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NIXTAMALIZATION ASSISTED WITH ULTRASOUND: EFFECT ON MASS TRANSFER AND PHYSICOCHEMICAL PROPERTIES OF NIXTAMAL, MASA AND TORTILLA

NIXTAMALIZACIÓN ASISTIDA CON ULTRASONIDO: EFECTO EN LA TRANSFERENCIA DE MASA Y PROPIEDADES FISICOQUÍMICAS DE NIXTAMAL, MASA Y TORTILLA

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Abstract

The effects of ultrasound during maize nixtamalization at different temperatures on the properties of nixtamal, masa and tortilla were evaluated. White maize was nixtamalized at 85 and 95 °C, with and without ultrasound (843 $W{\cdot}m^{-2}$). Pericarp removal, the water and calcium absorption kinetics, and the apparent diffusion coefficients (D_A) from Fick's second law, the thermal and structural properties of the nixtamalized flours and the texture of the masa and tortillas were determined. The ultrasound and temperature affected significantly the water and calcium absorptions kinetics, showing linear and asymptotic tendencies during cooking and steeping, respectively. These were described adequately by Fick's model, and the ultrasound significantly increased the D_A of water (2.54-3.93×10^{-10} $m^2{\cdot}\ s^{-1}$). Over 80% of the pericarp was removed during cooking. The gelatinization enthalpy showed a decrease at 95 °C. Microscopy images showed spherical and polygonal granules at 85 °C, and a shape loss with increasing diameter by ultrasound effect (95 °C). Nixtamalization assisted by ultrasound reduced the processing time compared with traditional or industrial process.

Keywords: nixtamalization, ultrasound, calcium, texture, tortilla.

Resumen

Se evaluó el efecto del ultrasonido durante la nixtamalización a diferentes temperaturas en las propiedades de nixtamal, masa y tortilla. Se nixtamalizó maíz blanco a 85 y 95 ºC, con y sin ultrasonido (843 $W{\cdot}m^{-2}$). Se determinaron cinéticas de remoción de pericarpio, de absorción de agua y calcio, coeficientes de difusión aparente (D_A) a partir de la segunda ley de Fick, propiedades térmicas y estructurales de las harinas nixtamalizadas y textura de masa y tortillas. El ultrasonido y la temperatura afectaron significativamente a las cinéticas de absorción de agua y calcio, mostrando una tendencia lineal y asintótica durante la cocción y el reposo, respectivamente. Estas fueron descritas adecuadamente por el modelo de Fick, y el ultrasonido incrementó significativamente el D_A de agua (2.54-3.93×10^{-10} $m^2{\cdot}s^{-1}$). Más del 80% del pericarpio fue removido durante la cocción. La entalpía de gelatinización mostró una disminución a 95 ºC. Las imágenes de microscopía mostraron gránulos esféricos y poligonales a 85 ºC, y una pérdida de su forma con incrementos en el diámetro por efecto del ultrasonido (95 ºC). La nixtamalización asistida con ultrasonido redujo el tiempo de proceso comparado con un proceso tradicional o industrial.

Palabras clave: nixtamalización, ultrasonido, calcio, textura, tortilla.

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1 Introduction

Maize processing through nixtamalization has allowed the development of traditional and innovative products with high consumer acceptability. Nixtamalization is an ancient process through which maize (*Zea mays* L.) is cooked in ash-lime. Nowadays, it involves cooking maize in a $Ca(OH)_2$ solution, then steeping, washing and grinding, to obtain flour, masa, tortillas and other by-products (Martinez-Bustos *et al*., 2001). During nixtamalization, physical and chemical changes occur, as well as mass transfer, which are influenced by the process conditions such as the cooking temperature, agitation and concentration of the alkaline medium (Laria *et al*., 2005). The main changes that take place are the water and calcium absorption, brought about by the interaction of the alkali solution with the components in the maize. Calcium and water uptake are essential for the formation of amylose networks during nixtamalization (Alvarado *et al*., 1999; González *et al*., 2004; Laria *et al*., 2007). Calcium absorption is initially limited by the presence of pericarp. Once the pericarp has been removed, water and calcium diffusion increases to a level at which physical and chemical changes can occur; such changes are associated with the quality of the final products (González *et al*., 2005). During nixtamalization, it is desirable to predict the uptake of components as a function of time and temperature (Ruiz-Gutiérrez *et al*., 2012) to improve the process control. Previous studies have reported the moisture and calcium uptakes in maize (Charan and Prasad, 1996; Mondragón *et al*., 2004; Fernández-Muñoz *et al*., 2006; Laria *et al*., 2007; Ruiz-Gutiérrez *et al*., 2010; 2012). Mathematical models such as Fick's second law have also been used to predict water and calcium absorption (Lu *et al*., 1994; Rodríguez *et al*., 1995; Verma and Prasad, 1999) at different temperatures and solute concentrations (Ruiz-Gutiérrez *et al*., 2010; 2012). The relationship between the water and calcium absorption and pericarp removal and the changes in the starch during the cooking process were reported by Ruiz-Gutiérrez *et al*. (2010 and 2012), who showed that the starch gelatinization and thermal properties were affected by the temperature and cooking time. These properties are associated with the final characteristics and quality of the masa and tortilla, such as their consistency, flexibility, rollability, firmness, structural uniformity, color, and shelf life, as well as sensory characteristics such as flavor, aroma and texture (Suhendro *et al*., 1998; González *et al*., 2005; Estrada-Girón *et al*., 2014). Despite the benefits of nixtamalization, this process involves high energy and water consumption and leads to environmental pollution (Rosentrater *et al*., 2003). Alternative methods such as extrusion-cooking (Mensah-Agyapong and Horner, 1992; Sánchez-Madrigal *et al*., 2014), steam and pressure (Lloyd and Millares-Sotres, 1952; Bedolla and Rooney, 1982), infrared cooking (Johnson *et al*., 1980), and dielectric heating for cooking (Gaytan-Martínez *et al*., 2000) have been used. Other alternative technologies such as ultrasound are being investigated and are proposed to overcome the issues of long processing time and high-energy consumption (Chemat *et al*., 2011). In the ultrasound-assisted extraction, high-intensity sound waves are propagated through the liquid medium, causing the oscillation of its molecules and generating voids or cavities in a phenomenon known as cavitation; this creates alternating cycles of compression and rarefaction that cause pressure changes. During cavitation, the bubbles collapse in succeeding compression cycles, generating energy for chemical and mechanical effects (Mason *et al*., 2003; O'Donnell *et al*., 2010). This mechanical effect in raw plant tissue causes disruption in the cell walls, facilitating both solvent penetration into the cell and the release of biocomponents into the continuous phase (Toma *et al*., 2001; Lingyun *et al*., 2006; Ebringerová and Hromádková, 2010; Quiroz-Reyes *et al*., 2013; Soto-León *et al*., 2014). Ultrasound has been explored previously in maize processing. A recent study by Janve *et al*. (2013) showed that power ultrasound-assisted nixtamalization resulted in a reduced process time and softer nixtamal with lower solid losses in nejayote (cooking liquor). However, the effect of ultrasound on the physical and chemical changes during nixtamalization and its impact on the qualities of masa and tortilla has not been presented. The objective of this study was to evaluate the effect of ultrasound during maize nixtamalization at different temperatures on the physical and chemical properties of grain, masa and tortillas.

2 Materials and methods

2.1 Materials

White dent maize (*Zea mays* L.) was used in this study. Moisture, total protein, total fat, fiber, ash,

and calcium contents of the maize were analyzed according to AOAC (1998) methods 950.02, 960.52, 920.39, 962.09, 923.03, and 968.08, respectively. In addition, the pericarp content and dimensions of the grain were determined according to a method reported by Ruiz-Gutiérrez *et al*. (2010). For the nixtamalization process, solutions of food-grade calcium hydroxide [$Ca(OH)_2$, 98%] were used.

2.2 *Nixtamalization process assisted with ultrasound*

Maize (lots of 4 kg) was nixtamalized in a $Ca(OH)_2$ [1.2% (w/v)] solution in a maize/solution ratio of 1:5. The nixtamalization was performed at 85 and 95 °C with and without ultrasound for 60 min of cooking and 8 h of steeping. The alkalinity of the system during the process was monitored by measuring the pH of the nejayote, keeping it at 12.6 ± 0.1. The lots of maize were placed on stainless steel grid (49.53×10^{-2} m l (length) × 99.822×10^{-3} m w (width) × 72.898×10^{-3} m h (height)) and immersed in 20 L of calcium hydroxide solution (1.2%) in a stainless steel ultrasonic tank (60.96×10^{-2} m (l) × 30.48×10^{-2} m (w) × 40.64×10^{-2} m (h), volume 60.3 L) insulated with fiberglass of thickness 6.35×10^{-2} m. It was equipped with two electrical resistors with a temperature control, and an immersible transducer with a radiant surface of 45.72×10^{-2} m (l) by 15.24×10^{-2} m (w) (Branson EB618-25-12). This was adapted to the bottom of the tank and connected to an ultrasonic generator (Branson S8525-12-500W) of variable power from 125 to 500 W. The operating ultrasound frequency was 25 kHz with an ultrasound intensity (UI) of 843 $W{\cdot}m^{-2}$ using continuous pulsation. The energy input was controlled by setting the power modulation of the transducer. The ultrasound intensity was determined using the calorimetric method described below. During nixtamalization with and without ultrasound, nixtamal samples were taken every 15 min until 60 min of cooking time had elapsed. After the cooking process, the grains were steeped in nejayote and sampled every 2 h for 8 h. The kinetics for the pericarp removal and water and calcium absorption were obtained. From the data for water and calcium absorption, the apparent diffusion coefficients were also determined using Fick's second law. Thermal properties such as the temperatures and enthalpies of gelatinization were determined for the nixtamalized dried grain samples. All treatments were performed in duplicate.

2.3 *Nixtamalization assisted with ultrasound for masa and tortilla production*

Maize (lots of 8 kg) was nixtamalized at 85 and 95 °C with and without ultrasound for 60 min of cooking with different steeping times until the selected moisture content (44 g water·100 g^{-1}) was achieved through each treatment, according to the kinetics of water absorption obtained previously. The steeping applied in each treatment was 60 min without ultrasound or 30 min with ultrasound at 85 °C, and 30 min without ultrasound or no steeping with ultrasound at 95 °C. Subsequently, the nixtamal was milled with an industrial stone grinder for masa preparation. A sample of milled nixtamal was used for the determination of thermal and microscopic properties, while the other part was used for masa and tortilla preparation. The milled nixtamal with moisture content of 44 g water·100 g^{-1} was mixed with water until 58% moisture content was achieved for masa production, and the adhesiveness, cohesiveness and color were determined. The masa was shaped using a continuous industrial machine (Celorio, México) obtaining flat disks of 125 mm in diameter and 1 mm thickness. The tortillas were cooked on the continuous band during 30 s at 340 °C. Then, the tortillas with 44% moisture content were stacked, packed in paper and stored in a thermal container. Finally, 30 min later the rollability, extensibility and color were determined.

2.4 *Analytical and physical investigations of nixtamal*

The percentage of removed pericarp, moisture, and calcium content during nixtamalization were measured. The water and pericarp content were determined using gravimetric method 950.02 described by the AOAC (1998) and Ruiz-Gutiérrez *et al*. (2010), respectively. The calcium content was determined through flame atomic absorption spectrophotometry (FAAS) using a spectrophotometer (Perkin-Elmer model AA800, Überlingen, Germany) at 422.7 nm with an air-acetylene flame, as described in method 968.08 of the AOAC (1998). These analyses were carried out in triplicate, and the results are expressed in g·100 g^{-1} and for calcium in mg $Ca^{2+}\cdot 100\ g^{-1}$.

2.5 *Ultrasonic intensity calculation*

The ultrasound intensity was calculated using the calorimetric method described by Rawson *et al.* (2011). Briefly, the calcium hydroxide solution temperature as a function of time was recorded under adiabatic conditions using a thermocouple incorporated into the tank, increasing the temperature from 25 to 45.5 °C. From the temperature versus time data collected, the initial temperature increase dT/dt was determined by polynomial curve fitting. The ultrasound power was determined according to Eq. (1):

$$UP = m \cdot C_p \left(\frac{dT}{dt}\right)_{t=0} \quad (1)$$

where (dT/dt) is the change in temperature over time ($°C \cdot s^{-1}$), C_p is the specific heat of cooking medium ($4.15\ kJ \cdot kg^{-1} \cdot °C^{-1}$), calculated according to Hatton *et al.* (1959) and Bonner and Cerutti (1976), and m is the mass (kg). Finally, the ultrasound intensity was calculated using Eq. (2):

$$UI = \frac{UP}{A} \quad (2)$$

where UP is the ultrasound power (W) and A is the area of tank (m^2).

2.6 *Water and calcium apparent diffusion coefficients*

For the computation of the apparent diffusion coefficients of water and calcium (D_A), the corn grain was assumed to have finite laminar geometry. In the solution to the non-stationary Fick's second law equation for constant diffusivity, initial uniform calcium and water concentrations, negligible diffusion in the fluid surrounding the grain, and constant grain/water ratio over time were assumed. The solutions for water and calcium diffusion are:

$$E_w = \frac{C - C_\infty}{C_0 - C_\infty} = \frac{8}{n^2} \sum_{n=0}^{\infty} \frac{\exp\left[(2n+1)^2 \left(\frac{\pi}{2}\right)^2 \frac{D_A t}{a^2}\right]}{(2n+1)^2} \quad (3)$$

$$E_{Ca^{2+}} = \frac{C - C_\infty}{C_0 - C_\infty} = \frac{8}{n^2} \sum_{n=0}^{\infty} \frac{\exp\left[(2n+1)^2 \left(\frac{\pi}{2}\right)^2 \frac{D_B t}{a^2}\right]}{(2n+1)^2} \quad (4)$$

where E_w, $E_{Ca^{2+}}$ are the water and calcium fraction absorbed, C is the average concentration at time t, C_0 is the initial concentration, C_∞ is the equilibrium concentration, $D_{A,B}$ are the apparent diffusion coefficients ($m^2 \cdot s^{-1}$) for both solutes respectively and a is the thickness of the grain.

The applied estimation procedure considered $n = 2$ in Eq. 3 and 4 to achieve very good convergence and to minimize the error. These nonlinear regressions were performed with a program built in MAPLE (v 16), which runs the optimization package NLPSolve to compute a minimum corresponding to the best fit between experimental and theoretical data. Since a nonlinear regression was performed, the standard error of estimated values (SEE) was calculated to provide some insight into the fitting among all cases with Eq. (5):

$$SEE = \sqrt{\frac{\sum_{i=1}^{n} (y_{exp-i} - y_{est-i})^2}{n}} \quad (5)$$

where y_{exp} and y_{est} indicate experimental and theoretical data, respectively.

2.7 *Thermal properties*

Before taking differential scanning calorimetry (DSC) measurements, the nixtamal samples were dehydrated at 50 °C in a vacuum drying chamber for 24 h, and then milled and sieved to obtain a particle size of 0.149-0.123 mm. Briefly, two milligrams of preconditioned nixtamal was placed in a pan with 20 μL of water and hermetically sealed; an empty pan was used as a reference. Both were placed into a TA Instruments (Q-200, Crawley, England) calorimeter. All scans were performed at heating rate of 5 ºC/min. The onset or initial temperature (T_0), peak or gelatinization temperature (T_p), final temperature of starch gelatinization (Te), and the gelatinization enthalpy (ΔHg) were obtained using the method described by Estrada-Girón *et al.* (2014).

2.8 *Texture of masa and tortillas*

The adhesiveness and cohesiveness of masa, maximum rollability force (MRF) and maximum extensibility force (MEF) of tortillas were measured using a texturometer (TA.XT2, Texture Analyzer plus, UK). Texture profile analysis (TPA) was performed to evaluate the adhesiveness and cohesiveness of masa according to the method reported by Ruiz-Gutiérrez *et al.* (2012). Briefly, cubes of masa measuring 2 × 2 × 2 cm were compressed 90% at a velocity of 300 mm/min with a 5 cm-diameter disc. Two seconds later, they were compressed again. This was performed on ten cubes from each treatment and an average value was reported. The MRF was determined according to the method described by Suhendro *et al.* (1998). This test involved rolling the tortilla on an acrylic

platform with a rotary cylinder, and was performed in a tension mode at 3.0 mm/s until a distance of 100 mm was reached. Ten tortillas from each treatment were evaluated and an average value was reported. The MEF was evaluated according to the method described by Cortés-Gómez *et al*. (2005), using an HDP/TPB accessory, in which the tortilla was placed between two rings and fixed with screws. Each tortilla was compressed with a 2.5 cm-diameter ball probe at 2 mm/s, until a distance of 25 mm was reached. Fifteen tortillas from each treatment were evaluated and an average value was reported.

2.9 Color of masa and tortillas

The color of masa and tortillas was measured by tristimulus colorimetry using a Konica Minolta CR-400/410 colorimeter (Minolta Co., Osaka, Japan), which was calibrated using a white tile as a blank. The L^* (luminosity), a^* (green to red) and b^* (blue to yellow) parameters were determined from five readings for five tortillas from each treatment and ten readings for masa from each treatment.

2.10 Scanning electron microscopy

Nixtamal samples dehydrated, milled and sieved at particle sizes <0.15 mm and a moisture content of 1% were stuck to stubs with two-sided adhesive tape and coated with a gold layer in a high vacuum using a Denton vacuum evaporator (Desk II) set to a pressure of 7.031×10^{-2} kg·cm^{-2}. The samples were examined with a scanning electron microscope (JSM-5800LV, JEOL, Akishima, Japan) equipped with a secondary electron detector at an acceleration rate of 10 kV.

2.11 Statistical analysis

The data obtained from the analysis of the removed pericarp, moisture and calcium absorption, thermal properties of nixtamal, texture and color in masa and tortillas were subjected to a variance analysis using Minitab version 16 software (Minitab, 2010, PA, USA). Additional water and calcium kinetics data were used to calculate the apparent diffusion coefficients by nonlinear regressions. Differences between the means were evaluated using Tukey's test ($P < 0.05$).

3 Results and discussion

3.1 Physicochemical characterization of maize

The white maize had moisture, total protein, total fat, fiber, ash and carbohydrate content of 10.87 ± 0.17, 9.52 ± 0.49, 3.38 ± 0.10, 1.58 ± 0.13, 1.24 ± 0.02 and 73.4 ± 0.87 g·100 g^{-1}, respectively. The calcium content and pericarp content were 7.67 ± 0.58 mg Ca^{2+}· 100 g^{-1} and 3.91 ± 0.07 g·100 g^{-1}, respectively. The white maize measured 12.07 ± 1.11 mm in length, 9.06 ± 0.83 mm in width, and 4.30 ± 0.66 mm in thickness. These results are in agreement with the reported by Ruiz-Gutierrez *et al*. (2010).

3.2 Pericarp removal kinetics

According to analysis of variance (Table 1), the cooking temperature and ultrasound had a significant effect ($P < 0.05$) on the removal of pericarp. The pericarp removal during nixtamalization (Fig. 1) increased with cooking time at both temperatures, with and without ultrasound. A linear tendency was observed at the beginning of cooking, while asymptotic behavior was detected after cooking. High amounts of pericarp were removed in short cooking times. Nixtamalization at 85 °C with ultrasound treatment showed a marked effect on pericarp removal and a reduction in cooking time compared to nixtamalization without ultrasound at the same temperature. To attain over 80% pericarp removal, the cooking times required were 45 and 60 min for nixtamalization with and without ultrasound, respectively (Fig. 1a, b). On the other hand, nixtamalization at 95 °C did not show marked differences (Fig. 1c, d) with or without ultrasound, and over 80% pericarp removal was achieved with a cooking time of 30 min for both treatments. These results are due to hydrolysis and softening of the grain cellulose in the alkaline solution (González *et al*., 2004; 2005; Laria *et al*., 2005), which are favored at high temperatures, with a null effect of ultrasound at these conditions. This is due possibly at the counteracting effect of the temperature over the sonication, which minimizes the cavitation phenomenon at high temperatures (Pardo-Rueda *et al*. 2015; Mason, 2000). Other reports show that hydrolysis and softening of the cellulose are accelerated by increasing temperatures (Ruiz-Gutiérrez *et al*., 2010) and sonication at room temperature causing the softening and removal of the

Table 1. Analysis of variance of the kinetics of pericarp removal, water and calcium absorption

Source	DF	Pericarp removal	Water absorption	Calcium absorption
Temperature (T)	1	0.000	0.000	0.000
Ultrasound (U)	1	0.008	0.000	0.002
Time (t)	8	0.000	0.000	0.000
T*U	1	0.000	0.917	0.879
T*t	8	0.000	0.000	0.001
U*t	8	0.003	0.000	0.036
T*U*t	8	0.000	0.977	0.655

Significant at $P < 0.05$.

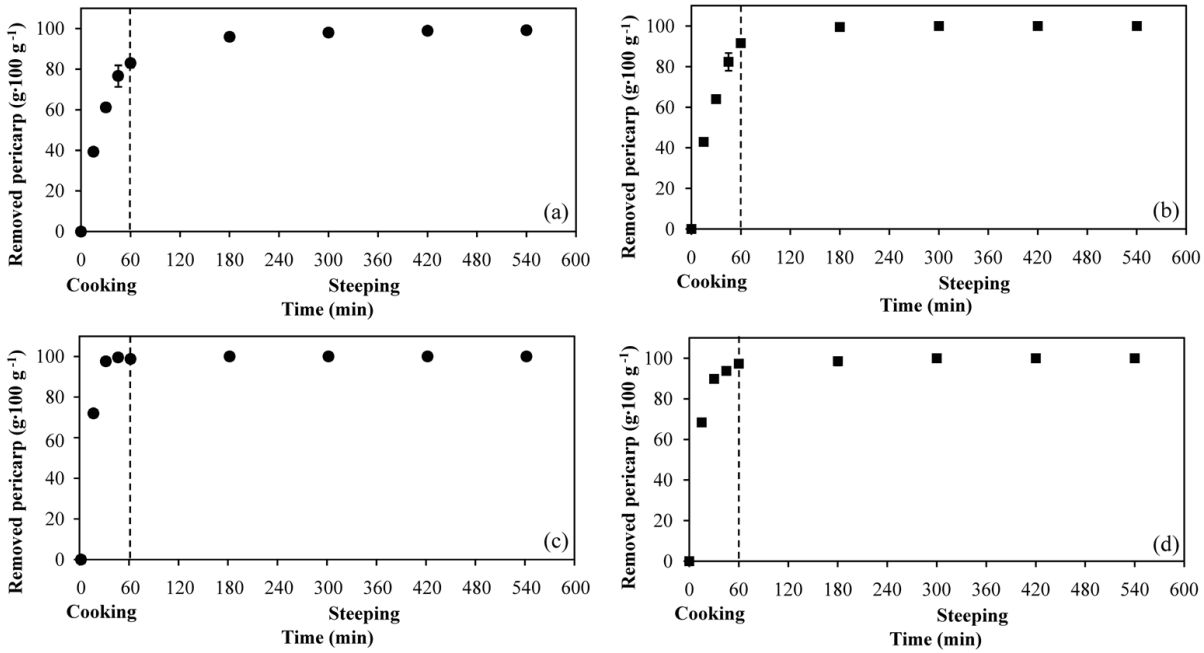

Fig. 1. Removed pericarp at different nixtamalization temperatures with ■ and without • ultrasound. (a) 85 °C. (b) 85 °C. (c) 95 °C. (d) 95 °C.

pericarp corn (Yang *et al*., 2002; Wang *et al*., 2006) which, in turn, promotes the absorption of water and calcium into the grain.

3.3 Water absorption kinetics

The analysis of variance (Table 1) showed that both the nixtamalization temperature and ultrasound significantly influenced ($P < 0.05$) water absorption kinetics. Nixtamal water absorption increased with temperature and cooking time in both processes, with and without ultrasound (Fig. 2). During nixtamalization assisted by ultrasound, water absorption exhibited linear behavior during cooking and steeping at both temperatures.

Also, in the nixtamalization without ultrasound, nixtamal water absorption displayed exponential behavior during the steeping stage, and after 300 min, an asymptotic tendency was observed (Fig. 2a, c). Nixtamalization assisted with ultrasound yield the highest water absorption values in the nixtamal with moisture contents of 63 g water·100 g^{-1} of nixtamal at 85 °C and 70 g water·100 g^{-1} of nixtamal at 95 °C. The quick water absorption into the maize grain during cooking has been attributed to the water diffusion into the grain through its tip cap, because this is a weak physical barrier to the diffusion of water facilitating hydration (Fernández-Muñoz *et al*., 2004; González *et*

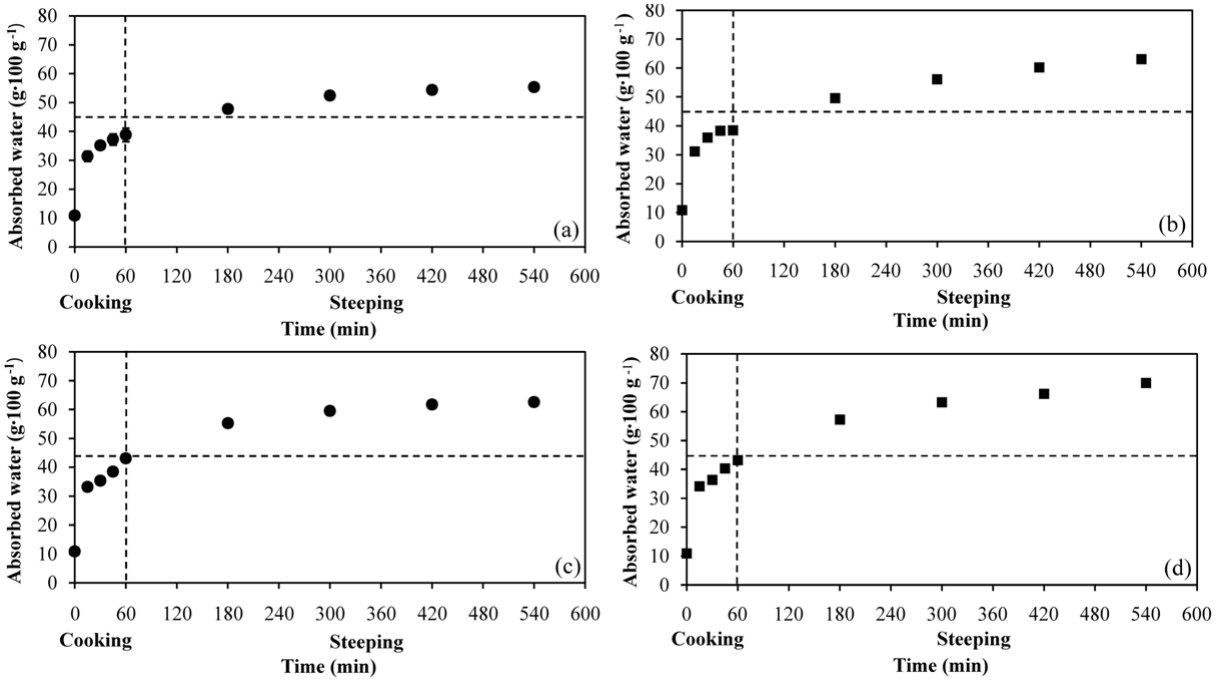

Fig. 2. Absorbed water at different nixtamalization temperatures with ■ and without • ultrasound. (a) 85 °C. (b) 85 °C. (c) 95 °C. (d) 95 °C.

al., 2004; Laria *et al*., 2005). In addition, the removal of large amounts of pericarp (over 80% in the first 30 min) facilitated this quick hydration during cooking, which decreased gradually allowing the equilibrium during steeping (Ruiz-Gutiérrez *et al*., 2010; 2012). The water absorption values obtained during cooking and steeping assisted with ultrasound suggest that ultrasound causes more damage to the structure of the grain (Yang *et al*., 2002; Wang *et al*., 2006), increasing the removal of pericarp and the water absorption into the grain. This has been attributed to intense shear forces, which cause the fragmentation of particles through the cavitation process (Janve *et al*., 2013), increasing the absorption of water into the maize grain.

3.4 Calcium absorption kinetics

Table 1 shows that calcium absorption into the nixtamal was significantly affected ($P < 0.05$) by the linear effect of ultrasound and temperature, but significant interaction of both variables was not observed. Grain calcium absorption showed variations in the absorption tendencies throughout the process (Fig. 3). The Ca^{2+} absorption increased during the first 15 min, but decreased afterwards. The absorption increased again after cooking and during the steeping stage under any experimental conditions. The obtained maximum concentrations of calcium in the nixtamalized grains correspond with the ultrasound treatments, with calcium contents of 297.80 and 460.07 mg $Ca^{2+}\cdot$ 100 g^{-1} of nixtamal at 85 °C without and with ultrasound, respectively, and 445.32 and 508.69 mg $Ca^{2+}\cdot$ 100 g^{-1} of nixtamal at 95 °C without and with ultrasound, respectively. The observed behavior of calcium absorption (Fig. 3) during cooking (decreasing at the beginning of cooking) can be explained as follows. Calcium ions are retained in the pericarp primarily by the acidic groups of hemicelluloses (mainly uronic acid). However, during the alkaline cooking, the pericarp is progressively degraded and loses the ability to retain Ca^{2+} because the hemicellulose fraction passes to the cooking liquor, decreasing the amount of calcium initially retained (González *et al*., 2005). During the steeping (Fig. 3), the calcium absorption increased after pericarp elimination without reaching the equilibrium concentration, which is in agreement with previous results reporting equilibrium after 960 min (Ruiz-Gutiérrez *et al*., 2010; 2012).

3.5 Water and calcium apparent diffusion coefficients

The apparent diffusion coefficients obtained from Fick's second law for water and calcium absorption

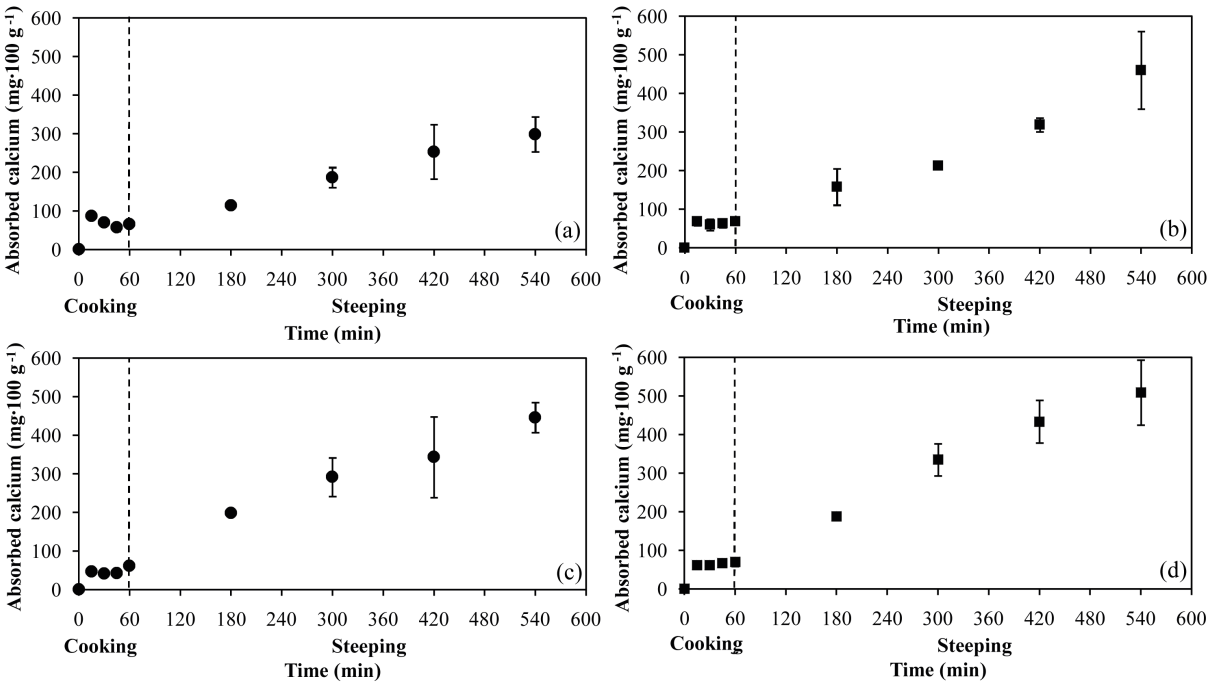

Fig. 3. Absorbed calcium at different nixtamalization temperatures with ■ and without • ultrasound. (a) 85 °C. (b) 85 °C. (c) 95 °C. (d) 95 °C.

Table 2. Water and calcium diffusion coefficients* in nixtamalized maize at different cooking temperatures with and without ultrasound

CT	UI	Water		Calcium	
(°C)	($W \cdot m^{-2}$)	D_A ($m^2 \cdot s^{-1}$) $\times 10^{-10}$	SEE	D_A ($m^2 \cdot s^{-1}$) $\times 10^{-10}$	SEE
85	0	2.45 ± 0.12^b	5.51	1.37 ± 0.70^a	3.57
85	843	3.61 ± 0.17^a	3.82	1.69 ± 0.19^a	7.13
95	0	2.45 ± 0.07^b	3.56	2.26 ± 0.60^a	6.77
95	843	3.93 ± 0.07^a	4.29	3.02 ± 0.73^a	9.02

*Mean ± standard deviation. Column means with different superscript shows significant differences, Tukey's test ($P < 0.05$). CT, cooking temperature; UI, ultrasound intensity; SEE, standard error of estimated values.

at different nixtamalization temperatures with and without ultrasound are shown in Table 2. The water diffusion coefficients showed significant differences ($P < 0.05$) between treatments with and without ultrasound at both temperatures; the highest water diffusion coefficients were found for nixtamalization assisted with ultrasound at both temperatures. As mentioned above, the transport phenomenon of water and Ca^{2+} through the grain boundary is adequately represented by a Fickian model. A variation in the data of 95-96% supports this assumption. Very interesting facts can be noted from Table 2. First, the magnitudes of D_A, for any experimental condition, are always higher for water than for Ca^{2+}. This is physically explained if we consider that both the ultrasound effect and high temperatures accelerate the transport mechanism of water. Thus, a suitable combination of ultrasound and high temperature during nixtamalization degrades the pericarp so that the diffusion processes are increased (Yang *et al.*, 2002; Wang *et al.*, 2006). The calcium diffusion coefficients did not show significant differences under any experimental conditions. The water diffusion coefficients were between 2.54 and 3.93×10^{-10} $m^2 \cdot s^{-1}$, while the calcium diffusion coefficients values were between 1.38 and 2.90×10^{-10} $m^2 \cdot s^{-1}$.

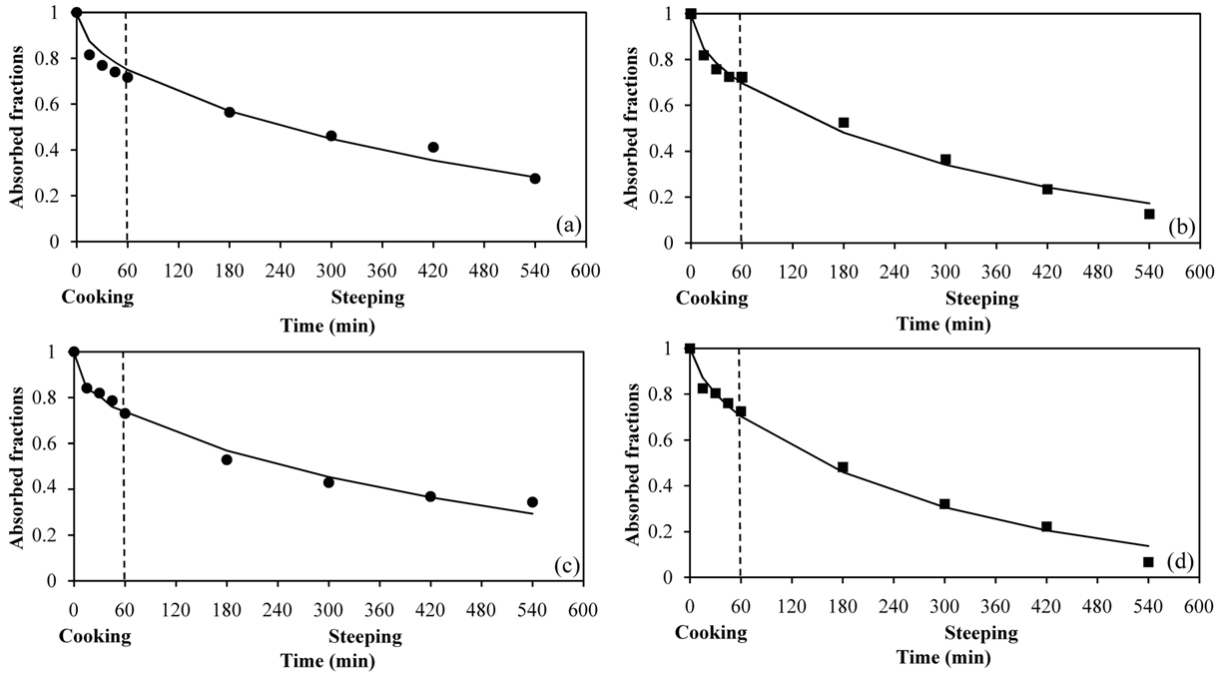

Fig. 4. Absorbed fractions of water at different nixtamalization temperatures with and without ultrasound. (a) 85 °C. (b) 85 °C. (c) 95 °C. (d) 95 °C. — predicted values and experimental values with ■ and without • ultrasound.

Table 3. Temperatures and enthalpy of gelatinization* of nixtamalized maize at different cooking temperatures with and without ultrasound

CT (°C)	UI (W·m^{-2})	T_o (°C)	T_p (°C)	T_e (°C)	ΔHg (J/g)
85	0	70.77 ± 0.70^a	76.488 ± 0.98^a	83.48 ± 3.59^a	4.5018 ± 0.81^{ab}
85	843	70.19 ± 1.43^a	75.943 ± 1.62^a	81.90 ± 1.21^a	4.9615 ± 0.77^a
95	0	71.01 ± 1.93^a	76.428 ± 1.55^a	82.02 ± 1.35^a	3.2028 ± 0.81^b
95	843	71.95 ± 1.40^a	76.520 ± 0.28^a	82.11 ± 0.43^a	3.8890 ± 0.60^{ab}

*Means ± standard deviation. Column means with different superscript shows significant differences, Tukey's test ($P < 0.05$). CT, cooking temperature; UI, ultrasound intensity; T_o, onset gelatinization temperature; T_p, peak gelatinization temperature; T_e, end gelatinization temperature; ΔHg, gelatinization enthalpy.

Calcium diffusion coefficient values in this study are lower than those where calcium is diffused in water (0.792×10^{-9} m^2·s^{-1}) at 25 °C (Lide, 2005). The difference between diffusion coefficients could be attributed to related factors such as molecular size and to different diffusion mechanisms presented by both solutes (water and calcium ion). Quintero-Ramos *et al*. (2003) described different mechanisms for calcium diffusion into vegetable tissue. The first one is related to the mass transfer resistance into the tissue, and the other is attributed to some reactions occurring during the diffusion through a solid matrix, which causes calcium interactions with functional groups of components contained in the solid providing mass transfer resistance. These mechanisms probably lead to the insignificance in the calcium diffusion under the different experimental conditions, even though the experimental data seems to support the phenomenon. As explained above the ability of components of pericarp to retain calcium at the beginning of cooking (González *et al*., 2004; 2005) could be the causes of decreasing of the absorption rate during calcium diffusion, fitting in lesser extent to predicted values. The water diffusion coefficients obtained in this study for treatments without ultrasound were similar to those reported by Ruiz-Gutiérrez *et al*. (2010; 2012).

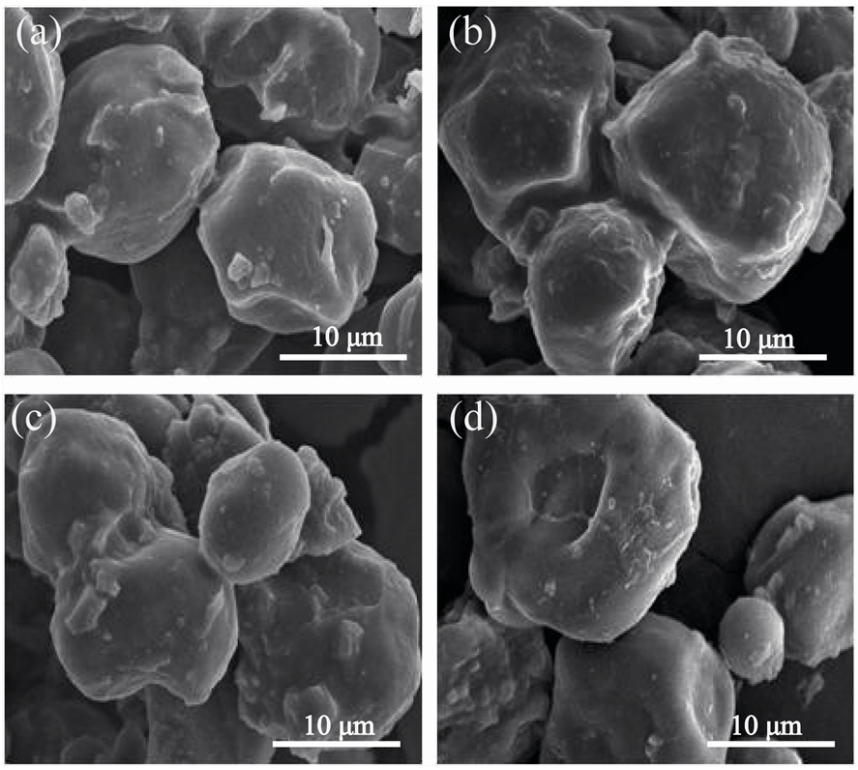

Fig. 5. Micrographs of nixtamalized maize at different temperatures with and without ultrasound. (a) 85 °C without ultrasound. (b) 85 °C with ultrasound. (c) 95 °C without ultrasound. (d) 95 °C with ultrasound, 4000×.

3.6 Thermal properties

The main results of thermal analysis of the nixtamalized maize flours are shown in Table 3. The starch gelatinization temperatures (T_o, T_p and T_e) and gelatinization enthalpy (ΔHg) did not show significant differences ($P > 0.05$) between treatments, except for the ΔHg values of the treatments at 85 °C with ultrasound and at 95 °C without ultrasound ($P < 0.05$). It was also observed that the gelatinization enthalpies for the treatments at 95 °C were lower in comparison with treatments at 85 °C; these low gelatinization enthalpy values indicate that there is a large amount of gelatinized starch, so that less energy was required to gelatinize the rest of the starch (Mondragón *et al*., 2004) owing to the higher process temperature causing greater starch damage. The gelatinization temperatures and enthalpy were similar to those reported by Mondragón *et al*. (2004) and Ruiz-Gutiérrez *et al*. (2010; 2012).

3.7 Scanning electron microscopy

The scanning electron micrographs (Fig. 5) revealed structural changes in the starch granules with increasing nixtamalization temperature, but no clear changes were observed for the treatments assisted with ultrasound. The micrographs showed minimal damage in the starch granules; nixtamalized grains at 85 °C without ultrasound showed spherical and polygonal shapes of the starch with increased volume (Fig. 5a) due to structural changes in the molecule, while those at 85 °C with ultrasound showed an increased volume of the starch (Fig. 5b). Nixtamalization at 95 °C without ultrasound led to a loss of shape of the starch granule (Figure 5c). An increase in diameter and agglomeration of the starch granules resulted at 95 °C with ultrasound. This agrees with the results reported by Huijbrechts *et al*. (2008), who described the morphology of the corn starch granules as cubic and spherical shapes with soft surfaces and few pores. The process of granule swelling during gelatinization leads to a loss of shape and agglomeration of the granules (Atkin, 1998). Robles *et al*. (1988) studied the changes in maize starch during the alkaline nixtamalization treatment, and found that during the preparation of nixtamal, starch gelatinization is not excessive. This could be because of the stabilization of the starch granules by calcium ion interactions.

Table 4. Masa and tortilla texture properties* of nixtamalized maize at different cooking temperatures with and without ultrasound

CT (°C)	UI ($W \cdot m^{-2}$)	Masa		Tortillas				
		TPA		Entensibility			Rollability	
		Adhesiveness (N)	Cohesiveness	MEF (N)	Distance (mm)	Work (N·mm)	MRF (N)	Work (N·mm)
85	0	-1.73 ± 0.36^{a}	0.122 ± 0.01^{a}	3.26 ± 0.61^{ab}	11.08 ± 1.16^{a}	11.33 ± 0.98^{a}	0.212 ± 0.02^{b}	3.58 ± 0.84^{a}
85	843	-1.81 ± 0.36^{a}	0.123 ± 0.01^{a}	3.59 ± 0.51^{ab}	11.53 ± 1.45^{a}	12.53 ± 1.09^{a}	0.258 ± 0.01^{a}	4.38 ± 0.58^{a}
95	0	-1.01 ± 0.32^{b}	0.110 ± 0.01^{a}	3.06 ± 0.52^{b}	11.08 ± 0.55^{a}	12.52 ± 1.82^{a}	0.251 ± 0.04^{a}	3.67 ± 0.96^{a}
95	843	-1.08 ± 0.32^{b}	0.116 ± 0.01^{a}	3.64 ± 0.94^{a}	11.61 ± 1.76^{a}	12.67 ± 3.13^{a}	0.260 ± 0.01^{a}	3.72 ± 0.56^{a}

*Means ± standard deviation. Column with different superscript shows significant differences, Tukey's test ($P < 0.05$). CT, cooking temperature; UI, ultrasound intensity; TPA, texture profile analysis; MEF, maximum extensibility force; MRF, maximum rollability force.

Table 5. Masa and tortilla color* of nixtamalized maize at different cooking temperatures with and without ultrasound

CT (°C)	UI ($W \cdot m^{-2}$)	Color		
		L*	a*	b*
Masa				
85	0	83.52 ± 1.47^{b}	-1.04 ± 0.19^{bc}	15.24 ± 0.89^{b}
85	843	83.38 ± 0.96^{b}	-1.06 ± 0.09^{c}	14.44 ± 0.39^{c}
95	0	81.27 ± 0.95^{c}	-0.73 ± 0.08^{a}	15.39 ± 0.20^{b}
95	843	84.70 ± 1.23^{a}	-0.96 ± 0.09^{b}	16.66 ± 1.25^{a}
Tortillas				
85	0	74.39 ± 2.36^{a}	-1.50 ± 0.19^{b}	15.39 ± 0.89^{a}
85	843	73.12 ± 1.28^{a}	-1.68 ± 0.13^{c}	15.68 ± 0.48^{a}
95	0	73.93 ± 0.73^{a}	-1.36 ± 0.15^{a}	15.38 ± 0.44^{a}
95	843	73.62 ± 2.51^{a}	-1.44 ± 0.24^{ab}	15.16 ± 2.54^{a}

*Means ± standard deviation of 10 replicates. Column with different superscript shows significant differences, Tukey's test ($P < 0.05$). CT, cooking temperature; UI, ultrasound intensity.

3.8 Texture of masa and tortillas

The textural properties in masa and tortillas are attributed to physical and chemical phenomena that occur in starch during nixtamalization and subsequent steps for the production of flour or masa. It is important to know these textural properties because a masa with high or low cohesiveness and adhesiveness can lead to problems during tortilla manufacturing (Arámbula-Villa *et al*., 2001; Ruiz-Gutiérrez *et al*., 2012). Masa from nixtamal at 95 °C without ultrasound is shown in Table 4, and had low values of cohesiveness and adhesiveness, without significant differences in cohesiveness ($P < 0.05$) between treatments. The treatments at 85 °C with and without ultrasound had the highest adhesiveness values with significant differences ($P < 0.05$) from the treatments at 95 °C with and without ultrasound. The values of adhesiveness and cohesiveness in this study are lower than those found by Ruiz-Gutiérrez *et al*. (2012). It is important to note that the treatments leading to lower adhesiveness (at 95 °C) had lower enthalpy values (Table 3), and the high amount of gelatinized starch during the nixtamalization may lead to low adhesiveness values.

Tortillas made with ultrasound treatment exhibited greater MEF values and distance to rupture. Despite this, no significant differences ($P > 0.05$) were observed between treatments with and without ultrasound (Table 4). Tortillas with lower MEF values were obtained at 95 °C without ultrasound; this may also be due to the greater amount of gelatinized starch during the nixtamalization with this treatment (Table 3). The extensibility values were similar to those reported by Cortés-Gómez *et al*. (2005) for blue maize tortillas obtained by traditional

nixtamalization, and lower than those reported by Ruiz-Gutiérrez *et al*. (2012) for tortillas nixtamalized in a two-step nixtamalization process with different calcium salts. Tortillas obtained from nixtamalization assisted with ultrasound had higher MRF values, and required more force and work to be rolled (Table 4); only the treatment at 85 °C without ultrasound was significantly different ($P < 0.05$). The treatments at 95 °C showed no differences, probably owing to the diminished effect of ultrasound treatment at high temperatures. The MRF results obtained were similar to those reported by other authors (Suhendro *et al*., 1998; Ruiz-Gutiérrez *et al*., 2012).

3.9 Color of masa and tortillas

The color parameters of masa and tortillas are shown in Table 5. Masa with the highest luminosity (L^*) value was obtained at 95 °C with ultrasound, while that with the lowest luminosity value was obtained at 95 °C without ultrasound; the treatments were significantly different, and showed significant differences ($P < 0.05$) from the masas obtained at 85 °C with and without ultrasound (Table 5). On the other hand, the color descriptor b^* (blue-yellow tendency) values showed a lower yellow color in masa elaborated at 85 °C with ultrasound. The masa elaborated at 95 °C with ultrasound shows an intense yellow color. The results showed that the tortillas obtained from all treatments were not significantly different ($P > 0.05$) in luminosity and yellow coloration. The parameter a^* (green-red tendency), values in tortillas presented significant differences ($P < 0.05$) only at 85 °C, with an increase with ultrasound treatment indicating more pronounced red coloration (Table 5).

Conclusions

The application of ultrasound technology to maize nixtamalization may represent an improvement in pericarp removal and water absorption. Fick's model satisfactorily predicted water absorption during nixtamalization with and without ultrasound, with low dispersion between estimated and experimental data. In the case of calcium diffusion, it was found that Fick's model fit well but with greater dispersion. The gelatinization temperatures did not show significant changes for the different treatments, but the gelatinization enthalpy showed a decrease upon increasing the cooking temperature. The temperature of nixtamalization did not affect the texture of tortillas, while the masa adhesiveness value decreased with increasing cooking temperature. The masa color was affected significantly in terms of the luminosity and yellow coloration by temperature and ultrasound, whereas the tortillas did not show statistical differences in color. The ultrasound treatment reduced the nixtamalization time to 30 min, achieving adequate moisture levels for grain milling (44 g water·100 g^{-1}) to obtain masa and tortillas.

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Nomenclature

D_A	apparent diffusion coefficients, $m^2 \cdot s^{-1}$
l	length, m
w	width, m
h	heigth, m
UI	ultrasound intensity, $W \cdot m^{-2}$
UP	ultrasound power, W
dT/dt	change in temperature over time, $°C \cdot s^{-1}$
C_p	specific heat, $kJ \cdot kg^{-1} \cdot °C^{-1}$
m	mass, kg
A	area, m^2
SEE	standard error of estimated values
DSC	diferential scanning calorimetry
T_0	onset or initial temperature, ºC
T_p	peak or gelatinization temperature, ºC
T_e	final temperature, ºC
ΔHg	gelatinization enthalpy, J/g
MRF	maximum rollability force
MEF	maximum extensibility force
TPA	texture profile analysis

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