

Selective Nixtamalization of Fractions of Maize Grain (*Zea mays* L.) and Their Use in the Preparation of Instant Tortilla Flours Analyzed Using Response Surface Methodology

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ABSTRACT

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Using a continuous decorticating machine, white dent corn was efficiently separated, after brief steeping in water, into two fractions: the first (12.5%) consisting mainly of pericarp, germ, and tip cap (PGT); the second (87.5%) consisting of endosperm. Nixtamalization of the maize fractions in the presence of 0.6% (w/w) lime caused an increase in the hot-paste viscosity at 90°C, while nixtamalization of PGT at lime inputs <0.6% (w/w) resulted in decreased viscosity. Three domains were found for the viscosity of nixtamalized endosperm at 90°C: lower concentrations of lime (<0.15%, w/w) resulted in lower viscosity values; increased lime

(0.15% – <0.3%, w/w) increased the viscosity values; and a lime concentration of 0.3% (w/w) resulted in a lower viscosity value. The response variables (water absorption index, water solubility index, initial viscosity, and viscosity at 90°C for nixtamalized PGT, and compression force and compression area of tortillas) indicated that the mathematical models fit the experimental data and the variance of the models was highly significant. Tortillas of good functional characteristics similar to tortillas produced by the traditional process were obtained when 5% nixtamalized fractions of PGT were blended with 95% nixtamalized endosperm.

Tortilla production is one of the industrial sectors of highest socioeconomic impact in Mexico, creating micro industries and 225,000 direct and indirect jobs (Gargallo 2000). An important part of the Mexican population obtains 70% of their caloric intake, 50% of their protein intake, and 37% of their calcium requirement from tortillas produced from nixtamalized maize (Paredes-Lopez and Saharopulos 1982; Trejo-González et al 1982). The traditional process for producing nixtamalized flour has not been improved substantially in recent years; it still suffers from the drawbacks of generating contaminating effluents on a large scale, long processing time, and high water use (1.2 m³/ton of processed maize). It also carries an important energy cost due to the low efficiency of heat transfer during tortilla baking. The severe alkaline-heat treatment involved in traditional nixtamalization causes losses of 8.5–12.5% of dry mass, containing fractions of pericarp, starch, protein, and germ. These losses increase with the use of higher concentrations of lime, higher processing temperature, and longer cooking and steeping time (Plugfelder et al 1988; Sahai et al 2001).

Previous efforts have been directed at improving the efficiency of the traditional nixtamalization process and the quality of the end product. These alternative processes include the use of steam and pressure (Lloyd and Millares-Sotres 1952; Mendoza 1975; Bedolla and Rooney 1982; Montemayor and Rubio 1983; Sterner and Zone 1984), traditional processes using prior steeping of the maize grain (Morad et al 1986), milling and drying of the cooked grain (Diez de Sollano and Berriozabal 1955), spray dryers (Molina et al 1977), microwaves (Martínez-Bustos et al 1996), dielectric cooking (Gaytán et al 1997), infrared cooking (Johnson et al 1980), and extrusion (Mensah-Agyapong and Horner 1992; Martínez-Bustos 1996). Vaqueiro and Reyes (1986) and Martínez-Montes et al (2001) described a process of selective nixtamalization of the components of the maize grain. This process produced tortillas with characteristics similar to those prepared using the traditional process, with an efficient use of water, energy, processing time, and use of the whole maize grain.

The aim of this work was to evaluate the process of selective nixtamalization using response surface methodology to obtain tortillas

with functional properties similar to those prepared by the traditional nixtamalization process.

MATERIALS AND METHODS

Commercial white dent maize (grown in the state of Guerrero, Mexico, 1999) and calcium hydroxide (Merck) were used. Ten different samples of commercial dough prepared by the traditional nixtamalization process were obtained from commercial mills (México D.F., Mexico) and used as a control.

Clean maize grain (5 kg) was steeped in water (1:0.8, w/w) at different temperatures and stirred intermittently following the experimental design shown in Table I. The steeped grain was fed into a continuous decorticating device (CICATA-IPN, Mexico), keeping the residence time constant at 1 min and the screw speed constant at 800 rpm. The fractions were separated using pneumatic equipment (CICATA-IPN, México D.F., Mexico).

Nixtamalization of Samples

The fractions were nixtamalized separately in a stainless-steel double-arm blender (Type Z, Teledyne Readco, NY) equipped with temperature control and a steam-heated jacket. First, the alkaline solution was added to the blender and heated to a temperature close to that of the experimental design. Then the maize fraction was added, with continuous heating, until the temperature specified in the experimental design (Tables II and III) was reached. The pericarp, germ, and tip cap (PGT) fractions were nixtamalized using 200 g of PGT fractions in each assay. The variables and variation levels are shown in Table II. The cooking temperature was kept constant at 92 ± 2°C. The endosperm was nixtamalized using 1,000 g in each assay. All the assays were performed according to the experimental design shown in Table III. The ratio of water to endosperm was kept constant at 1:1 (w/w). Samples were dried at 60°C for 5 hr using a tray dryer (F.J. Stockes Corp., PA) and milled at 3,000 rpm using a hammer mill (Mikro Pulverizer, model 2TH, stirrup type) equipped with a 60-mesh sieve.

Physical Properties of Samples

The moisture content of the samples was determined according to Approved Method 44-15A (AACC 2000). Water absorption index (WAI) and water solubility index (WSI) were analyzed following the method of Anderson et al (1969).

A Rapid Visco Analyzer (RVA-4; Newport Scientific Pty, Australia) was used to perform viscosity measurements on the maize fractions. Samples of 4 g each were adjusted to 14% (w/w) moisture. Distilled water was added to keep the total weight of water and

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sample constant at 28 g. Rotating paddles were used to ensure uniform dispersion. The mixture was maintained at room temperature for 8 sec to stabilize the temperature, warmed up over 5 min from 50 to 90°C, and then held at a constant temperature of 90°C for 4.50 min. The samples were then cooled down to 50°C over 5.50 min. The total time for this test was 15 min.

The consistency of the dough was determined following the shear force or dough extrusion method proposed by Khan et al (1982). Samples of 35 g each were placed in a cylinder and compacted using a piston; then the dough was forced through an orifice 1.25 cm in diameter at a speed of 1.5 mm/sec using a cylindrical Perspex piston.

Tortilla Preparation and Evaluation

Tortillas were prepared by blending different proportions of nixtamalized PGT flours and nixtamalized endosperm fraction flours according to the experimental design and variation levels shown in Table IV. The PGT flour proportion was brought up to 100% by adding nixtamalized endosperm flour. The dough was prepared by mixing the fractions with water in a domestic blender (Kitchen Aid, MK4588WH) until a dough consistency similar to that of the control (7,000 g force) was achieved.

The fresh dough was rounded and shaped into flat disks using a manual machine (Casa Herrera, México D.F., Mexico). The dough disks were baked on a hot griddle at $270 \pm 10^\circ\text{C}$ for 15 sec on one side, followed by 30 sec on the other side, and then again on the first side until puffing of the tortilla occurred. The tortillas were 1.19 ± 0.1 mm thick, 12.5 ± 0.1 cm in diameter, and weighed 20 ± 0.5 g. Tortillas from each treatment ($n = 20$) were evaluated after cooling for 30 min at room temperature (25°C).

TABLE I
Experimental Design for Steeping Maize Grain: Coded and Real Values

Trial	Coded Values		Real Values	
	x_1^a	x_2^b	X_1^a	X_2^b
1	-1	-1	30	10
2	-1	1	30	20
3	1	-1	50	10
4	1	1	50	20
5	0	0	40	15
6	0	0	40	15
7	0	0	40	15
8	0	0	40	15
9	0	0	40	15
10	1.414	0	54	15
11	-1.414	0	26	15
12	0	1.414	40	22
13	0	-1.414	40	8

^a x_1 and X_1 , steeping temperature ($^\circ\text{C}$).

^b x_2 and X_2 , steeping time (min).

TABLE II
Variables and Variation Levels for Pericarp, Germ, and Tip Cap (PGT) Nixtamalization

Variables	Variation Levels				
	-1.682	-1	0	1	1.682
x_1 = Ratio of water to PGT	3.5:1	4:1	4.75:1	5.5:1	6:1
x_2 = Lime (% w/w)	0	0.16	0.4	0.64	0.8
x_3 = Cooking time (min)	10	12	15	18	20

TABLE III
Variables and Variation Levels for Endosperm Nixtamalization

Variables	Variation Levels				
	-1.682	-1	0	1	1.682
x_2 = Lime (% w/w)	0	0.09	0.15	0.24	0.3
x_3 = Cooking time (min)	1.2	4.40	7.0	10.20	12.0
x_3 = Cooking temp. ($^\circ\text{C}$)	70.0	74.5	81.0	87.5	92.0

The texture of the tortillas was determined using a texture analyzer (TA-XT2i, Texture Technologies Corp., Scarsdale, NY). The compression force (CFT) and compression area of the tortillas (CAT) were analyzed using an HDP/TPB pastry burst ring. The ring consists of two plates that can be bolted together with the sample sandwiched in between. The plates have holes at the center exposing a circular section of the tortilla, allowing an acrylic sphere 2.5 cm in diameter to be pushed through. This test was conducted at a speed of 2 mm/sec to a depth of 30 mm.

Experimental Design and Data Analysis

All treatments were performed randomly and the data was analyzed by response surface methodology using TS020 version 6.12 SAS software (SAS Institute, Cary, NC). The significance of the models was tested using variance analysis (F test) and the correlation coefficient R^2 . The effect of the variables was displayed in surface graphs. A compound central experimental design with 13 treatments and five repetitions in the central point was used for the fractions steeping process. A compound central design (Table V) of 20 assays with six repetitions in the central point (Montgomery 1999) was used for the nixtamalization process and tortilla preparation.

RESULTS AND DISCUSSION

Separation of Fractions

The model for the separation percentage (SP) of the PGT fractions from the endosperm fraction was

$$SP = 20.55485966 - 0.29823178 x_1 - 0.53556056 x_2 + 0.015085 x_1 x_2 \quad (1)$$

where: x_1 is the steeping temperature and x_2 is the steeping time. The significance of the model was $R^2 = 0.315$ and $P = 0.3099$.

The PGT separation percentage decreased when the temperature was increased with steeping time constant at 8 min, whereas the separation percentage gradually increased at higher steeping time combined with higher temperature, reaching an 11% separation percentage at 22 min and 54°C . However, the best SP (12.5%) was obtained at a steeping temperature of 26°C and a steeping time of 8 min (Fig. 1). A value of 12.5% (or close to this value) indicates that all of the pericarp (5.3%), tip cap (0.8%), and germ (6.4%) was efficiently separated from the grain, as similar values for the composition of components of maize grain were reported by Watson and Ramstad (1987).

Response Variables

The estimated regression coefficients for each dependent variable were calculated from the results and the model fit for each equation was tested using the correlation coefficient R^2 value and the F test probability for each dependent variable.

The correlation of WAI and WSI viscosity was $R^2 < 0.6$ at 90°C for nixtamalized endosperm, indicating that the mathematical models fit the experimental data. The probability F value indicated that the variance of the models was highly significant.

Viscosity for Nixtamalized PGT Fractions

The best explanatory equation for the initial viscosity (IVIS) of the nixtamalized PGT fractions showing a relationship between independent variables such as the ratio of water to PGT (x_1), lime concentration (x_2), and cooking time (x_3) is

$$\begin{aligned} IVIS = & -386.8507865 + 66.4218426 x_1 - 219.2336215 x_2 \\ & + 40.0027073 x_3 + 108.5939170 x_2^2 + 97.6092065 x_1 x_2 \\ & - 6.9029939 x_1 x_3 - 21.7773706 x_2 x_3 \end{aligned} \quad (2)$$

The model was sufficiently accurate ($R^2 = 0.6619$ and $P = 0.0653$). The statistical analyses indicated that the lime concentration ($P < 0.0618$), as well as the interaction of water to PGT to lime concentration ($P < 0.0729$) were significant and influenced the viscosity.

The equation for viscosity at 90°C (V90°C) was

$$V90^{\circ}\text{C} = -643.0409311 + 202.5939728 x_1 - 2.8355420 x_2 + 28.3635711 x_3 - 14.7019666 x_1^2 - 74.0905334 x_2^2 + 43.9481472 x_1 x_2 - 5.4081963 x_1 x_3 - 8.4225223 x_2 x_3 \quad (3)$$

This model was not significant ($P > 0.1721$ $R^2 = 0.5722$).

The V90°C of nixtamalized PGT was related to the ratio of water to PGT and to the lime concentration used during the process. The maximum values of IVIS (101 cP) were found using a water to PGT ratio of 6:1 (w/w) with 0.8% lime and a cooking time of 10 min (Fig. 2). Hydrolysis of the components of the outer layers of the grain increased (viscosity close to zero) when the cooking time was increased from 15 to 20 min at the maximum lime concentration (0.8%, w/w) and a 6:1 (w/w) ratio of water to PGT. Decreasing the ratio of water to PGT to 3.5:1 without adding lime increased viscosity values, which was attributable to less hydrolysis of the components of the outer layers of the grain.

The values for V90°C showed an hydrolysis tendency similar to that observed for IVIS. Higher viscosity values were found with 0.8% (w/w) lime, a ratio of 6:1 for water to PGT, and a cooking time of 10 min (Fig. 3). Increasing the cooking time to 15 or 20 min reduced the viscosity values. Nixtamalization of isolated pericarp causes the rupture of the cellulose-hemicellulose-lignin structure and the concomitant release of hemicellulose and small quantities of lignin and proteins into the solution, thus improving the rheological quality of the tortillas (Martínez et al 2001). According to ^{13}C NMR, the hemicellulose isolated from cooking liquid consists mainly of galactoglucuronoarabinoxylans (Saulnier et al 1993). The use of 2 or 5% lime caused a small loss of the mass of the pericarp, accompanied by an abrupt fall in the concentration of the OH-ions. A suitable kinetic model for the decrease in the mass of the pericarp is a two-phase experimental equation. The second phase is the slower one and it depends on the quantity of lime employed

TABLE IV
Variables and Variation Levels for Preparation of Tortillas

Variables	Variation Levels				
	-1.682	-1	0	1	1.682
x_1 = PGT ^a (% w/w)	5	7	10	13	15
x_2 = PGT initial viscosity (cP)	0	20	50	80	101
x_3 = Endosperm viscosity (cP)	916	1,173	1,549	1,925	2,182

^a Pericarp, germ, and tip cap (PGT).

TABLE V
Experimental Design for Nixtamalization of Fractions and Tortilla Preparation

Trial	Coded Values		
	x_1	x_2	x_3
1	-1	-1	-1
2	-1	-1	1
3	-1	1	-1
4	-1	1	1
5	1	-1	-1
6	1	-1	1
7	1	1	-1
8	1	1	1
9	-1.682	0	0
10	1.682	0	0
11	0	-1.682	0
12	0	1.682	0
13	0	0	-1.682
14	0	0	1.682
15	0	0	0
16	0	0	0
17	0	0	0
18	0	0	0
19	0	0	0
20 ^a	0	0	0

^a Central point repeated six times.

(Martínez et al 2001). Alkaline cooking alters the outer layers of the grain, whereby its components are hydrated and the pericarp fraction assumes a gummy and sticky texture caused by the release of pericarp gums. This improves the viscosity, cohesiveness, and adhesiveness of the tortillas (Martínez-Bustos et al 2001).

Viscosity at 90°C and 50°C for Nixtamalized Endosperm

The viscosity of the nixtamalized endosperm at 90°C is described as

$$V90^{\circ}\text{C} = -28098.37231 + 32253.32047 x_1 - 117.98881 x_2 + 721.01738 x_3 - 40006.49899 x_1^2 - 4.51190 x_3^2 - 231.37109 x_1 x_3 + 1.44853 x_2 x_3 \quad (4)$$

This assumed model was significant at the 5% level ($R^2 = 0.6489$, $P = 0.0384$).

The viscosity of the nixtamalized endosperm at 50°C is described as

$$V50^{\circ}\text{C} = -33376.58519 + 15096.66177 x_1 - 163.17644 x_2 + 908.93543 x_3 - 70884.58322 x_1^2 - 5.81258 x_3^2 + 1290.19841 x_1 x_2 \quad (5)$$

This assumed model was significant at the 5% level ($R^2 = 0.5837$, $P = 0.0439$).

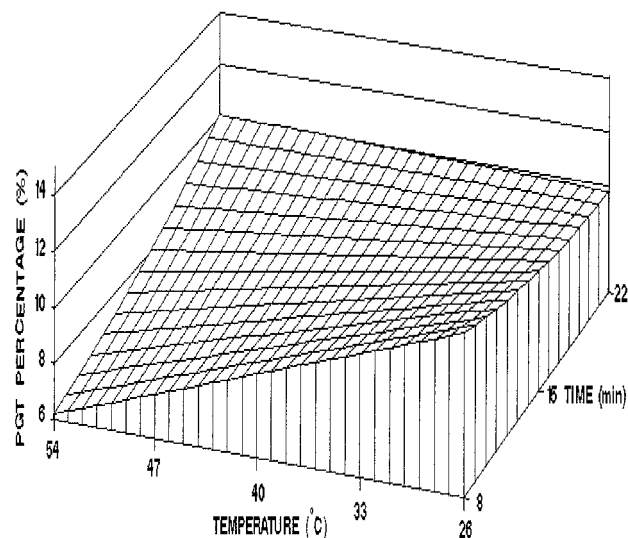


Fig. 1. Separation of pericarp, germ, and tip cap (PGT) as a function of nixtamalization temperature and nixtamalization time.

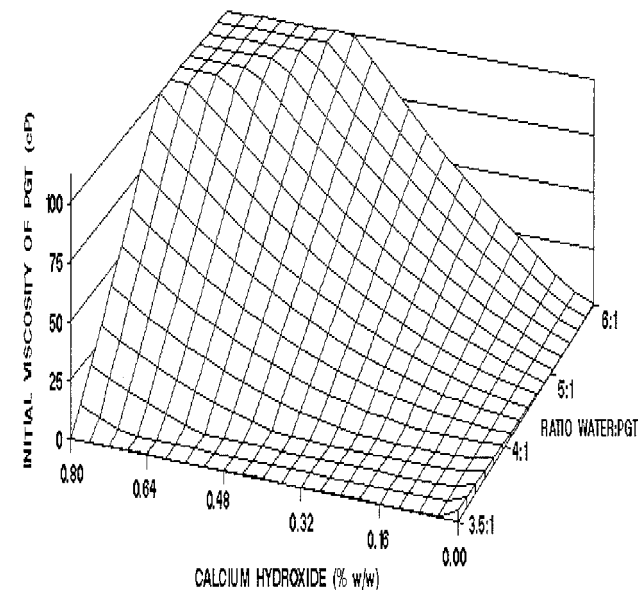


Fig. 2. Initial viscosity of nixtamalized pericarp, germ, and tip cap (PGT) cooked for 10 min.

V90°C and V50°C were influenced by the linear terms of the lime concentration and of the nixtamalization temperature; the nixtamalization time had a negative effect. In this case, only the quadratic term of the lime concentration was highly significant for V90°C ($P < 0.0034$) and V50°C during the cooling cycle ($P < 0.0036$).

Figure 4 shows the V90°C for endosperm nixtamalized at 80°C. With no input or low input of lime, starch granules undergo gelatinization, leaching, and hydrolysis of amylose and amylopectin, resulting in lower viscosity values (916 cP). The interaction of the components of the starch granules with the lime increases with rising lime concentration (0.15%, w/w). The formation of complexes and increased interaction of the hydroxyl groups of the amylose chains and amylopectin with the Ca^{++} ions or $\text{Ca}(\text{OH})^+$ in the starch granules and probably increased the viscosity values (1,866 cP). Higher concentrations of lime (0.3%, w/w) resulted in lower viscosity values (1,233 cP), probably attributable to leaching and hydrolysis of some components of the starch granule. Nurul-Islam and Mohd-Azemi (1992, 1994) and Fernandez-Muñoz et al 2002 found that calcium interacts with starch granules, which results in the formation of complexes.

Rendleman (1966a-c; 1978a,b) indicated that under alkaline pH and

extrusion conditions, starch undergoes hydration and gelatinization. Additionally, starch components could unfold and expose their reactive sites, allowing calcium to bind.

Bryant and Hamaker (1997) reported the effect of lime cooking and lime concentration (from 0.0 to 1.0%) on the viscosity of defatted corn flour-water suspensions using a Brabender amylograph. They found that the hot-paste peak viscosity was highest at 0.1% lime concentration. Further addition of lime (0.1–0.5%) reduced the peak viscosity. The results for hot-paste peak viscosity, minimum viscosity, and final viscosity up to a lime concentration of 0.1% found by these studies is similar to our results, obtained at different lime concentration.

The V50°C for the nixtamalized endosperm (Fig. 5) showed a trend similar to that found at 90°C. However, in this case, the nixtamalization time affected the viscosity values. A longer nixtamalization time (12 min) resulted in a higher viscosity value (3,680 cP) when 0.15% lime and 80°C were applied. A similar behavior was reported for samples of nixtamal and commercial nixtamalized flours, showing a lower initial viscosity and a higher viscosity during heating with decreasing viscosity during the constant temperature cycle and increasing viscosity during the cooling cycle (Flores-Farias et al 2000; Martínez-Bustos et al 2001).

WAI of Nixtamalized Endosperm

This parameter is based on the structural modification of the starch granules and its components and could be described as

$$\begin{aligned} \text{WAI} = & 3.01083949 - 20.3751 x_1 + 0.19289052 x_2 + 0.00148882 x_3 \\ & + 23.88160447 x_1^2 - 0.18823581 x_1 x_2 + 0.17937102 x_1 x_3 \\ & - 0.00190253 x_2 x_3 \end{aligned} \quad (6)$$

This model was significant ($R^2 = 0.6525$, $P = 0.0365$). Only the quadratic lime concentration term was highly significant ($P < 0.0025$). The nixtamalization temperature ($P > 0.0477$) was significant.

A nixtamalization temperature of 70°C and increased lime concentration up to 0.3% led to a pronounced decrease in the WAI (data not shown). However, an increase in the WAI values resulted at 90°C by increasing lime concentration from 0.15 to 0.3% (Fig. 6) with maximum values of 3.6 g of water/g of dry sample. In these conditions, the nixtamalized endosperm flour probably acquires a new structure that is able to retain a greater amount of water and is affected by the nixtamalization time. Studies reported by Thomas and Atwell (1999) indicated that lightly cross-linked starch (starch with a low degree of substitution) tends to show a granular stability and improved paste texture and becomes more resistant to the changes generally associated with cooling and pasting. Also, cross-linking of

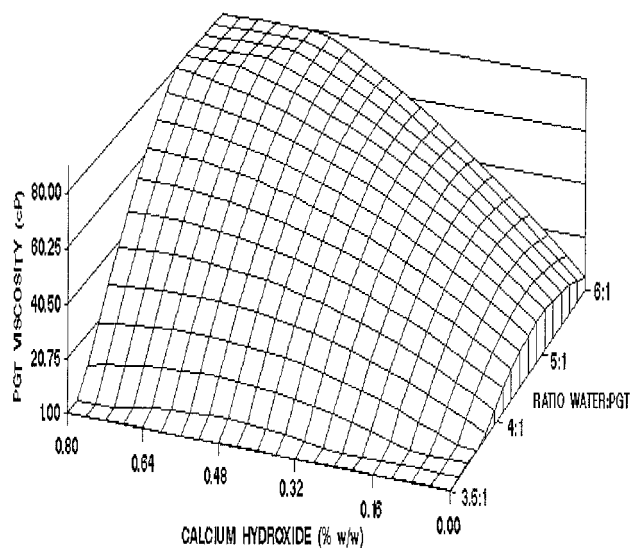


Fig. 3. Viscosity (90°C) of nixtamalized pericarp, germ, and tip cap (PGT) cooked for 10 min.

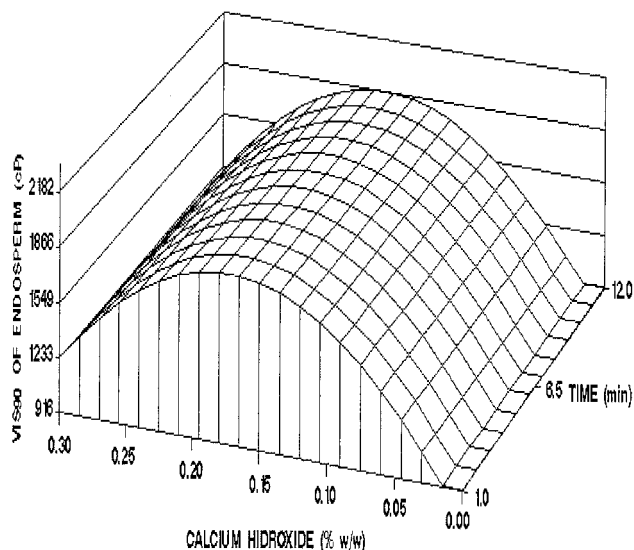


Fig. 4. Viscosity (90°C) of nixtamalized endosperm cooked at 80°C.

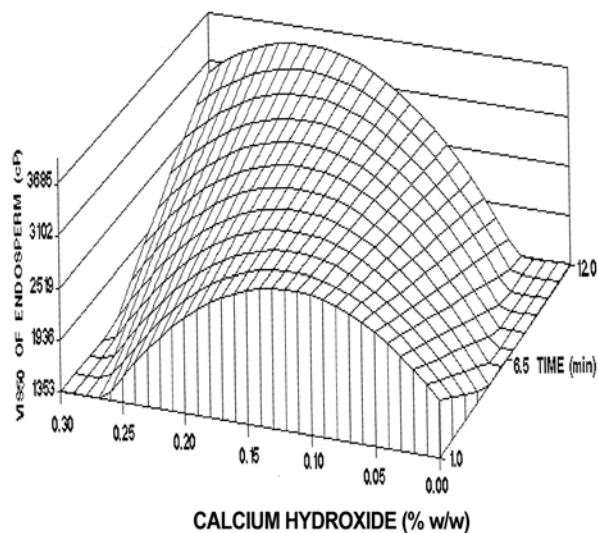


Fig. 5. Viscosity (50°C) of nixtamalized endosperm cooked at 80°C.

the starch reduces the amount of the soluble materials that leach out of the swollen granule during cooking (Kite et al 1957). Martínez-Flores et al (1998) reported similar results for the WAI and WSI of extruded corn meal when increasing lime input.

Bryant and Hamaker (1997) reported the effects of treatment with lime at different concentrations (0–1.0%) on the water retention capacity (WRC) of defatted corn flour when cooked in boiling water for 15 min. The results showed that the WRC rose as the lime concentration increased from 0 to 0.1%. However, further increases in lime concentration from 0.2 to 0.6% produced a dramatic decrease in WRC. They postulated that these phenomena were produced by the interaction of Ca^{++} and $\text{Ca}(\text{OH})^+$ cations with starch hydroxyl sites reaching saturation. Contrary to the results of these authors, who found an increased WRC at a lime concentration of 0.1%, the results reported here show a decrease in WAI for 0–0.1% lime and greater WAI values for lime concentrations at 0.1–0.3%. Oosten (1982) suggests that divalent cations bind tightly with starch molecules, causing a decrease in WAI.

WSI of Nixtamalized Endosperm

The WSI exhibited a significant regression model ($P < 0.0029$, $R^2 = 0.859$). The three variables (lime concentration, nixtamalization time, and nixtamalization temperature) had a negative linear effect. The nixtamalization temperature (linear term: $P < 0.003$, quadratic

term: $P < 0.00071$) and the interaction of temperature-lime concentration ($P < 0.0050$) were highly significant.

The best explanatory model equation for the WSI is

$$\text{WSI} = 86.23607331 - 74.64093954 x_1 - 1.11303013 x_2 - 1.66599202 x_3 - 0.39022306 x_1^2 + 0.00216651 x_2^2 + 0.00841216 x_3^2 + 0.018566 x_1 x_2 + 0.89369132 x_1 x_3 - 0.00197590 x_2 x_3$$

The WSI is a property that reflects the quantity of soluble solids in the water, indicating the extent of cooking undergone by the nixtamalized flour (Flores-Farias et al 2000). The maximum WSI value for the nixtamalized endosperm was reached at 70°C for 1.0 min with 0.00% lime (data not shown). When the lime concentration was increased, solubility decreased, probably induced by the formation of insoluble water complexes, favored by the higher temperature, as was observed at nixtamalization time of 1.0 and 12 min. It was also observed that the solubility of the endosperm was increased (7.5 WSI) with a 0.3% lime concentration, a cooking temperature of 90°C, and a processing time of 12 min (Fig. 7). This means that the components of the endosperm (starch, proteins, and lipids) were hydrolyzed to a greater extent, thus increasing the solids dissolved

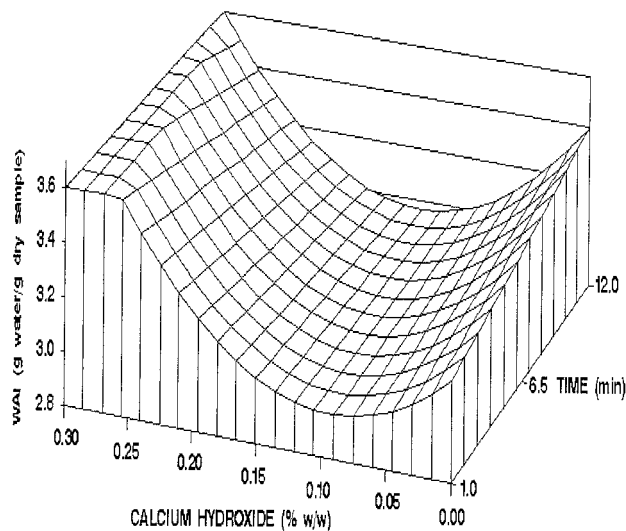


Fig. 6. Water absorption index of nixtamalized endosperm cooked at 90°C.

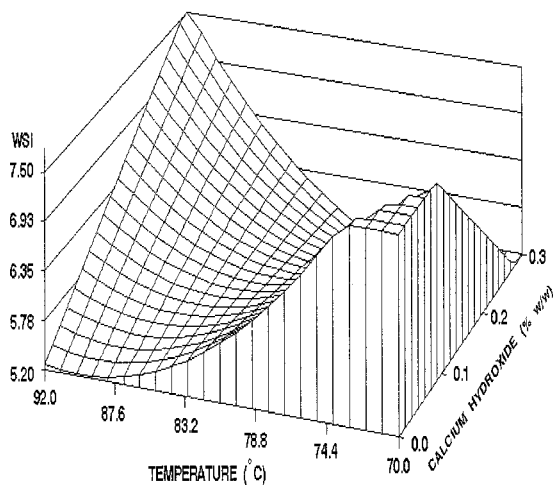


Fig. 7. Water solubility index of nixtamalized endosperm cooked for 12 min.

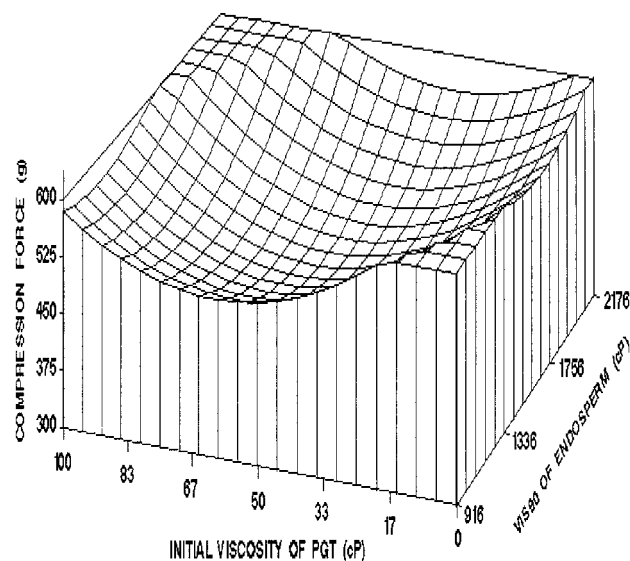


Fig. 8. Compression force of tortillas with 5% pericarp, germ, and tip cap (PGT).

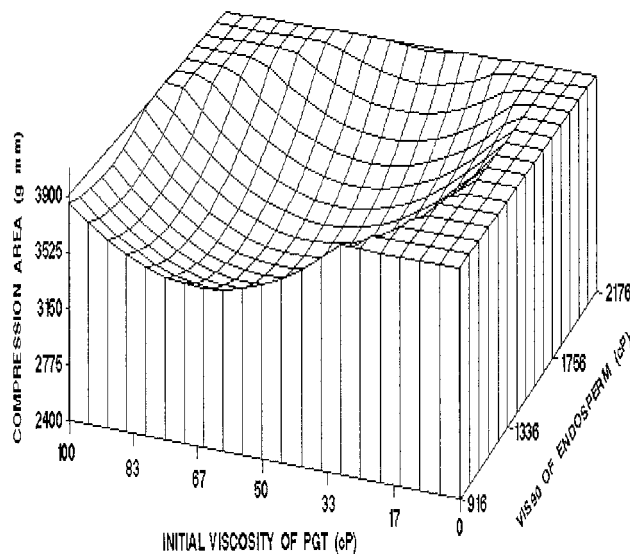


Fig. 9. Compression area of tortillas with 5% pericarp, germ, and tip cap (PGT).

in the water. The WSI of commercial nixtamalized maize flours increased when the WAI decreased (Flores-Farias et al 2000).

The observed results for the WSI may be attributable to ionic interactions of Ca^{++} or $\text{Ca}(\text{OH})^+$ with the hydroxyl groups of amylose and amylopectin, making the molecule more compact (Bryant and Hamaker 1997). The interaction of $\text{Ca}(\text{OH})_2$ with starch granules could explain the solubility behavior of the grain components as a function of lime concentration and nixtamalization temperature.

Texture of Tortillas

The CFT and CAT were influenced by the percentage of PGT used in the tortilla. The PGT ratio was highly significant for CFT ($P < 0.0069$) and CAT ($P < 0.0107$) in the linear terms. This was also the case for the quadratic term for CFT ($P < 0.0102$) and compression area of tortilla (CAT) ($P < 0.018$). The best explanatory model equation for CFT showing a relationship between the independent variables such as PGT (x_1), PGT viscosity (x_2), and endosperm viscosity (x_3) is

$$\begin{aligned} \text{CFT} = & 1599.72747 - 108.55382 x_1 - 7.370395 x_2 - 0.724805 x_3 \\ & + 5.439469 x_1^2 + 0.050977 x_2^2 + 0.000224 x_3^2 - 0.153242 x_1 x_2 \\ & - 0.004406 x_1 x_3 + 0.002101 x_2 x_3 \end{aligned} \quad (8)$$

This model was significant ($R^2 = 0.846$, $P = 0.011$).

The best explanatory model equation for CAT is

$$\begin{aligned} \text{CAT} = & 11221.99552 - 815.1111 x_1 - 55.91481 x_2 - 4.64988 x_3 \\ & + 37.90571 x_1^2 + 0.355548 x_2^2 + 0.00141 x_3^2 - 0.67613 x_1 x_2 \\ & - 0.00166 x_1 x_3 + 0.01331 x_2 x_3 \end{aligned} \quad (9)$$

The model for CAT was also significant ($R^2 = 0.84$, $P = 0.0103$).

The test for the CFT for tortillas with 5% PGT showed zones of maximum force (600 g force), when the initial viscosity of the pericarp had a value of zero or 100 cP and the endosperm fraction had a viscosity of 916 or 2,176 cP. Minimum values (500 g force) were found using fractions of PGT with an initial viscosity of 50 cP and endosperm viscosity of 1,300 cP (Fig. 8). The CFT value for tortillas prepared by the traditional nixtamalization method was 456.35 g force, a value located near the minimum point in the nixtamalization process evaluated in this study.

The CAT for tortillas prepared by selective nixtamalization using 5% PGT showed the same tendency as that observed for the CFT. The CAT value for tortillas prepared by the nixtamalized traditional method was 3,922 g mm, a value equivalent to the point of minimum viscosity (17 cP) for PGT fractions and with endosperm viscosity values >916 cP (Fig. 9). Similar values were reported by Martínez-Bustos et al (2001) for tortillas prepared by the traditional process of nixtamalization. They also reported that such tortillas were significantly different ($P < 0.05$) in this respect from tortillas prepared with exhaustively washed nixtamal with the germ removed. Flores-Farias et al (2000) found lower values for commercial nixtamalized maize flours than for traditional tortillas.

CONCLUSIONS

The continuous decorticating equipment showed an efficient separation of the grain components. The response variables were influenced by the lime concentration during the nixtamalization process. The response variables of water absorption index, water solubility index, initial viscosity, and viscosity at 90°C of nixtamalized PGT, and compression force and compression area of the tortilla indicated that the mathematical models fit the experimental data and that the variance of the models was highly significant. The interaction of Ca^{++} and $\text{Ca}(\text{OH})^+$ cations with starch hydroxyl sites modified the physical and chemical properties of the endosperm, using a lime concentration of 0.1%, it diminished endosperm solubility. Nevertheless, it increased the solubility and the WAI when both temperature and lime concentration were increased. On the other hand, this interaction caused increased endosperm viscosity at a lime concentration of 0.15%, and disruption of the starch granules

at a lime concentration of 0.3%, leading to a decrease in the viscosity at 90°C. Tortillas of good functional characteristics, similar to tortillas produced by the traditional process, were obtained by blending 5% nixtamalized PGT fractions and 95% nixtamalized endosperm. The blends were performed using a water to PGT ratio of 6:1 (w/w), a lime concentration of 0.8%, and a cooking time of 20 min for the PGT fractions, and 0.2% lime and 12 min cooking time at 80°C for the nixtamalized endosperm. The evaluated process shows several advantages over the traditional nixtamalization process such as decreased processing time and water consumption, the absence of polluting effluents, and the use of the whole grain. The preparation of nixtamalized fractions facilitates their control during processing and distribution.

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LITERATURE CITED

- American Association of Cereal Chemists. 2000. Approved Methods of the AACCI, 10th ed. The Association: St. Paul, MN.
- Anderson, R. A., Conway, H. F., Pfeifer, V. F., and Griffin, E. L., Jr. 1969. Gelatinization of corn grits by roll and extrusion-cooking. *Cereal Sci. Today* 14:4-12.
- Bedolla, S., and Rooney, L. W. 1982. Cooking maize for masa production. *Cereal Foods World* 27:219-221.
- Bryant, C. M., and Hamaker, B. R. 1997. Effect of lime gelatinization of corn flour and starch. *Cereal Chem.* 74:171-175.
- Diez de Sollano, C., and Berriozabal, J. M. 1955. Method of producing corn tortilla flour. US patent 2,704,257.
- Fernandez-Muñoz, J. L., Rodríguez, M. E., Pless, R. C., Martínez-Flores, H. E., Leal, M., Martínez, J. L., and Baños, L. 2002. Changes in nixtamalized corn flour dependent on postcooking steeping time. *Cereal Chem.* 79:162-166.
- Flores-Farias, R., Martínez-Bustos, F., Salinas-Moreno, Y., Chang, Y.-K., González Hernández, J., and Ríos, E. 2000. Physicochemical and rheological characteristics of commercial nixtamalized Mexican maize flours for tortillas. *J. Sci. Food Agric.* 80:657-664.
- Gargallo, C. J. 2000. La industria de la tortilla en México. Memorias de la 2a. expo-tortilla México. Retos y avances de la industria frente al nuevo milenio. Asociación de Industriales de la Tortilla: México.
- Gaytán, M. M., Martínez-Bustos, F., and Morales, S. E. 1995. Aplicación de un proceso de cocimiento dieléctrico de maíz integral en la elaboración de harinas instantáneas de maíz amarillo para preparación de frituras de masa y tortillas. CINVESTAV-IPN: Querétaro, México.
- Johnson, B. A., Rooney, L. W., and Khan, M. N. 1980. Tortilla making characteristics of micronized sorghum and corn flours. *J. Food Sci.* 46:671.
- Khan, M. N., Des Rosiers, M. C., Rooney, L. W., Morgan, R. G., and Sweat, V. E. 1982. Corn tortillas: Evaluation of corn cooking procedures. *Cereal Chem.* 59:279-284.
- Kite, F. E., Schoch, T. J., and Leach, H. W. 1957. Granule swelling and paste viscosity of thick boiling starches. *Baker's Dig.* 3:42.
- Lloyd, R. W., and Millares-Sotres, R. 1952. Method of making a tortilla flour. US patent 2,584,893.
- Martínez-Flores, H. E., Martínez-Bustos, F., Figueroa, C. J. D., and González-Hernández, J. 1998. Tortillas from extruded masa as related to corn genotype and milling process. *J. Food Sci.* 63:131-133.
- Martínez-Bustos, F., Figueroa, J. D. C., Sánchez-Sinencio, F., González-Hernández, J., Martínez, J. D. L., and Ruiz, M. T. 1996. Extrusion apparatus for the preparation of instant fresh corn dough or masa. US patent 5,558,886.
- Martínez-Bustos, F., Martínez-Flores, H. E., San Martín-Martínez, E., Sánchez-Sinencio, F., Chang, Y. K., Barrera-Arellano, D., and Ríos, E. 2001. Effect of the components of maize on the quality of masa and tortillas during the traditional nixtamalization process. *J. Sci. Food Agric.* 81:1-8.
- Martínez-Montes, J. L., Sánchez-Sinencio, F., Ruiz, M. T., and Martínez-Bustos, F. 2001. Selective nixtamalization process for the production of fresh whole corn masa, nixtamalized corn flour and derived products. US patent 6,265,013.
- Martínez, R., Mendoza, S., Reguera, E., Ortiz, P., and Martínez-Montes, J. L. 2001. Kinetic approach to nixtamalization of corn pericarp. *Cereal*

- Chem. 78:107-110.
- Mendoza, F. C. 1975. Method for obtaining nixtamalized flours. US patent 3,859,452.
- Mensah-Agyapong, J., and Horner, A. F. W. 1992. Nixtamalization of maize (*Zea mays* L.) using a single screw cook-extrusion process on lime-treated grits. *J. Sci. Food Agric.* 60:509-514.
- Molina, M. R., Letona, M., and Bressani, R. 1977. Drum drying technology for the improved production of instant tortilla flour. *J. Food Sci.* 42:1432-1434.
- Montemayor, E., and Rubio, M. 1983. Alkaline cooked corn flour: technology and uses in tortilla and snack products. (Abstr) *Cereal Foods World* 28:577.
- Montgomery, D. C. 1999. Capítulo 16: Métodos y diseños de superficie de respuesta. Pages 467-509, 589 in: *Diseño y análisis de experimentos*. Grupo Editorial Iberoamérica: México.
- Morad, M. M., Iskander, F. Y., Rooney, L. W., and Earp, D. F. 1986. Physico-chemical properties of alkali-cooked corn using traditional and presoaking procedures. *Cereal Chem.* 63:255-259.
- Nurul-Islam, M. M., and Mohd-Azemi, B. M. N. 1992. Effect of molar substitution (MS) on calcium binding by hydroxypropyl rice starches. *Starch* 44:332-334.
- Nurul-Islam, M. M., and Mohd-Azemi, B. M. N. 1994. Effect of pH and reaction time on calcium binding by hydroxypropyl rice starches. *Starch* 46:349-354.
- Oosten, B. J. 1982. Tentative hypothesis to explain how electrolytes affect the gelatinization temperature of starches in water. *Starch* 34:233-239.
- Paredes-López, O., and Saharopoulos, M. E. 1982. Scanning electron microscopy studies of limed corn kernels for tortilla making. *J. Food Technol.* 17:687-693.
- Pflugfelder, R. L., Rooney, L. W., and Waniska, D. R. 1988. Dry matter losses in commercial corn masa production. *Cereal Chem.* 65:127-132.
- Rendleman, J. A. 1966a. Complexes of alkali metals and alkaline-earth metals with carbohydrates. Pages 209-271 in: *Advances in Carbohydrate Chemistry*, Vol. 21. Academic Press: New York.
- Rendleman, J. A. 1966b. Alkali metal complexes of carbohydrates. I. Interaction of alkali metal salts with carbohydrates in alcoholic media. *J. Org. Chem.* 31:1839-1845.
- Rendleman, J. A. 1966c. Alkali metal complexes of carbohydrates II. Interaction of bases with carbohydrates in alcoholic media. *J. Org. Chem.* 31:1845-1851.
- Rendleman, J. A. 1978a. Metal-polysaccharide complexes. I. *Food Chem.* 3:47-79.
- Rendleman, J. A. 1978b. Metal-polysaccharide complexes. II. *Food Chem.* 3:127-162.
- Sahai, D., Mua, J. P., Surjewan, I., Buendia, M. O., Rowe, M., and Jackson, D. S. 2001. Alkaline processing (nixtamalization) of white Mexican corn hybrids for tortilla production: Significance of corn physicochemical characteristics and process conditions. *Cereal Chem.* 78:116-120.
- Saulnier, L., Mestres, C., Doublier, J. L., Roger, P., and Thibault, J. F. 1993. Studies of polysaccharides solubilized during alkaline cooking of maize kernels. *J. Cereal Sci.* 17:267:276.
- Sternner, M. L., and Zone, R. O. 1984. Method of grinding and cooking whole grain. US patent 4,463,002.
- Thomas, D. J., and Atwell, W. A. 1999. Starch modifications. Pages 31-48 in: *Starches*. Eagan Press: St. Paul, MN.
- Trejo-González, A., Feria-Morales, A., and Wild-Altamirano, C. 1982. The role of lime in the alkaline treatment of corn for tortilla production. Pages 245-263 in: *Modification of Protein: Food, Nutritional and Pharmacological Aspects*. *Advances in Chemistry, Series 198*. R. E. Feeney and J. R. Whitaker, eds. Am. Chem. Soc.: Washington, DC.
- Vaqueiro, M. C., and Reyes, P. 1986. Process for producing nixtamalized corn flour. México. US patent 4,594,260.
- Watson, S. A. 1987. Structure and composition. Pages 53-82 in: *Corn: Chemistry and Technology*. S. A. Watson and E. P. Ramstad, eds. Am. Assoc. Cereal Chem.: St. Paul, MN.

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