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# The effect of the structure of native banana starch from two varieties on its acid hydrolysis

Alejandro Aparicio-Saguilán [a,1], Andrés Aguirre-Cruz [a,1], Guadalupe Méndez-Montealvo [b,*,2], Sandra L. Rodriguez-Ambriz [c,3], Francisco J. Garcia-Suarez [c,3], Delia E. Páramo-Calderón [a,1], Luis A. Bello-Pérez [c,3]

[a] Instituto de Biotecnología, Universidad del Papaloapan, Circuito Central 200, Col. Parque Industrial, C.P. 68301 Tuxtepec, Oaxaca, Mexico
[b] Instituto Politécnico Nacional, CICATA Querétaro, Cerro Blanco No. 141. Col. Colinas del Cimatario, C.P. 76090 Santiago de Querétaro, Querétaro, Mexico
[c] Instituto Politécnico Nacional, CEPROBI, Km. 6.5 Carr. Yautepec-Jojutla Col. San Isidro, C.P. 62731 Yautepec, Morelos, Mexico

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ABSTRACT

Two banana starches were studied to analyze the effect of the acid hydrolysis on their molecular structure, and the impact in their physicochemical features. The native banana starches exhibit differences in the amylose content, molar mass, gelatinization parameters, X-ray diffraction pattern, and pasting profile. These results suggested that different acid hydrolysis mechanisms may be operative in these two starches. The kinetic hydrolysis is different in both banana starches that are related to the crystalline packing of the starch molecules. This was confirmed by the amylose content, the X-ray diffraction pattern, and the thermal study in the acid hydrolyzed starches at different times. The acid-treated Roatan starch showed higher retrogradation than Macho starch, a phenomenon that increases in the sample hydrolyzed for the longer time. This pattern is related to the amylose/amylopectin ratio, the reduction in the molar mass and the gyration radius. The acid hydrolysis of banana starches, although they have some similarities, they are different.



## 1. Introduction

In recent years, there has been increasing interest in starch from novel or non-conventional botanical sources because these starches may have different physicochemical and functional properties than those of conventional starches (e.g., corn, rice, wheat, and potato). Several studies have revealed the potential of banana starch due to its physicochemical and functional features (Zhang, Whistler, BeMiller, & Hamaker, 2005), its digestibility (Zhang & Hamaker, 2012), its chemical modification (Bello-Pérez, Agama-Acevedo, Zamudio-Flores, Mendez-Montealvo, & Rodriguez-Ambriz, 2010), and food applications (Hernández-Nava, Berrios, Pan, Osorio-Díaz, & Bello-Pérez, 2009). Banana starch has recently been isolated from diverse cultivars, showing difference in the morphological, structural, physicochemical, functional, and digestibility characteristics (Agama-Acevedo et al., 2014). It is well known that native raw banana starch is highly resistant to hydrolysis by the amylases present in the digestive system of human, but that cooking increases its digestibility (Lehmann, Jacobash, & Schmiedl, 2002). Several studies have been conducted to change banana starch digestibility, by chemical modification such as mild-acid treatment (Aparicio-Saguilán, Flores-Huicochea, Tovar, & García-Suárez, 2005), and molecular cross-linking (Aparicio-Saguilán, Gutierrez-Meraz, García-Suarez, Tovar, & Bello-Pérez, 2008). Acid hydrolysis of banana starch from two cultivars was carried out, with the objective to increase the slowly digestible starch content (Espinosa-Solís, Sánchez-Ambriz, Hamaker, & Bello-Pérez, 2011). Data from this previous study have shown similar morphological features, gelatinization temperatures and enthalpy, as well as resistant starch content, but different crystallinity, for both native banana starches (banana and plantain). However, the acid hydrolysis of banana starch with different structural, molecular, and physicochemical features may be important to understand of this kind of chemical modification and how the acid treatment modifies

* Corresponding author. Tel.: +52 442 2290804x81004; fax: +52 442 2290538.
*E-mail addresses:* alejandrosag@hotmail.com (A. Aparicio-Saguilán), antarcruz@hotmail.com (A. Aguirre-Cruz), cmendez@ipn.mx, mgmendezm@yahoo.com (G. Méndez-Montealvo), srodrigueza@ipn.mx (S.L. Rodriguez-Ambriz), fgarcias@ipn.mx (F.J. Garcia-Suarez), de_paramo@yahoo.com.mx (D.E. Páramo-Calderón), labellop@ipn.mx (L.A. Bello-Pérez).
[1] Tel.: +52 287 8759240; fax: +52 287 8759240x111.
[2] Tel.: +52 442 2290804x81004; fax: +52 442 2290538.
[3] Tel.: +52 735 3942020; fax: +52 735 3941896.

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the starch structure and changes the physicochemical characteristics of banana starch. Hence, the aim of this study is to evaluate the effect of the acid treatment on the native banana starch structure and its physicochemical features.

## 2. Materials and methods

### 2.1. Materials

Starch was isolated from Roatan and Macho crops. Unripe bananas were a gift from the farm "Mundo Nuevo" in Tuxtepec, Oaxaca, Mexico.

### 2.2. Methods

#### 2.2.1. Starch isolation

To obtain starch from unripe bananas, the methodology reported by Flores-Gorosquera, García-Suárez, Flores-Huicochea, Nuñez Santiago, and Bello-Perez (2004) was followed. After the banana peel was removed the fruit was ground in an industrial type blender (Waring Laboratory, model CB 15, Waring Factory Service Center, Torrington, CT, USA). In order to prevent the oxidation of the fruit, 3.6 kg of peeled banana were milled with 6 L of 0.3 g/100 mL citric acid solution. The resulting mixture was filtered using an electrical sieve (Retsch, model AS 200, Retsch Gmgh, Haan, Germany). Different mesh sizes (Lab. Test Sieve) were used: No. 40 (0.420 mm), 100 (0.15 mm), and 270 (0.053 mm). At each step of sieving, the product was washed with a sufficient volume of water until the aqueous solution showed no apparent starch residues. At the end of the sieving operation, the starch was allowed to precipitate overnight. Thereafter, the supernatant was decanted and the starch was washed with distilled water. This process was repeated three times. Finally, the product was dried in a tray dryer (manufactured by SUSESA) at 40 °C overnight. The powder obtained was sieved using a mesh of 0.15 mm, weighed and stored in a container until use. Starch purity was measured as total starch, using K-TSTA 04/2009 kit Megazyme, according to "C determination" for samples containing RS, but not D-glucose and/or maltodextrins.

#### 2.2.2. Acid treatment of starch

Starch lintnerization was conducted by the method proposed by Shin, Woo, and Seib (2003). To banana starch (500 g) were added to 0.4 L of HCl (1.6 mol/L), and maintain during 9 days at 35 °C, using constant agitation of 50 rpm; aliquots were taken at different hydrolysis reaction times (1, 3, 5, 7, and 9 days). After each hydrolysis time, the reaction was neutralized with NaOH (1.6 mol/L) to adjust to pH 7. Starch solids were washed six times with distilled water. The product was dried at 50 °C for 24 h. Subsequently, it was ground and sieved with a mesh of 0.15 mm. Finally, the samples were stored at room temperature for analysis.

$$\text{Degree of hydrolysis (\%)} = 100 \times \frac{\text{Soluble sugar produced by acid hydrolysis}}{\text{Total starch dry weight}}$$

#### 2.2.3. Amylose percentage

The amylose percentage was measured following the method of Juliano et al. (1981). A sample of 100 mg ± 0.5 mg was placed in a 100 mL volumetric flask (twice for each sample). Then 1 mL of ethanol was added to each sample, carefully washing down any sample adhering to the side of the flask. The sample were shaken slightly in order to wet the entire sample, followed by the addition of 9.0 mL of sodium hydroxide solution (1 mol/L) and then mixed. A blank (i.e., no sample) was prepared and to ensure that starch was dissolved completely by standing overnight. Make up to 100 mL volume with water. A 0.5 mL aliquot of each sample was pipetted into a test tube and 5 mL of water, 0.10 mL of acetic acid, 0.20 mL of iodine solution, and 4.2 mL water were added for a final volume of 10 mL. Then, the solution was well mixed in a vortex mixer (VX-200, Labnet International, Inc. NJ, USA). The absorbance was measured immediately at 720 nm against the blank solution.

The amylose percentage was calculated with an equation obtained from the calibration curve, as describe here. For each of the five calibrated amylose standard with known amylose content, place 100 mg ± 0.5 mg into a 100 mL volumetric flask (not necessary to repeat these in triplicate), carefully add 1 mL of ethanol (washing down any sample that adheres to the side of the flask). Shake slightly in order to wet the entire sample. Add 9.0 mL of sodium hydroxide solution (1 mol/L) and mix. Prepare a blank (no sample) to ensure that the starch is dissolved completely by standing overnight. Pipette a 0.5 mL aliquot of each sample into two test tubes. Add 5 mL of water and 0.10 mL of acetic acid and 0.20 mL of iodine solution and 4.2 mL of water (to reach a final volume 10 mL). Mix well in a vortex mixer (VX-200, Labnet International, Inc. NJ, USA). Measure the absorbance immediately at 720 nm against the blank solution. Plot the calibration curve, and the lineal equation is obtained from the graph.

To prepare the iodine solution (0.2 g/100 g iodine in 2 g/100 mL KI): Weigh, to the nearest 5 mg, 2.0 g of potassium iodide in a weighing bottle fitted with a stopper. Add sufficient water to form a saturated solution. Add 0.2 g of iodine, weighed to the nearest 1 mg. When all the iodine has dissolved, transfer the solution quantitatively to a 100 mL volumetric flask, dilute with water to a final volume of 100 mL and mix. Store in an amber bottle to protect the solution from light.

#### 2.2.4. X-ray diffraction patterns

Samples were stored in a sealed container at a relative humidity of 85% for achieving constant moisture content. X-ray diffraction patterns were obtained with a Bruker AXS D8 Advance diffractometer (Bruker AXS, Inc, Madison, WI, USA) using Ni-filtered-CuK-a radiation, operated at 30 mA and 40 kV. The diffractograms were collected in $2\theta$ range of 3 to 40°, using a scan rate of 1 °/min.

#### 2.2.5. Rheological properties: rotational test

The pasting profile of the starch dispersion (5 g/100 mL, db) was measured by running a rotational test in a AR1000 Rheometer (TA Instruments, New Castle, DE, USA) using parallel plates that had been sandblasted plate with a diameter of 40 mm and a gap of 1000 μm at a heating or cooling rate of 2.5 °C/min and a shear rate of 50 $s^{-1}$. The parallel plates were covered with mineral oil to avoid water evaporation during the test. The rheometer was programmed for running time sweeps of the heating cycles from 25 °C to 90 °C, then holding at 90 °C for 10 min, cooling from 90 °C to 25 °C, and maintaining 25 °C for 5 min. These results were obtained in triplicate.

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