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Comparative Studies on Some Physico-chemical, Thermal, Morphological, and Pasting Properties of Acid-thinned Jicama and Maize Starches

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Abstract Comparative studies on acid hydrolysis of jicama and maize starch were carried out using concentrations of hydrochloric acid of 1.5%, 3.0%, and 4.5% (w/v), for 3 and 6 h, at 40°C. Native maize and jicama starches showed important morphological, thermal, and structural differences from those of tubers and cereals which potentially offer diverse industrial applications. Jicama starch showed low amylose content (12%) and small size of starch granules. Due to these characteristics, jicama starch was more susceptible to degradation during hydrolysis process than maize starch. Under the experimental conditions

employed, the acid degradation was not particularly severe, as shown by scanning electronic microscopy analysis which showed small degraded zones and similar X-ray patterns in both starches. However, jicama starch was more susceptible to acid hydrolysis than maize starch, as revealed by the considerable increase in water solubility index, damaged starch, and crystallinity values. Also, the higher susceptibility of jicama starch than maize starch to the hydrolysis conditions was reflected in the sugar content release during hydrolysis. The relative crystallinity of hydrolyzed maize starches decreased during hydrolysis, while those of hydrolyzed jicama starches increased attributable to the lower amylose content of jicama starch in relation to maize starch. Maize and jicama hydrolyzed starches showed low viscosity values with relation to their native starch counterparts. However, native jicama starch showed lower viscosity values than maize starch, suggesting a lower internal stability of the starch granules during hydrolysis. Both native and hydrolyzed maize starches showed higher enthalpy, T_o , T_p , and T_c values than jicama starch and the broadening of the endotherms decreased during the hydrolysis of both starches.

Keywords Jicama · Maize · Starch · Hydrolyzed · Properties

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Introduction

The primary objective of acid hydrolysis is to reduce the hot viscosity of the starch paste so that higher concentrations of starch can be dispersed without excessive thickening. These products are commonly referred to as “thin-boiling” or “acid-thinned” starches (Thomas and Atwell 1999a, b). Acid-thinned starches can be prepared

by chemical, physical, and enzymatic methods (Agboola et al. 1991). Hydrolyzed starches are produced when a concentrated suspension of starch (30–40 g/100 g solids) is treated with acid, at temperatures lower than those of gelatinization (30–60°C), during one or many hours of reaction time. Mineral acids commonly used are hydrochloric acid or sulfuric acid (Fleche 1985; Pomeranz 1991). Starches or starch products can act as stabilizers and can also help in forming a thin, clear, non-tacky film on the product. They also help to soften the surface texture so that a hard shell does not form. These types of starches are particularly useful in confections where jelly with shorter texture and flexible properties is required. Candies, gum jellies such as jellybeans, gummy bears, and orange slices contain these starches (Zallier 1988). Highly crystalline starch is also used in diverse industries, including food, paper, and textile manufacture (Jenkins and Donald 1997). Although the selection of a starch that is compatible with a particular process is one of the most critical aspects of achieving proper starch performance (e.g., viscosity, gel formation, and water binding) and the desired product quality, many types of starch could be used depending on the application. Generally, maize starch is preferred because of its abundance and cost. However, new sources of native starch, appropriately modified, can demonstrate the specific properties necessary for use in the food and other industries. The particle size of the starch granules, dispersibility of starch granule components, molecular weight of the starch polymers, and water-binding ability are controlled to yield the fat-replacing qualities required. Starch-based, fat-replacing ingredients can be either starches or converted starch products such as hydrolyzed starch. Jicama (*Pachyrhizus erosus* L. Urban) belongs to the Leguminosae family, Papilionoidea subfamily. This leguminous plant is native to the Amazon region and to the semiarid region of Mexico. It grows well in tropical and sub-tropical regions, in both acidic and basic soils, and has a high nitrogen fixation potential (Sorensen 1996; Stamford et al. 2002). However, its use as food is limited and large volumes are lost during pre-harvest (36%), which is attributed to the lack of a culture of rotation, and excess humidity during its physiological maturation. Further, inadequate handling during storage results in losses that can reach 13% of material with no commercial value (Sorensen 1996). Some of the alternatives to add value to this raw material and to expand its applications have been previously reported in the preparation of pre-gelatinized flours (Martínez-Bustos et al. 2005), starches with reduced particle size (Martínez-Bustos et al. 2007), and as fat substitute in yogurt (Amaya-Llano et al. 2008). Also, these authors reported that jicama and maize acid-thinned

starches had the capacity to form gel and thermo-reversibility with some differences in gel strength. Maize gels were firmer, although mild hydrolysis of jicama starches did result in slightly firm gels. These starches developed a sticky consistency with high hydrolysis conditions; they were probably more susceptible to the acid hydrolysis due to its low amylose content. However, the physico-chemical characteristics of native and thinned jicama starch have not been studied. Thus, the aim of the present investigation was to compare some of the physico-chemical, thermal, morphological, and pasting properties of jicama and maize starches after acid thinning.

Materials and Methods

Materials

Maize starch was purchased from National Starch, Co. (Hammond, IN, USA). Jicama was purchased from a local market in Queretaro, Qro., Mexico, and starch was isolated following the method reported by Galván-Mendoza et al. (2001). Jicama was wet milled at low temperature, using a stone mill. The milled jicama was filtered through sieve no. 100 marks Mont-inox and the filtrate was centrifuged and dried (40°C, 8 h).

Chemical Analysis

Official methods (AOAC 1990) were used to analyze the content of moisture (no. 925.09), protein (no. 979.09), lipid (no. 923.05), and ash (no. 923.03).

Total Carbohydrate

Samples were extracted with alcohol–benzene at room temperature and the residue was treated with 72% sulfuric acid (v/v) for 2 h at room temperature (15–20°C) with occasional stirring. The mixture was diluted with distilled water to make a 2.5% sulfuric acid solution and autoclaved at 120°C for 60 min. After cooling, the residue was filtered and washed with water through a porous crucible (G4), dried at 105°C and weighed as insoluble acid residue. The filtrate (autoclaved sulfuric acid solution) was used for total carbohydrate analysis. The amount of carbohydrates in the filtrate was estimated by the phenol–sulfuric acid method (Dubois et al. 1956). One milliliter of 5% phenol (v/v) and 5 ml of 98% sulfuric acid (v/v) were added to the filtrate. The optical density of the solution then was measured by a spectrophotometer at 490 nm, using known concentrations of D-glucose as standards.

Preparation of Acid-thinned Starch

Acid-thinned starches were prepared following the methodology of Zambrano and Camargo (2002). Starch slurry was prepared by dispersing starch (40 g dry basis) in aqueous HCl (5 g of pure HCl/100 ml) in a water bath at 40°C with constant stirring. The hydrolysis conditions are shown in Table 1. After each assay of hydrolysis, the pH was adjusted to 5.5 ± 0.2 by slowly adding aqueous sodium hydroxide (5 g/100 ml). The starch was washed three times with the twofold volume of deionized water prior to filtration and dried in a convection oven at 45°C for 48 h. The dried powder was sieved through a 0.150-mm sieve to obtain acid-modified powder.

Physico-chemical Properties of Starch

Damaged Starch

When the maize or jicama are milled, a portion of the starch granules are damaged. The damaged starch absorbs more water than intact starch granules. Damaged starch shows many physical characteristics, which are similar to those shown by gelatinized granules. Farrand (1969) developed a procedure for the determination of damaged starch and alpha-amylase as they relate to flour water absorption and bread quality. The iodometric method is based on the increased extractability of amylose from damaged starch granules. The extracted amylose reacts with iodine and is measured spectrophotometrically, amperometrically, or colorimetrically. The iodine reaction was evaluated using spectrophotometric procedures (Williams and Fegol 1969).

Table 1 Hydrolysis conditions

Assay	Starch type	AC (%)	<i>T</i> (h)
NM	Native maize	—	—
NJ	Native jicama	—	—
T1	Maize	1.5	3
T2	Maize	1.5	6
T3	Maize	3.0	3
T4	Maize	3.0	6
T5	Maize	4.5	3
T6	Maize	4.5	6
T7	Jicama	1.5	3
T8	Jicama	1.5	6
T9	Jicama	3.0	3
T10	Jicama	3.0	6
T11	Jicama	4.5	3
T12	Jicama	4.5	6

T1...T12 hydrolysis assays, *AC* acid concentration, *T* hydrolysis time

The damaged starch value was expressed in Farrand equivalent units (FEU), using the regression equation $X = 0.286 + 5.30Y$, where X = FEU and Y = absorbance value. The Farrand units were related to the percentage of damaged starch (% DS) using the expression $FEU = (5.2 \times DS) - 10.3$, where DS is the damaged starch. So, $DS = \frac{(FEU + 10.3)}{5.2}$

Apparent Amylose (ApA)

The colorimetric method was used to determine the apparent amylose content of starch and the total amylose content of lipid-free starch. Samples were dissolved in a mixture of 9 vol of DMSO and 1 vol of 1 M urea. An aliquot of starch-urea-dimethylsulfoxide was dissolved with an appropriate amount of water, then an I₂-KI solution was added and the resultant solution was mixed immediately. Solution absorbances were measured at 635 nm 15 min after addition of I₂-KI reagent. The absorption curves were also determined in the range 400–800 nm. Calculation and temperature corrections of blue values were done according to Morrison and Laignelet (1983). Amylose content of the native and acid-thinned starch was determined in triplicate

X-Ray Diffraction Patterns and Crystallinity

Ground samples with a 9.5–10.5% moisture content that had passed through a 2.50-mm mesh sieve were packed onto a glass sample plate (0.5 mm deep) and mounted on a diffractometer (Carl Zeiss). The diffractometer was operated at 16 mA and 30 kV. The scanning region of the diffraction angle (2θ) ranged from 5° to 50°, with a 0.02° step size, intervals of 0.04°, and a counting time of 4.5°/min. The percentage of relative crystallinity of the starch was measured by separating the crystalline and amorphous area in the X-ray diffractograms. The percentage of relative crystallinity was calculated as: (crystalline area / total area) \times 100. Two determinations were made for each sample.

Water Absorption Index (WAI) and Water Solubility Index (WSI)

The WAI and WSI were measured using a technique developed for cereals (Anderson 1982). Maize or jicama starch (2.5 g) was suspended in distilled water at 30°C at a concentration of 2.5 g in 30 ml. The suspension was centrifuged at 3,000 \times g for 10 min in a 50 ml tared centrifuge tube. The supernatant liquid was then decanted into a tared evaporating dish of known weight. The weight

of dried solid following evaporation in an air-circulating oven at 103°C was recorded. WAI was reported as grams of water/grams of dried sample. The WSI, expressed as grams solids/grams original solids, was calculated from the weight of dry solids recovered by evaporating the supernatant at 110°C for 12 h. Determinations were made in triplicate.

Viscosity Profiles

The viscosity of the samples was determined using a 3D Rapid Visco Analyzer (Newport Scientific Pty Ltd, Sydney, Australia). The analysis was performed following the method 61-02 (AACC 1999). The following parameters were measured: initial gelatinization temperature, maximum viscosity at 92°C, initial cooling viscosity, final viscosity, and retrogradation viscosity (RV). Retrogradation “setback” (Stbk; cp): It is the arithmetical difference between the registered viscosity in the final point at 50° and the minimum viscosity “holding” (Thomas and Atwell 1999a, b).

Scanning Electron Microscopy (SEM)

Particle size and changes in the physical structure of starch during hydrolysis were examined using the scanning electron microscope. Starch samples were dehydrated by lyophilization and stuck to stubs, coated with a gold layer in a high vacuum using a sputtering equipment (System Mod. LCV-76 SEM) for 3 min. Samples were examined using the scanning electron microscope ESEM-EDAX, using a secondary electron detector with 15 kV of acceleration.

Thermal Properties

Differential scanning calorimetry was used (DSC 822e, Birefrigerated, Mettler Toledo Lab Plant, Huddersfield, UK). Samples (3 mg, dry basis, db) were accurately weighed in an aluminum pan, mixed with 7 µl of deionized water, and sealed. The samples were heated from 20 to 120°C at a rate of 10°C/min in a high pressure pan of 40 µl. Two determinations were made for each sample.

Experimental Design and Data Analysis

A 3 × 2 factorial experimental design was used. All treatments were performed randomly using a block (starch-type) design, evaluating the hydrochloric acid concentration (AC), hydrolysis time (*T*), and their effect on the response variables. The data analysis was performed using the Experimental Design and descriptive statistics modules of Statistica software package, release 5.1 (Stat-

Soft, Inc. 1997). An incomplete quadratic mathematic model for fitting the data was used:

$$y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{1,2} X_1 * X_2$$

Results and Discussion

Chemical Composition

The composition (% db) of maize and jicama starches was moisture content 11.3 and 5.9; total protein 0.3 ± 0.004 and 0.09 ± 0.004 ; lipid 0.56 ± 0.04 and 0.7 ± 0.04 ; ash 0.24 ± 0.04 and 0.27 ± 0.04 , respectively. These values are within the range reported for different varieties of starches and cereals (Smith 1967; Hoover 2001).

Characteristics of Acid-thinned Starches

Apparent Amylose (ApA)

Table 2 shows ANOVA results for ApA. It was found that regression model (RSM) was significant for both starches (maize and jicama) with values of $R^2 > 0.73$ and p of $F > 0.001$; nevertheless, jicama starch displayed lack of fit ($p = 0.005$). In Table 3 are shown the mathematical models of the responses and the effects of terms of the model. Acid concentration (AC) was the variable that had the greatest effects in quadratic and linear terms ($p < 0.001$) for both starches. Figure 1 shows that an increase in AC (1–3%) increased the values of ApA. Nevertheless, a greater increase (3–5%) decreased the values of ApA. In addition, it was found that jicama starch showed more significant changes. Native maize and jicama starches had ApA values of 18% and 12%, respectively. The ApA values of hydrolyzed starches were higher than native starches. Sandhu et al. (2007) found lower values of amylose content of acid-hydrolyzed normal maize starches than those found in this work (12.6 and 13.7 g/100 g). These differences can be attributed to the differences in starch source and hydrolysis conditions. The ApA of maize and jicama starches hydrolyzed with hydrochloric acid showed the lowest values at low hydrolysis time and acid concentrations, although the values of ApA of hydrolyzed maize starch were higher than those of hydrolyzed jicama starch. This increase of ApA was attributed to the rapid degree of depolymerization of amylopectin and the liberation of more linear fragments. Also, the increase of ApA can be attributed to the formation of intramolecular and intermolecular linkages between residues of amylose, which increases the length of these chains and their capacity to form complexes with iodine, increasing the ApA values. Crystalline regions of starch formed of amylopectin were

Table 2 ANOVA of the evaluated responses

Response	Starch	R^2	F value	P of F (model)	Lack of fit (p)
ApA (%)	Maize	0.73	12.81	<0.001	0.12
	Jicama	0.79	17.57	<0.001	0.005
DS (%)	Maize	0.46	4.07	0.02	0.05
	Jicama	0.68	10.25	<0.001	0.19
WAI (%)	Maize	0.61	7.56	<0.001	0.98
	Jicama	0.88	34.49	<0.001	0.57
WSI (%)	Maize	0.22	1.34	0.29	0.95
	Jicama	0.60	6.13	0.002	0.008

ApA apparent amylose, DS damage starch, WAI Water Adsorption Index, WSI Water Solubility Index

preferentially attacked by hydrochloric acid and transformed into short chains. Starches that contain chains of amylopectin showed a large overestimation of amylose values (Biliaderis et al. 1981; Biliaderis 1991; Betancourt and Chel 1997). Effect of hydrolysis of different acids on starch amylose was also reported, with native maize starch amylose content 24% increasing to 55%, 45%, and 29% after HCl, HNO₃, and H₂SO₄ hydrolysis, respectively and decreasing to 22% after H₃PO₄ hydrolysis (Singh and Ali 2000). Some hydrolyzed samples showed similar or higher ApA content with relation to their counterparts of native starch. Studies conducted by Kerr (1952) and BeMiller (1967) reported that waxy maize starch was hydrolyzed more quickly than normal maize starch. According to these researchers, the acid attacked the highly branched molecules (amylopectin) more easily than the linear molecules (amylose) inside the granule, which causes an increase in the content of ApA. Contrary to our results, Sandhu et al. (2007) found a significant reduction in amylose content after the acid treatment of normal maize starch attributable to differences in starch source and hydrolysis time.

Damaged Starch (DS)

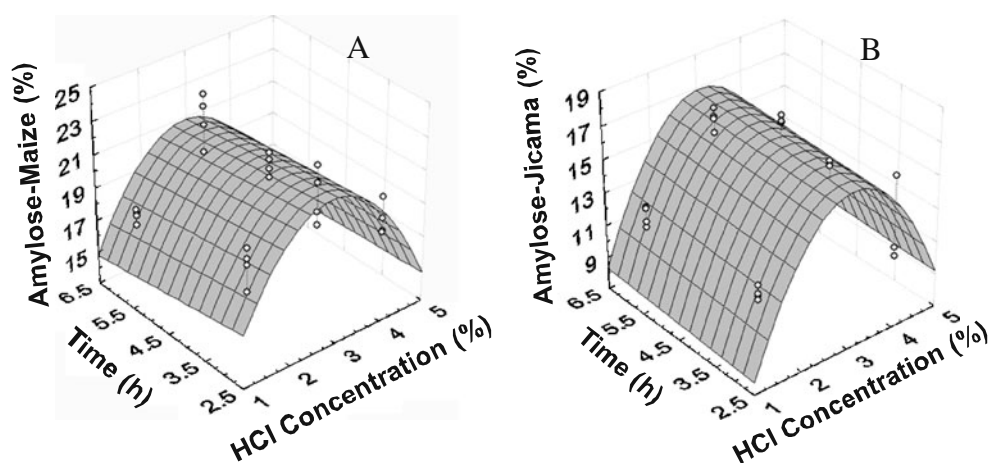
ANOVA results for DS (Table 2) showed that regression model (RSM) was significant for both starches (maize and jicama) with values of p of $F > 0.05$ and did not show lack of fit ($p > 0.05$); nevertheless, the R^2 values were moderated for jicama starch ($R^2 = 0.68$) and regular for maize starch ($R^2 > 0.46$). The effects of variables (Table 3) did not show significant effects of the variables ($p > 0.05$) on both starches (Fig. 2). The DS of native maize starch was 2.56% and did not show significant changes in DS during hydrolysis process. Nevertheless, these values increased with the increasing of AC (Fig. 2a). On the contrary, hydrolyzed jicama starch was more susceptible to acid and

Table 3 Regression coefficients of the evaluated responses

	Coefficients							
	ApA maize	ApA jicama	DS maize	DS jicama	WAI maize	WAI jicama	WSI maize	WSI jicama
Intercept (b_0)	9.838 (0.002) ^a	1.184 (0.69)	3.64 (<0.001)	12.900 (0.002)	0.446 (0.02)	2.111 (<0.001)	1.782 (<0.001)	2.681 (<0.001)
AC (b_1)	8.893 (<0.001)	10.875 (<0.001)	-0.364 (0.47)	0.051 (0.98)	-0.098 (0.35)	0.198 (0.54)	0.125 (<0.001)	-0.153 (<0.001)
T (b_2)	-0.444 (0.32)	0.752 (0.12)	-0.045 (0.75)	-0.472 (0.42)	0.092 (0.005)	-0.129 (0.17)	0.006 (0.91)	-0.028 (0.02)
AC ² (b_{11})	-1.582 (<0.001)	1.975 (<0.001)	-0.006 (0.93)	0.225 (0.47)	0.022 (0.18)	0.009 (0.85)	-0.016 (0.58)	0.018 (0.005)
AC* T ($b_{1,2}$)	0.067 (0.62)	0.295 (0.05)	0.077 (0.09)	0.054 (0.76)	-0.014 (0.14)	0.052 (0.08)	-0.013 (0.44)	0.006 (0.08)

AC acid concentration (%), T time (h), ApA apparent amylose, DS damaged starch, WAI Water Adsorption Index, WSI Water Solubility Index, $b_{01,2}$ coefficients of regression. * p value

Fig. 1 Effects of acid concentration and hydrolysis time on apparent amylose content of jicama (b) and maize (a) starches



increased the percentage of damaged starch from 4.04% for native starch to 10–18% for hydrolyzed jicama starch (Fig. 2b); the lower size of granule related to maize starch and structural differences in both starches influenced this behavior. Also, it is possible that the process of extraction of jicama starch increased the level of damaged starch, which affected its functional properties.

Water Absorption Index (WAI) and Water Solubility Index (WSI)

ANOVA results for WAI and WSI are shown in Table 2. It was found that regression model for WAI (RSM) was significant for both starches with values of $R^2 = 0.88$ and 0.61 for jicama and maize starches, respectively, values of p of $F < 0.001$ and did not show lack of fit ($p > 0.05$). The regression model for WSI only was significant for jicama starch with values of $R^2 = 0.60$, p of $F = 0.002$, nevertheless, showed lack of fit ($p = 0.005$). The effects of the variables on WAI of jicama and maize starches are shown in Table 3. Only the lineal term of time (T) had

significant effect ($p > 0.05$) on maize starch. The WAI values of hydrolyzed maize starches were lower than these hydrolyzed jicama starches (Fig. 3a, b), indicating lesser damage during hydrolysis than maize starch as indicated in the DS results. Damaged starch tends to absorb major amount of water due to the water filtration through fissures. Thomas and Atwell (1999a, b) reported that tuber starches have a greater granule size than those from cereals, thus starch granules from cereals can be more sensitive to gelatinization. Normal maize starch had a value of 0.11, which increased during hydrolysis from 0.49 and to 0.91. Nevertheless, these changes were not significant. Attributable to the fragmented granules that are not capable of reaching an organized structure. Therefore, they cannot retain water inside the structure as a result of the hydrolysis (Betancourt and Chel 1997). John et al. (2002) and Sandhu et al. (2007) also reported a decrease in swelling power and increase in solubility on acid hydrolysis of arrowroot starch and maize starch respectively. The WAI values of hydrolyzed jicama starches increased when the acid concentration and hydrolysis time were increased (Fig. 3b). The hydro-

Fig. 2 Effects of acid concentration and hydrolysis time on damaged starch (DS) of jicama (b) and maize (a) starches

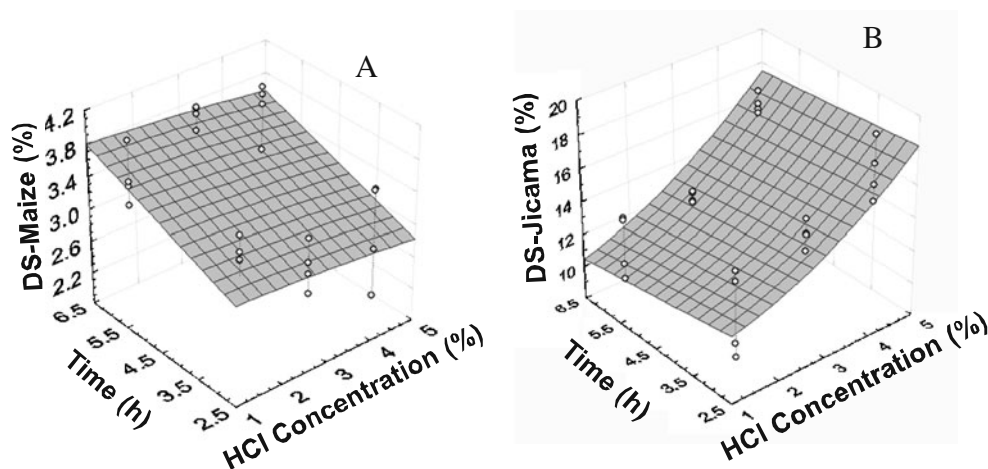
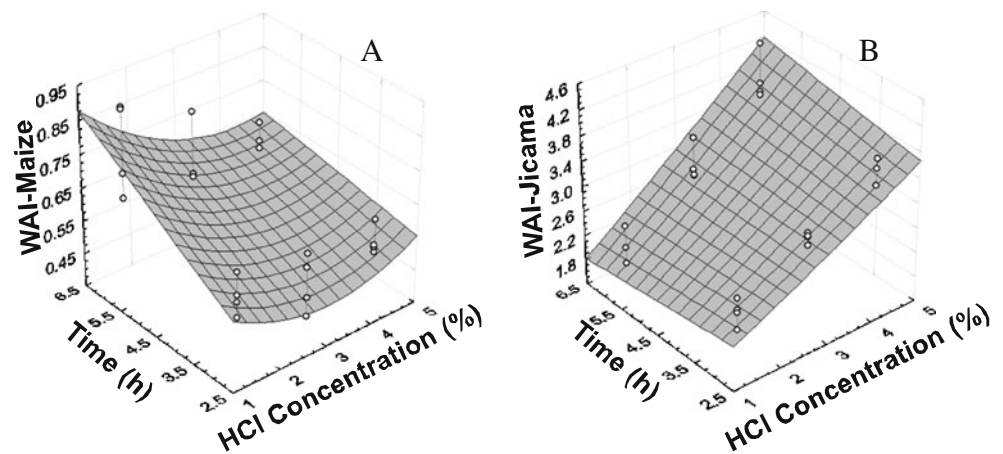


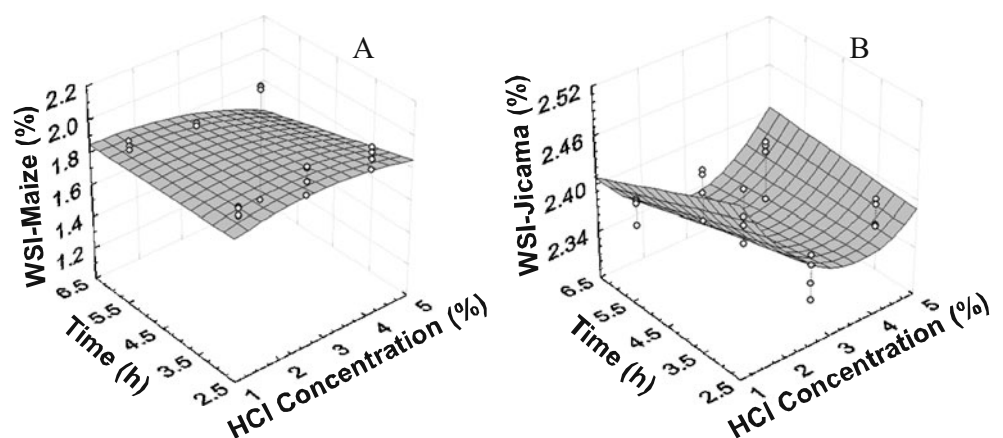
Fig. 3 Effect of acid concentration and hydrolysis time on water absorption index (WAI, g H₂O abs/g dry solid) of jicama (b) and maize (a) starches



lyzed jicama starches did not show significant differences with relation to T . According to Beleia et al. (1980), the differences observed between both starches can be attributed to the type of crystalline structure, which depends on the degree of association of the components of the starch, amylose and amylopectin proportions, molecular weight, and its extension of the external chains of amylopectin. Zambrano and Camargo (2002) evaluated the effect of the interaction of T (3, 6, 9, 11 h), AC (1.5, 2.0, 3.0, 4.0, 4.5%), and hydrolysis temperature (40, 43, 45, 47, 51, 54°C) on diverse characteristics of cassava starch. These researchers found that treatment with 3.0% HCl, 40°C/6 h, showed appropriate functional properties of gel formation, thermo-reversibility, and WAI and WSI with desirable low hydrolysis degree and functionality as a fat replacer in cakes. Lawal et al. (2005) reported that an increase in swelling and solubility in oxidized and thinned hybrid maize starch was a result of increased mobility of starch molecules, which facilitated easy percolation of water. Also, these researchers suggested that an increase in the starch crystallinity possibly restricted absorption of both water and oil within the matrices of starch molecules. Only jicama starch was affected significantly on WSI for most of

the terms of the model (p of $F < 0.05$) except the interaction AC– T (Table 3). Figure 4 shows the WSI behavior with relation to the analyzed variables. An increase in WSI of hydrolyzed starches was found in relation to native starches and was higher for jicama starches than those of maize starch that were not significant, which indicated that jicama starch was more susceptible to degradation due to hydrolysis process than maize starch (Fig. 4). According to diverse researchers, acid thinning increased the solubility of starches (Whistler and Daniels 1990; Kim and Ahn 1996; Osunsam et al. 1989; Sandhu et al. 2007). Kim and Ahn (1996) reported an increase in the solubility of red bean maize starches hydrolyzed with acid, attributed to the damaged structures of starch granules. The severity of acid hydrolysis increased the amount of soluble carbohydrates (Kerr et al. 2000). During acid hydrolysis, the acid first attacks the amorphous zone of the granule, degrading its structure according to the reaction conditions that is reflected in the WSI (Aboubacar and Hamaker 2000). Basman et al. (2005) reported that acid-modified maize starch samples (0.5 to 4.0h hydrolysis time with 6% HCl) showed viscosity values, molecular weight, and solubility lower than those for native starch.

Fig. 4 Effect of acid concentration and hydrolysis time on water solubility (WSI) index of jicama (b) and maize (a) starches



Pasting Properties

Maize and jicama hydrolyzed starches showed low viscosity values in relation to the native starches (Table 4). The effect of the hydrolysis that the amorphous region of the granules of starch undergoes was showed by the fall in viscosity. The viscosity profiles of jicama starch compared with maize starch showed a singular behavior, especially native starch. Native jicama starch showed low viscosity values, with a peak that gradually increased during heating and cooling cycles, suggesting a lower internal stability of the starch granules during hydrolysis. As hydrolyzed jicama starches reached maximum viscosity, the viscosity decreased during the cycle at constant temperature, indicating that the granules are not able to maintain their structure. Further, in general, when the cooling cycle started, the viscosity decreased. The retrogradation of jicama starch was higher than maize starch in the assays T7 and T8, therefore the reduction as a result of the acid hydrolysis was more evident in jicama starch at low AC concentrations, which reached the lowest values as hydrolysis time and acid concentration were increased. Hydrolysis time and acid concentration practically did not affect the initial temperature of gelatinization of maize starch; however, the values of hydrolyzed jicama starch were decreased. In general, the retrogradation values were decreased for both starches during hydrolysis and hydrolyzed jicama starches showed the lowest values in some samples. The maximum viscosity of hydrolyzed maize starch samples did not show significant differences with 3 h of hydrolysis time for all the acid concentrations used. However, a greater reduction of viscosity values was found for jicama starch with 3.0% or

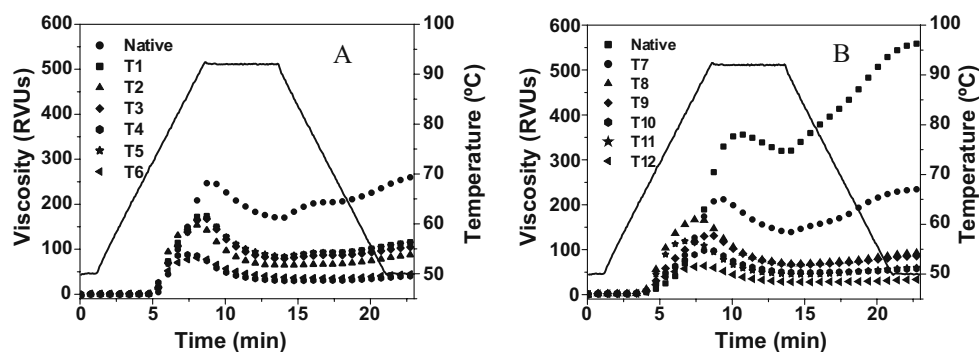
4.5% AC that had a significant difference with the samples hydrolyzed with 1.5% AC that reveals greater susceptibility of jicama starch to the effects of high concentrations of hydrochloric acid. The decrease in viscosity values during the cooling cycle, according to Vasanthan and Bhatti (1996) and Zobel (1988a), was attributed to a decrease of lintnerized hydrated starch granules. The retrogradation rate of acid-thinned starch gels increased as hydrolysis proceeded (Kang et al. 1997). Acid modification increased solubility and gel strength and decreased the viscosity of starches (Kim and Ahn 1996; Osunsam et al. 1989). According to Wang and Wang (2001), the behavior of acid-thinned starches is associated with the average chain length polymerization degree (PD). Hydrolyzed maize starches (Fig. 5) showed a decrease in final viscosity, decreasing retrogradation, even though these values were higher than hydrolyzed. Acid-thinned maize starches showed a higher final viscosity, indicating that gelatinized acid-thinned maize starch re-associated and formed a network structure faster than did gelatinized acid-thinned jicama starch, attributed to its low amylose content (12%). Han et al. (2002) reported that the increase of damaged starch decreased starch viscosity considerably. It is possible that the hydrolysis causes a significant breaking of the glycoside linkages of the long amylopectin chains, which apparently causes the fall in viscosity. Acid-treated rice starches with hydrochloric acid at different acid concentrations and hydrolysis times showed that acid concentration had a more pronounced effect on the degree of polymerization (DP) and viscosity than hydrolysis time (Thirathumthavorn and Charoenrein 2005). The higher susceptibility of jicama starch than maize starch to the

Table 4 Viscosity values of native and hydrolyzed maize and jicama starches

Sample	Initial temperature (°C)	Maximum viscosity at 92 °C (RVUs)	Minimum viscosity (RVUs)	Final viscosity (RVUs)	Retrogradation (RVUs)
Jicama	71.5	363.9	317.0	561.1	244.1
Maize	74.7	249.9	168.3	262.3	94.0
T1	72.6	177.5	81.4	116.4	35.0
T2	72.6	154.7	62.8	87.9	25.1
T3	72.3	170.8	79.0	105.5	26.5
T4	72.4	88.4	29.7	41.0	11.3
T5	72.8	174.3	83.5	114.7	31.2
T6	72.6	81.3	36.0	45.4	9.4
T7	67.9	214.9	138.2	234.9	96.67
T8	66.2	176.1	66.3	93.5	27.2
T9	66.1	133.1	66.3	86.0	19.7
T10	65.7	99.8	48.6	57.1	8.5
T11	66.4	133.1	43.6	60.5	16.9
T12	64.6	63.7	27.7	33.7	6.0

RVUs relative viscosity unities

Fig. 5 Effect of acid concentration and hydrolysis time on viscosity profiles of jicama (b) and maize (a) starches



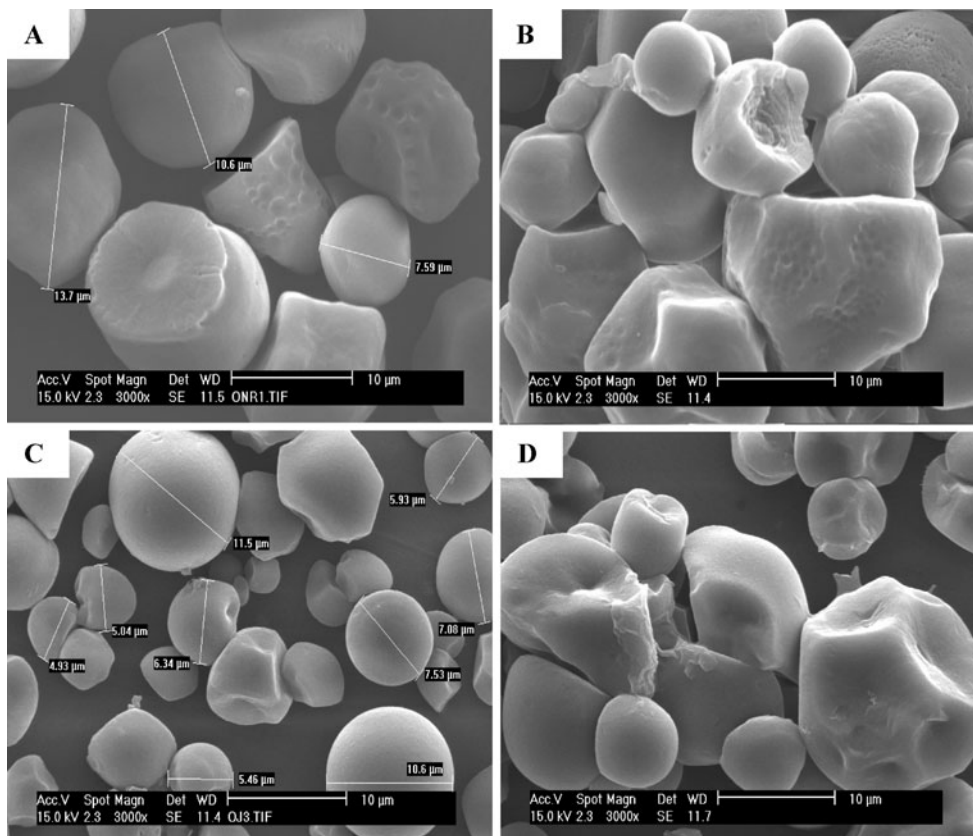
hydrolysis conditions was reflected in the sugar content release during hydrolysis; from 7.62 to 114.50 mg/g for hydrolyzed maize starches and from 14.42 to 210.70 mg/g for hydrolyzed jicama starches. Tester and Morrison (1990) reported that lintnerized starches that develop a low gelatinization temperature had less crystallinity than their counterparts, which develop high gelatinization temperatures due to smaller structural differences in amylopectin.

Scanning Electron Microscopy (SEM)

Figure 6 shows the structure of jicama (a) and maize (c) starches. The granules of native jicama starch showed round and polygonal form similar to native maize starch,

but had smaller size that varied from 3.77 to 11.50 μm , whereas those of native maize showed a size that varied from 4.68 to 15.90 μm . It is also possible to observe the degradation caused by the effect of hydrochloric acid (Fig. 6b, d). Both hydrolyzed starches were more porous (small orifices), due to the degradation of the amorphous zone and to the leaching caused by acid. Native maize starch showed a more uniform surface with respect to hydrolyzed jicama starch, indicating that jicama starch was more susceptible to the acid hydrolysis using the same hydrolysis conditions. Jayakody and Hoover (2002) postulated that the pores in the surface of the granules allow the direct access of acids to the interior of the granule. This behavior corroborated the higher starch damage found in

Fig. 6 Microphotographs (SEM) of native (a) and hydrolyzed maize starch, assay T1 (b), and native (c) and hydrolyzed jicama starch, assay T7 (d)



the samples of jicama starch. According to Atichokudomchai et al. (2000) when increasing the hydrolysis time from 12 to 96 h, an exposed corrosion on the surface of the granules is observed, which is not observed with low hydrolysis times similar to those observed for native starches. Franco et al. (2002) did not find any difference in the external structure of commercial native cassava starch and commercial fermented cassava starch hydrolyzed with 2.2N HCl at 38°C. However, observation under a scanning electron microscope exposed fissures in the granules. Wang and Wang (2001) found that acid modification changed the physico-chemical properties of acid-thinned maize, potato, and rice starches without destroying their granule structure, and the properties of the acid-thinned starches differ according to their origins. Sandhu et al. (2007) reported that due to the low level of hydrolysis of maize starch, the microscopy did not show any destruction of granular structure upon acid thinning. According to Shujun et al. (2007), the acid corrosion started from the exterior surface of B-type starch which started to crack at the hydrolysis period of 4 days. The crystalline type of B-type starch was not changed with increasing hydrolysis time.

X-Ray Diffraction and Crystallinity

X-ray powder diffraction has been used to reveal the presence and characteristics of the crystalline structure of the starch granules (Hoover 2001). The X-ray diffraction patterns of the acid-thinned starches of maize and jicama, and their native counterparts, are shown in Fig. 7. The acid-thinned samples showed similar X-ray patterns to the native starches, with the distinctive attribute of A-type starch of cereals with strong peaks at 2θ about 15°, 17°, 18°, and 23°. The intensity of the three main peaks increased for the samples of maize starch hydrolyzed with 1.5% or 3.0% of HCl for 3 h. X-ray diffraction patterns of native and acid-modified jicama starch showed the distinctive attributes of B-type starch of tubers with the strongest diffraction peak at 17° 2θ . There were also a few small peaks at 2θ values of

20°, 22°, and 24° (Cheetham and Tao 1998; Zobel 1988b). Kainuma and French (1971) suggested that cleavage of starch chains in the amorphous regions allows re-ordering of the chain segments to give a more crystalline structure with a sharper X-ray pattern. The relative crystallinity values and standard deviation values are shown for native and hydrolyzed starches in Fig. 8. The relative crystallinity of hydrolyzed maize starches decreased during hydrolysis, while those of hydrolyzed jicama starches increased, and the highest relative crystallinity values were for the samples T5 and T7 of hydrolyzed jicama starch (1.5/3 h and 4.5/3 h), this being attributable to the lower amylose content and relative crystallinity of native jicama starch compared to native maize starch. According to Biliaderis et al. (1980), the hydrolysis of the amorphous zones allows the reorganization of the segments of the chains and the formation of a more crystalline structure. The relative crystallinity of hydrolyzed maize starches decreased with the acid treatment, indicating weaker internal structures as a result of the acid attack. According to some authors (Atichokudomchai et al. 2002; Kainuma and French 1971), amylopectin content and average chain length are among the factors that influence the degree of hydrolysis, and the amorphous regions in different starches might be different in terms of dimension and molecular arrangement. Atichokudomchai et al. (2000) found that, with cassava starch hydrolyzed with hydrochloric acid, an increase in hydrolysis caused a decrease in the amylose content as well as an increase in the relative crystallinity similar to the behavior found in hydrolyzed jicama starch.

Thermal Properties

Native and hydrolyzed maize starches had higher enthalpy, onset (T_o), peak (T_p), and conclusion (T_c) temperatures than jicama starches (Table 5). The broadening of the endotherms and enthalpy of gelatinization decreased during the hydrolysis of both starches, compared to their counterpart native starches. A reduction in the ΔH occurs because acid primarily attacks the amorphous regions in the granules and

Fig. 7 Diffractograms of native maize starch (NMS) and hydrolyzed maize starch (a) and native jicama starch (NJS) and hydrolyzed jicama starch at different hydrolysis conditions (b)

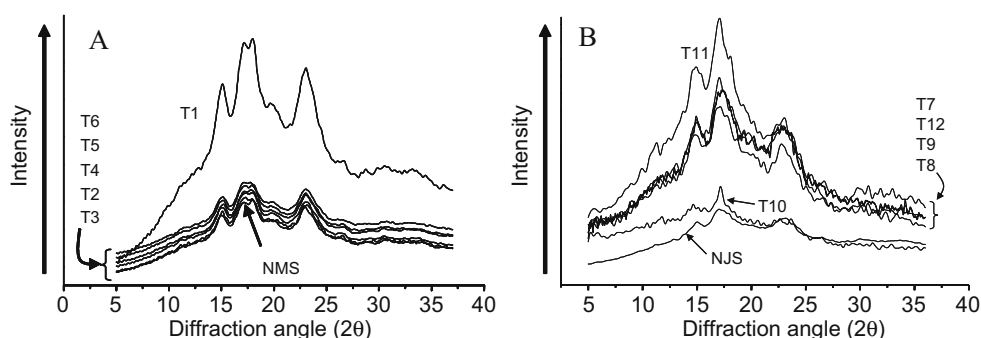
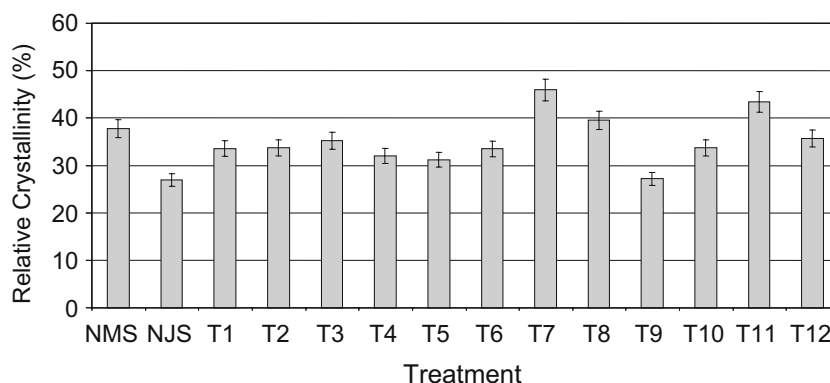


Fig. 8 Relative crystallinity of native (NMS, NJS) and hydrolyzed maize and jicama starches



these regions play an important role in the thermodynamics of gelatinization (Blanshard 1987). Sandhu et al. (2007) found similar values to those found in this research of T_o , T_p , and ΔH for native maize starch (65.6–68.3, 69.9–73.3°C, and 11.3–11.6 J/g, respectively), whereas their counterparts of acid-thinned starches showed higher values (66.9–69.2, 71.6–74.6°C, and 10.2–10.9 J/g, respectively). A similar decrease in the enthalpy value after acid treatment has been shown earlier (Wang and Wang 2001; Wang et al. 2003). Muhr et al. (1984) attributed this decrease to the loss of some degree of order of the amorphous regions prior to gelatinization. Some variability in the thermal behavior may exist within populations of maize and significant differences among plants within the same population due to fine structural differences during development and kernel maturity, and environmental effects (White 2004; Eyherabide et al. 2007). Diverse studies on thermal transition of thinned tapioca, potato, waxy and non-waxy barley, rice, wheat, and

pea starches have also shown that their thermal properties appear over a broader temperature range and are higher in onset and peak temperatures as compared to their counterpart native starch endotherms (Jenkins and Donald 1997; Morrison et al. 1993; Shi and Sieb 1992; Chun et al. 1997; Jacobs et al. 1998). During acid thinning action, the crystallites are decoupled from and no longer destabilized by the amorphous parts. Consequently, the starch crystallites of the hydrolyzed starches melt at higher temperatures and exhibit a broader temperature range. Atichokudomchai et al. (2002) reported that, in the case of potato starch, as the hydrolysis time increased, T_o , T_p , and T_c temperatures of gelatinization also increased, up to a critical value, then decreased with a significant broadening of the endotherms. According to Biliaderis (1991), the enthalpy of transition is significantly affected by the rate of heating, the content of water, and by the proportion of damaged starch in the samples. Thirathumthavorn and Charoenrein (2005) reported that the onset, peak, and conclusion temperatures of gelatinization of acid-hydrolyzed rice starch were increased significantly with hydrolysis time, while the gelatinization enthalpy was decreased.

Table 5 Gelatinization parameters of native and acid-thinned jicama and maize starches

Sample	ΔH (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	$T_c - T_o$
Maize	-11.1	66.1	71.1	76.6	10.5
Jicama	-9.5	52.3	58.4	67.5	15.2
T1	-11.1	65.8	70.3	74.6	8.8
T2	-11.6	64.9	70.0	75.5	10.7
T3	-11.0	65.8	70.2	74.7	8.8
T4	-10.8	66.0	70.2	75.1	9.1
T5	-10.7	66.2	70.6	75.3	9.1
T6	-11.0	66.9	71.3	76.1	9.2
T7	-9.0	59.4	62.5	68.7	9.3
T8	-9.2	59.5	63.3	69.4	10.0
T9	-8.8	58.7	62.5	70.1	11.4
T10	-9.0	60.1	63.7	70.7	10.6
T11	-8.8	59.1	63.0	70.7	11.6
T12	-8.7	59.7	63.4	70.4	10.8

Gelatinization parameters: T_o =onset, T_p =peak, and T_c =conclusion temperatures. ΔH =gelatinization enthalpy

Conclusions

The physico-chemical characteristics of normal maize and jicama starches were influenced after similar acid treatment. Jicama starches were more prone to acid hydrolysis than hydrolyzed maize starches, in terms of damaged starch, water absorption index and water solubility index, viscosity, and crystallinity values. In general, hydrolyzed maize starches decreased their thermal properties relative to native starch, whereas in hydrolyzed jicama starches these values were increased.

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