was placed on an 30 mm x 30 mm aluminum holder at room temperature and low humidity (Rojas *et al.*, 2007).

Rapid viscoamylography. For this measurement a rapid analyzer RVA-4 (RVA Rapid Visco Analyser, Newport Scientific Narabeen, NSW,- Australia) was used. A defined temperature profile from 50 to 90 °C, at 6 °C/min, keeping the temperature for 5 min and reducing it till 50 °C at 6 °C/min was implanted. For the flour 8% (b.s.) suspension in presence of α -amylase inhibitor (AgNO₃, 0.002 mol/l) was used and for the starch water and 7%concentration was used. The temperatures pasting onset, maximum viscosity for (Vmax), ease of cooking (FC), gel breakdown and, gel setback (Dufour et al., 2009). The tabulated, graphed results were and processed with the Origin 9.0 software.

Results and discussion

Proximal analysis

Flour. In Table 1 are observed the results of this analysis for the flour of Gros Michel banana and are compared to the results of Soto (2010) with *M. paradisiaca* flour and Cardona *et al.*, with cassava flour, highlighting the higher contents of water and fiber in the banana flour, and lower protein content (0.41%). Da Mota *et al.*, evaluated the proximal composition of flours from different genetic groups of musaceas, among them, Ouro colatina (AA), Nanica (AAA), Nanicão (AAA), Prata anã (AAB), Prata comum (AAB), Mysore (AAB),

Component	Musa	Corn	Cassava	Gros Michel ^a
(%)	paradisiaca			Banana
Humidity	5.72	11.6	8.5	12.5±0.87
Protein	2.34	11	4.4	0.41±0.01
Fiber	2.3	6.4	5.2	12.82±1.15
Ashes	2.6	3.8	5.2	2.26±0.53
Fat	0.57	7.7	0.7	1.83±0.12

 Table 1. Comparative values of the proximal analysis in different flour sources.

a. results from this research.

Source: *M. paradisiaca* = Soto Azurdy, 2010. Corn and cassava flours = Cardona *et al.*, 2001.

Maçã (AAB) and Ouro da mata (AAAB) and observed that only the protein content $(2.5\% \pm 0.02 - 3.3\% \pm 0.1)$ was higher than the values obtained in this study, for the other components (fat and humidity) the values were lower, while the ash content was similar.

In this study, the fiber contained in Gros Michel banana (AAA) was high (> 12%), value that doubles the one obtained with widely consumed varieties (7%) like Nanica and Nanicão (AAA). Dufour et al. (2009) and Gibert et al. (2009) determined the proximal composition of Gros Michel banana flour. which belong to the group of dessert bananas and compared it with the flour of bananas in the same group Bocadillo, Primitivo, Cavendish, Rollizo, Tafetán Morado, and with other musacea groups, among them, cooking plantains no-Plantain (guineo, guayabo, huamoa, cachaco and pelipita); dessert hybrids (Fhia 17, Fhia 1, Fhia 18, Fhia 25); cooking hybrids (Fhia 20, Fhia 21); plantains from the Plantain group (Africa, Dominico, Dominico Hartón, Hartón, Cubano blanco, Hartón Maqueño) and observed that the content of ashes, for all the groups, varied between 2.3 and 4.3%, values that agree with the findings of this study. In the last group, the crude fiber contents varied between 1.8 and 5%, values that are lower than the ones of this study $(12.82\% \pm 1.15)$; to the contrary, for the first ones the crude protein varied between 2.1 and 4.9%, a very high value if it is compared to the one found in this study

 $(0.41\% \pm 0.01)$ in the Gros Michel variety.

Starch. In starch, the contents of fat $5.3\%\pm0.04$, ashes $(1.72\%\pm0.01)$, fiber $(2.76\%\pm0.06)$, protein $(1.92\%\pm0.02)$ and humidity (16.44% ± 0.08) were higher than the ones found by Waliszewskia *et al.* (2003), differences that are due, possibly, to the maturity degree of the fruits and to the extraction and purification techniques used for starch (Bello *et al.*, 1999, 2000; Carmona *et al.*, 2009).

The ashes content in the starch is due to the presence of minerals like calcium, potassium and magnesium, especially in dessert banana (AAA) like Bocadillo, Primitivo, Cavendish, Rollizo, Tafetán morado, Valery and Gros Michel (Dufour *et al.*, 2009). The humidity was found to be in the acceptable range for commercialization and storage of starch (Waliszewskia *et al.*, 2003; Dufour *et al.*, 2009; Gibert *et al.*, 2009).

Differential scanning calorimetry (DSC)

Flour. In the Figure 1a it is shown the thermal transition of the Gros Michel banana flour. Initially is observed a light endothermal pick on the base line of the thermogram that start at 60.16 °C (Initial temperature-To). The value of the pick temperature (Tp) was 68.88 °C in which the highest heat absorption values were registered; at the time that the process is finalized, the system goes back to a state in which there are no changes in phase and composition of the sample, as is shown in the thermogram

when the endothermal pick is retracted until reaching the base line at a final temperature (Te) of 81.51 °C. The necessary energy to complete the process is known as enthalpy of gelatinization (Δ Hp) for banana flour and its value is 2.17 J/g. At the same time, the base line of the thermogram descends; therefore the system requires a higher heat or energy flux, in order to guarantee the gelatinization process.

Research by Tribess et al. (2008) with banana flour of the Nanicao (Musa cavedenshii) variety, show that the peak temperature (Tp) for this fruit is between 67.95 °C ± 0.31 till 68.63 °C ± 0.28 and for banana (M. acuminata) reached a Tp of 68.88 °C, showing no difference for this kind of fruits; on the other hand, their enthalpy of gelatinization varied between 9.04 \pm 1.71 J/g till 11.63 \pm 1.74 J/g. These results are compared with the ones reported by Sandoval et al. (2005) in cassava (Manihot esculenta) flour who found Tp values 77.6 °C \pm 1 and enthalpy (Δ Hp) of 13.8 J/g, and for wheat flour To: 57 °C, Tp: 62.3 °C and Δ Hp: 5.3 J/g found by Zaidul *et al.* (2008). Lii and Chang (1991) and Da Mota et al. (2000) observed a light change in the range for temperature of gelatinization due to the maturation degree of the fruit, varieties and interference of other compounds present in the flour. Dufour et al. (2008, 2009) found that the temperature of gelatinization for different musacea groups varied in a significant manner between 59.7 and 67.8 °C, between dessert banana, FHIA

hybrids, Plantain sub-group and cooking plantains different from the Plantain group. In the study there were no differences in the FHIA hybrid group (dessert and cooking) with the other cooking plantains. For the Gros Michel banana the gelatinization temperature was 63.2 ± 0.3 °C, a value that is higher than the one found in this study (60.16 °C). This difference in gelatinization reflects the behavior at cooking and contributes to explain the differences between varieties and genetic groups (Da Mota et al., 2000; Zhang et al., 2005) where the varieties of the Plantains group require more energy to gelatinize and take longer to cook and soften than the banana group.

Starch. In the Figure 1b it is observed the thermogram of starch of the Gros Michel banana were is shown a pronounced endothermic peak on the base line when the phase transition starts at an initial temperature (To) 33.59 °C, followed by the peak temperature (Tp) with the highest values for heat absorption (48.36 °C). In this point the sample passes to a rubbery state due to the onset of starch granules breaking. When the process is finished the system goes back to a state where no changes in phase or composition of the sample happen. The final temperature (Te) was 64.37 $^{\circ}$ C and the enthalpy of gelatinization (Δ Hp) was 44.62 J/g. These results are contrasting to the ones found by Zaidul et al. (2008) for potato starch, with high differences in values of To (63.4 °C), Tp (67.7 °C)



Figure 1. DSC thermograms for flour (left) and starch (right) in Gros Michel banana.

and Δ Hp (20.8 J/g) in this case; and with the ones found by Hyun- Jung *et al.* (2008) (To: 66.9 °C, Tp: 74.5 °C and Δ Hp: 89.0 J/g).

Nwokocha and Williams (2009), Núñez et al. (2004) and Zhang et al. (2005) observed higher temperatures of gelatinization (To) for starch of the Plantain group (*Musa* paradisiaca normalis) with values between 68 and 71.88 °C and enthalpy between 8.59 and 15.02 J/g. The differences in temperatures of gelatinization of the starch have been attributed to the interaction among composition, molecular structure of the amylopectin and granule architecture. The gelatinization range depends on the difference in the heterogeneity degree of the crystals inside the granules (Gunaratne and Hoover, 2002).

Thermogravimetric analysis (TGA)

Flour. In the Figures 2a and 2b are displayed the thermograms and the derivate of weight loss (dotted line), the percentage and weight loss in the banana flour and the approximate temperature registered.

The first zone registered the water loss of te sample, that was 7.96% (0.32 mg in weight) and happens between 100 and 150 °C, with a highest pick at 137 °C. In the second zone is shown the largest weight loss between 200 - 389 °C and a maximum velocity of decomposition at 284.51 °C, the weight loss happened at 56.43%, moment in which the low molecular weight carbohy-

drates that correspond to starch are decomposed. In the third or final zone is shown the polysaccharide decomposition with high molecular weight, like proteins and lipids, with temperature ranges between 389 and 668 °C and a maximum velocity of decomposition at 470.42 °C and, a weight loss percentage of 32.18%. The residues presented in the thermogram were 3.43% equivalent to the ash content of the sample. These results are similar to the ones presented by Lucas et al. (2013) with guineo (Musa sapientum) flour and allow the determination of the components of the sample and predict its thermal stability and establish the starch percentage in the flours (Pineda et al., 2011). In this study the amount of components of low molecular weight and the temperatures for decomposition of the components of high molecular weight were similar to the ones observed by Montoya et al. (2012) in wheat flour.

Starch. In the Figure 3 are observed three important zones the weight loss is represented according to the thermogravimetric analysis for the Gros Michel banana starch. In the zone 1 (Figure 1a) there was a weight loss of 11.70% between 96 and 220 °C with its higher loss at 214.57 °C. In the Zone 2 the highest weight loss happened between 230 and 393.9 °C equivalent to 71.97%, where the carbohydrates and low molecular weight components, like the starch, are decomposed. In the zone 3 happens the polysaccharide decomposition at a 15.43%



Figure 2. Thermogravimetric analysis (TGA) of Gros Michel banana flour.



Figura 3. Thermogravimetric analysis (TGA) of banana starch.

and, a temperature range between 393.9 and 571.80 °C and a residue of 1.17%which corresponds to the ash content in the starch. The speed of flour loss was 5.4%and for starch was 11%/min, respectively. The lower value of flour loss is due to its high content of components with high molecular weight (32.18%) in comparison to the starch (15.43%), as it is observed in the difference in the second peak presented in the derivate curves in Figure 2b and Figure 3b.

Scanning electron microscope in starch

The banana starch granules showed variable sizes, with equatorial length between 29.3 μ m and 48.53 μ m and transversal between 16.6 μ m till 40.55 μ m. The shape and structure of the granules was mainly ellipsoidal (Picture 1 and Picture 2). In the photomicrography analysis some granules



are covered by fiber sacs and other smaller (proteins) are adhered to the starch, which agrees with the high fiber content found in the proximal analysis (12.82%). Additionally, there are some granules that are smooth with no protuberances, fractures or breaks, which demonstrates that the isolation method for starch was adequate. These results agree with the ones obtained by Waliszewskia *et al.* (2003), Espinosa *et al.* (2009), Nwokocha and Williams (2009) and Núñez *et al.* (2004) who found longitudinal diameters of 39.39 μ m and transversal diameters of 29.47 μ m, with ellipsoidal, elongated and lenticular shape.

X rays difraction (XRD)

In the difractograms of flour and starch of Gros Michel banana (Figure 4a and Figure 4b) is observed the presence of peaks that define the crystallinity of the flour which, in



Picture 1. Photomicrographs of the equatorial cut of starch granules of Gros Michel banana.



Picture 2. Photomicrographs of the transversal cut of starch granules of Gros Michel banana.

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Figure 4. Difractograms of flour (left) and starch (right) of Gros Michel banana.

majority, coincide with the standards for starch due to its high flour content. The most representative peak was found at the angle $2\theta = 17^{\circ}$ with peaks from the angle 2θ = 11°, followed by the angles $2\theta = 15.5$, 18.3, 22, 23 and 30°, which allows to conclude that the flour and starch of this banana variety show peaks that correspond to the type B and C diffraction patterns, being the last ones a combination of type A and B patterns. According to Millan *et al.* (2005), Waliszewskia *et al.* (2003) and Lucas *et al.* (2013) the musaceas starch is a mix of B and C patterns.

Rapid viscosity analysis

The pasting curves for banana flour and starch showed that the temperatures for the start of gelatinization (Tg), where the starch granules start to swollen, loss crystallinity and increase the viscosity, were 76.3 and 70.75 °C, respectively (Figure 5) which indicates that the starches require less energy and less time to start the gelatinization process. The maximum viscosity (Vmax) for starch was 1120 cP and for starch was 2087 cP, highlighting the high viscosity of the starch in comparison to the flour, due mainly, to the diverse components that it has, which allows the determination of a potential use in the preparation of soups or in products that require high viscosities.

The gel breakdown is an indicative of the stability and resistance of the gel to cutting under agroindustrial processes; when this value is high the lower is the paste stability. The values found in this study -8 cP for flour and 2 cP for starch- indicate that they are very stable for these processes.

The setback evaluates the reassociation of polymers of soluble starch and the fragments of insoluble granules during the coo-



Figure 5. Viscoamylogram of Gros Michel flour and starch.

Table 2. Viscoamylographic analysis of different Musaceas genotypes.

Banana variety	Tg	Vmax	VPF	Setback
	(°C)	(cP)	(cP)	(cP)
M. Acuminata (Gros Michel) ^a	76.3	1120	1227	107
Cavendish ^b	71.6	2230	2736	506
Rollizo ^b	68.3	1860	1977	117

a. Results from this research.b. **Source**: Dufour *et al.*, 2008, 2009.

 T_g = temperature for gelatinization onset. Vmax = maximum viscosity. VPF = viscosity at the end point, Setback = gel reorganization.

ling. In this study the value was 107 cP for flours and 669 cP for starch. The ease of cooking or time that passes before the granule swallowing was 6.8 min for flour and 5.7 min for starch.

When comparing these results with the ones of Dufour et al. (2008, 2009) (Table 2) for different musacea varieties, there are differences especially in the temperature for the gelatinization onset and the maximum viscosity reached, showing the difference in behavior for cooking and contributes to explain the differences between varieties and genetic groups and, indicates that the gegroups with higher values netic for temperature of gelatinization (Tg) need more energy to gelatinize the starch and take longer to soften during cooking processes (Da Mota et al. 2000; Zhang et al., 2005; Lucas et al., 2013).

Conclusions

- The flour and starch obtained from Gros Michel banana show high contents of fiber, fat and protein, therefore, they have a good potential to elaborate or improve agroindustrial products of massive use.
- The scanning electron microscopy (SEM) and the X ray diffraction established that according to the size of the granules and the starch type, it is possible to use this banana variety in process that require the increase in viscosity, like sauces and compotes.
- The thermal characteristics of flour and starch obtained from Gros Michel banana reduce the energetic costs in different agroindustrial processes, since they

gelatinize at a relative low temperature and the maximum peak is rapidly reached, therefore, they are easy to cook and need less energy than other type of starch.

• The shape and size of the starch granules of Gros Michel banana favor the gel formation, with higher water absorption capacity, higher hydration velocity and fast disintegration.

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