Changes in mass transfer, thermal and physicochemical properties during nixtamalization of corn with and without agitation at different temperatures

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1. Introduction

In Mexico the consumption of nixtamalized corn products both individually or combined with other foodstuffs has increased lately partly due to their nutritional and organoleptic attributes. Today, approximately 300 g of nixtamalized products including tortilla and are consumed per capita on a daily basis. Nixtamalization is an ancient process, where corn grain is cooked in ash-lime to produce softening. Actually, it involves cooking corn grain (Zea mays L.) in a Ca(OH)₂ solution, followed by steeping, washing and grinding, to obtain flour, masa, tortillas and other by-products (Martínez et al., 2001).

During nixtamalization, physical and chemical changes occur, as well as mass transfer phenomena, which are influenced by process conditions such as cooking temperature, agitation and alkaline medium concentration (Laria et al., 2005). The main changes that take place are the absorption of water and calcium, produced by the interaction of the alkali solution with the components in corn grain, which results in softening and solubilization of the pericarp. Calcium and water uptake are essential for the formation of amylose networks during the nixtamalization (Alvarado et al., 1999; González et al., 2004; Laria et al., 2007). Calcium absorption is initially limited by the presence of the pericarp. Once it has been removed, water and calcium diffusion increases to a level in which physical and chemical changes can occur; these changes are directly associated with the final quality of the products (González et al., 2005). During nixtamalization it is desirable to predict the uptake of these components as a function of time and temperature (Verma and Prasad, 1999), to improve control of the process.

Previous studies have reported moisture uptake in grains as corn, sorghum, (Charan and Prasad, 1996; Fan et al, 1963; Zazueta et al, 2002), rice (Lu et al., 1994) and amaranth (Rodríguez et al., 1995), using mathematical models to predict water absorption. Fick’s model and modifications have been used to predict moisture absorption in grains (Lu et al., 1994; Rodríguez et al., 1995; Verma and Prasad, 1999) for different temperatures and solute concentrations. Studies (Laria et al., 2005, 2007; Bressani et al., 2004; Trejo et al., 1982; González et al., 2004, 2005; Fernández et al., 2007, Mondragón et al., 2004b) have reported calcium and water absorption and starch gelatinization in corn grain under different conditions.
nixtamalization conditions. However, few studies have reported the relationship between the water and calcium absorption and pericarp removal and solubilization, and the changes that starch undergoes during the cooking process.

During nixtamalization, structural changes in the grain occur, producing rheological, functional and textural properties that determine the acceptability of the final product (Mendez et al., 2006; Mondragón et al., 2004a). Among the structural changes is the gelatinization of the starch. Changes in starch have been related with their thermal properties. Among these properties are temperature, ranges and enthalpy of gelatinization (Arámbula et al., 2006; Mendez et al., 2006; Mondragón et al., 2004b; Robles et al., 1988). Changes in thermal properties are produced by annealing reactions in starch that take place when it is maintained below the temperature at which starch gelatinizes. This results in the reorganization and recrystallization of polymer chains, yielding a more stable molecular configuration, which leads to an increase in gelatinization temperatures (Gomez et al., 1992; Robles et al., 1988). Fernández et al. (2007) carried out a nixtamalization process at 94 °C during 45 min of cooking reported that starch gelatinization can be inhibited by calcium ions promoting the aggregations and possible crosslink interactions and Jane (1993) reported the possible interaction of calcium ions with amylose chains of the starch. However, this mechanism is not clear because other authors as Cortés et al. (2005) reported for a nixtamalization fractioned process an increase in the viscosity due to this same interaction at high calcium hydroxide concentrations. These changes in starch have been evaluated through changes in thermal properties (Arámbula et al., 2006; Mendez et al., 2006; Mondragón et al., 2004b; Robles et al., 1988). Water and calcium diffusion during nixtamalization were studied by Herrera et al. (1986) and Verma and Prasad (1999), using models based on Fick's second law using spherical geometry for predicting diffusion of these components into the grain.

Even though nixtamalization is a process that has been extensively studied, there is an evident lack of information on small and large scales regarding predictive models that describe the mass transfer phenomena that occur during the process and the relationship with chemical and physical changes associated with the quality of the final products. The development of these models could contribute to the optimization and control of the nixtamalization process and contribute to improvement in the quality of products. The purpose of this study was to evaluate the changes in physical, chemical and thermal properties during nixtamalization at different temperatures in systems with and without mechanical agitation at pilot plant scale, as well as the development of a mathematical model for the prediction of water and calcium absorption, based on Fick’s model.

2. Materials and methods

2.1. Materials

Lots of hybrid white dent corn (Z. mays L.) with thin pericarp was used for this study. For the nixtamalization process, a food grade calcium hydroxide [Ca(OH)2, 98%], was used. The corn used for this study was analyzed for moisture, protein, fat, crude fiber, ash and calcium according to AOAC methods 950.02, 960.52, 920.53, 920.39, 962.09, 923.03 and 968.08 (AOAC, 1998); the pericarp was determined according to the procedure described by Arámbula et al. (2006). Moisture and calcium content and pericarp were of 11.82 g water 100 g⁻¹, 7 mg Ca²⁺ 100 g⁻¹ and 3.9%, respectively. Average size and shape of the grains were also determined assuming rectangular parallelepiped geometry (Fig. 1).

Fig. 1. Corn grain approximated by a rectangular parallelepiped. Grain dimensions average: b = 0.01174 m, c = 0.00922 m and d = 0.00453 m.

2.2. Nixtamalization

Lots of 2 kg of corn were nixtamalized in a Ca(OH)₂ [1.2% (w/v)] solution in a 1:3 ratio. Alkalinity of the system during the process was monitored measuring the pH of the cooking liquor (referred to as nejayote), keeping it at 12.6 ± 0.1. Nixtamalization was carried out at different temperatures (70, 80, 90 and 100 °C), with and without mechanical agitation. In the system without mechanical agitation, the cooking liquor and the grain during cooking were moved by convention currents produced by application of heat. Both systems were carried out in a stainless steel steam kettle enclosed in a jacket; for the mechanical agitation the kettle had a propeller agitator of 40 cm diameter set at 25 rpm. Samples of nejayote were taken every 10 min until 60 min of cooking and after 60 min of steeping, whereas the grain samples were taken every 10 min during the 60 min of cooking. After the cooking process, grains were steeped in nejayote, and sampled at different times during steeping (10–2880 min). Nixtamalized grains were washed twice with distilled water to eliminate excess Ca(OH)₂ and remnant pericarp. In the nejayote samples pH, soluble and suspended solids and total sugar content were determined. While in the nixtamalized grains with pericarp removed, moisture and calcium contents were measured. Kinetics for soluble and suspended solids, total sugar content, removed pericarp, water and calcium absorption were obtained. From the data for water and calcium absorption apparent diffusion coefficients were determined, using Fick’s second law. Thermal properties such as temperatures and enthalpy gelatinization were determined on nixtamalized dried grain samples. All treatments were duplicated.

2.3. Methods

2.3.1. Soluble solids

Soluble solids were determined using the refractometric AOAC method 932.14 (AOAC, 1998), measuring the refraction index with an Abbe refractometer (Atago, Japan). The analysis of each treatment was carried out in triplicate.

2.3.2. Suspended solids

Suspended solids were determined using a gravimetric method (Method 2450-DAPHA, 1995). Results were expressed in mg L⁻¹, and the analysis was carried out in triplicate.
2.3.3. Total sugars
A spectrophotometric method (Lambda 25 UV–Vis, Perkin–Elmer, MA, USA) was used; the sample was treated according to the phenol–sulfuric acid method described by Dubois et al. (1956). Total sugar content was carried out in triplicate and expressed in mg L\(^{-1}\).

2.3.4. Removed pericarp
Pericarp removed was measured by a gravimetric method described by Arámbula et al. (2006). Samples of ten corn grains, were weighed in an (Explorer Ohaus, NJ, USA) analytical balance, and dried at 50 °C for 36 h. Once dry the pericarp was removed manually and dried at 105 °C for 24 h and then weighed to determine the pericarp percentage in the grain. The analysis was carried out in triplicate.

2.3.5. Moisture
Moisture was determined using a gravimetric method 950.02, (AOAC, 1998). The analysis was carried out in triplicate, and results were expressed in grams 100 g\(^{-1}\).

2.3.6. Calcium
Calcium content in the grain was determined with AOAC method 968.08 (AOAC, 1998) by Flame Atomic Absorption Spectrophotometry (FAAS). It was carried out using a Perkin–Elmer AA Spectrophotometer (Model AA800, MA, USA). The instrument conditions were: air–acetylene flame, Ca\(^{2+}\) hollow cathode lamp, (2006). Values obtained were initial temperature (England), by the procedure described by Altay and Gunasekaran analyzed using Universal Analysis software (TA Instruments, Crawley, and a temperature ramp of 5

2.3.7. Gelatinization temperature and enthalpy
This analysis was carried out by Differential Scanning Calorimetry (DSC), using the method described by Gunaratne and Corke (2004, 2005), related these changes to pericarp and endosperm solvents being lixiviated during the cooking stage. González et al. (2008), Cuevas et al. (2006). The corn samples were previously cooked. Laria et al. (2005) associated the changes with cracks in the phenol–sulfuric acid method described by Dubois et al. (1956). Total sugar content was carried out in triplicate and expressed in mg Ca\(^{2+}\)100 g\(^{-1}\).

2.3.8. Diffusion coefficients
In this study, apparent diffusion coefficients, \(D_a\), were determined using a series solution to Fick’s second law in unsteady-state, assuming the grain has a finite laminar geometry, by means of Eq. (1).

\[
E = \frac{C - C_{\infty}}{C_0 - C_{\infty}} = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left[ - \frac{(2n+1)^2 \pi^2 D_c t}{2 \alpha} \right]
\]  

Where \(E\) is the fraction absorbed, \(C\) is average concentration at time \(t\), \(C_0\) is initial concentration, \(C_{\infty}\) is equilibrium concentration which was accomplished during steeping time when the grain reached the maximum absorption of either calcium and water, \(D_c\) is the apparent diffusion coefficient (m\(^2\) s\(^{-1}\)) and \(\alpha\) is the thickness in each side of the grain.

For the determination of the diffusion coefficient we assumed: a constant diffusivity, initially uniform concentration in calcium and water content, negligible resistance of the diffusion in the fluid surrounding the grain, and that the grain:water ratio did not change over time.

The geometry of corn grains was considered as a rectangular parallelepiped, in which total diffusion occurs through the six faces (Fig. 1). The grain dimensions were used in the calculation of the apparent diffusion coefficients.

The total water and calcium absorbed fraction was calculated from Eq. (2) (Crank, 1975):

\[
E = f \left( \frac{d_0}{b} \right) f \left( \frac{d_0}{c} \right) f \left( \frac{d_0}{d} \right) = E_b E_c E_d = f(D_a; t, a)
\]  

Where \(E\) is the total absorbed fraction, \(b\), \(c\) and \(d\) are the length, height and width of the parallelepiped, respectively, which replace thickness \((a)\) in Eq. (1); \(E_b\), \(E_c\) and \(E_d\) are the absorbed fractions for \(b\), \(c\) and \(d\), respectively.

The least squares estimates for apparent diffusion, \(D_a\), were calculated by minimizing \(\sum (E = f(D_a; t, a))^2\) with an iterative procedure, where \(f(D_a; t, a)\) is a solution for Fick’s second law. \(R^2\) was used as an indicator of the fit of the predicted absorbed fraction with respect to the experimental absorbed fraction.

2.4. Statistical analysis
Water and calcium kinetics data were used to calculate apparent diffusion coefficients. The data obtained from the analysis of the soluble and suspended solids, total sugar content and diffusion coefficients for water and calcium and thermal properties were subjected to a variance analysis using MINITAB, version 13.20 (MINITAB, 2000, PA, USA). Differences among the means were evaluated using Tukey’s test, with \(x = 0.05\).

3. Results and discussion

3.1. Soluble and suspended solids and total sugar content
Temperature and agitation had a significant effect (\(p < 0.05\)) on soluble and suspended solids content, yielding an increase in the nejayote. The total sugar content increased significantly only by temperature. Increase of these components was linear during the cooking stage, and was markedly higher during the first hour of steeping (Fig. 2). This tendency is associated with these components being lixiviated during the cooking stage. González et al. (2004, 2005), related these changes to pericarp and endosperm solubilization, due to their interaction with alkali solution during cooking. Laria et al. (2005) associated the changes with cracks in the pericarp, which result in the hydrolysis and lixiviation of soluble components as starch and monosaccharides, among other substances into the nejayote.

3.2. Removed pericarp
Removal of the pericarp was significantly affected (\(p < 0.05\)) by the cooking temperature and system agitation. Removal of the pericarp in corn grains (Fig. 3) increased with the cooking time for each cooking temperature, with and without mechanical agitation. Kinetics showed an exponential trend up to 1 h of cooking, for temperatures over 70 °C in both systems, with over 90% of pericarp removed for a cooking time of 1 h and 1 h of steeping at the different temperatures used. However, at 100 °C this percentage of pericarp elimination was reached at 40 min of cooking. Also it was observed that at 70 °C agitation had a significant influence on pericarp elimination, with double the pericarp removal compared with systems without mechanical agitation (60 and 30%). These results correlate with those obtained for soluble and suspended solids and
total sugar content (Fig. 2), which increased during cooking and steeping. This is due to hydrolysis and softening of the grain during the alkaline cooking (González et al., 2004, 2005; Laria et al., 2005), and which is accelerated by increase in temperature, which is a critical variable in the degradation of the pericarp structure (Gutiérrez et al., 2008a).

3.3. Water absorption

Water absorption was significantly influenced ($p < 0.05$) by the temperature of the nixtamalization process, showing significant differences between 70 and 100 °C. Mechanical agitation had no effect. Water absorption (Fig. 4) showed a linear increase with the rise in temperature and cooking time, reaching values between 40 and 44 g water 100 g$^{-1}$ of nixtamal at temperatures over 80 °C, with and without mechanical agitation. On the other hand, an increase in the water absorption rate was observed during steeping; the highest value was reached at 480 min of steeping (8 h) for all temperatures, with and without agitation. Maximum water absorption values were between 45 and 59.72 g water 100 g$^{-1}$ of nixtamal for the different nixtamalization conditions. This can be attributed to the fact that, during the cooking process, water diffuses through the whole grain, with some limitation by pericarp, without reaching equilibrium during the cooking. Whereas that in the steeping, the water absorption shows a notable effects in the first hours, which can be attributed to the removal of a large portion of the pericarp (90%), allowing rapid diffusion, reaching an asymptotic equilibrium after 8 h of steeping.

Similar results were reported by Laria et al. (2005), which indicate that the increase in cooking temperature provokes an increase in water absorption by the corn grain during nixtamalization. Water absorption can also be increased or decreased during nixtamalization by other factors as the composition and dimensions of the grain, and also by the process variables (cooking temperature and agitation). Other studies, such as that carried out by Trejo et al. (1982) have reported water absorption values of 40% during the cooking process at 13 min, and water content of 105% at 135 min for popcorn. However this grain is smaller and has different features composition compared to other corn types for which lower water absorption values have been reported.
3.4. Calcium

Calcium absorption in nixtamalized grain significantly increased (p < 0.05) with increase in temperature only during the steeping stage, for systems with and without agitation. Fig. 5 shows the kinetics of calcium absorption under different nixtamalization conditions. It was observed that calcium ion diffusion during the cooking stage showed no significant differences at different temperatures, with or without mechanical agitation. Values reached during cooking were between 44.48 and 80.62 mg Ca²⁺/100 g at any nixtamalization temperature, which is 6–11 times more than the initial concentration. During steeping, calcium content increased with temperature for systems with and without agitation. The maximum equilibrium concentration was reached after 24 h of steeping at all temperatures for both systems. Calcium values obtained for the steeping stage were 131.13, 280.40, 447.83 and 463.19 mg Ca²⁺/100 g at 70, 80, 90 and 100 °C for systems without mechanical agitation, while for systems with agitation for the same temperatures, they were 217.63, 257.11, 385.46 and 496.69 mg Ca²⁺/100 g, respectively. It was noticed that agitation had a considerable effect in calcium absorption at low temperatures (70–80 °C), while at 90 and 100 °C for systems with agitation there was no significant increase (p < 0.05). Cooking temperature has a stronger effect than agitation, and similar findings were reported by Laria et al. (2005) and Gutiérrez et al. (2008b). These calcium content results are also consistent with those reported by Bressani et al. (2004), who carried out the nixtamalization process under similar conditions (94 °C, 1.2% calcium hydroxide, 70 min of cooking time and different steeping times).

During the cooking process, calcium diffusion to into the grain was limited by the presence of pericarp, which is a barrier to ion diffusion. However, during the steeping, calcium absorption increased significantly after pericarp elimination, which allowed calcium to reach the equilibrium concentration. Similar results were reported by Laria et al. (2005) and Gutiérrez et al. (2008b). Also, other studies showed similar behavior such as those reported by Fernández et al. (2006) and Trejo et al. (1982) for nixtamalization processes using microwaves and conventional nixtamalization, respectively, reaching equilibrium at shorter steeping times, which was attributed to type of energy used to cook, as well as to the composition and dimensions of the type of corn.

3.5. Apparent diffusion coefficients

Table 1 shows apparent diffusion coefficients for water and calcium absorption for the different temperatures and nixtamalization systems. Apparent diffusion coefficients of water showed significant differences (p < 0.05) at 70 and 100 °C, for systems with and without agitation. However, no significant difference was observed in calcium diffusion coefficient values for different experimental conditions. Fig. 6 shows experimental and adjusted fractional absorption values for water and calcium diffusion, where the proposed model explained from 98.9 to 93.4% of the experimental variation. Water diffusion coefficients were between 2.07 and 3.52 m² s⁻¹ × 10⁻¹⁰, with R² of 0.94–0.98. During the cooking stage, water content increased linearly, stabilizing during steeping.
The rapid absorption of water at the beginning of the process can be attributed to the physical and chemical changes that occur during nixtamalization, which provoke dissolution of the peripheral components of the grain, as shown in Fig. 2. Calcium diffusion into the grain under different experimental conditions was adequately described by Fick’s law. Values of diffusion coefficients were between $0.332 \times 10^{-10}$ and $1.43 \times 10^{-10}$ m$^2$ s$^{-1}$ with an $R^2$ of 0.84–0.96. The poorer fit compared with those obtained for water can be attributed to the molecular size and to the reaction of calcium with pericarp components (González et al., 2004, 2005) and starch (Robles et al., 1988; Tovar et al., 2004; Jane, 1993) during diffusion, causing a decrease in absorption rate. Diffusion coefficient values obtained in this study were lower than those reported for water at $25 \pm 176^\circ$C ($3.58 \times 10^{-10}$ m$^2$ s$^{-1}$) which indicates how the grain is a barrier to mass transfer, which limits free calcium diffusion during the nixtamalization process.

### 3.6. Gelatinization temperature and enthalpy

Temperature and enthalpy gelatinization were significantly affected ($p < 0.05$) by temperature and cooking time for both systems. The Tables 2 and 3 show the values of temperature, ranges and enthalpy of gelatinization for systems with and without mechanical agitation. A slight increase in temperature values, ranges and enthalpy gelatinization values was observed when cooking temperatures and time were increased, but no significant changes were observed in either of the systems. At 70 and $100^\circ$C, significant differences in $T_p$ were observed for both systems, an increase in gelatinization temperature was obtained for the system with mechanical agitation at $100^\circ$C, while the opposite effect was observed for the system without agitation. $T_p$ values ranged from 68.5 to 76.9 °C for the different cooking temperatures and times with 60 min of steeping in systems with and without agitation. No significant changes were observed in gelatinization enthalpy and temperature ranges for different cooking temperatures and times. Enthalpy values and ranges were between 2.3–5.2 J g$^{-1}$ and 9.8–19.3 °C, respectively, for systems with and without agitation.

### Table 1

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Agitation</th>
<th>Water $D$ (m$^2$ s$^{-1}$) $\times 10^{-10}$</th>
<th>Water $R^2$</th>
<th>Calcium $D$ (m$^2$ s$^{-1}$) $\times 10^{-10}$</th>
<th>Calcium $R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>70</td>
<td>Without</td>
<td>2.27$^a$</td>
<td>0.947</td>
<td>1.42$^a$</td>
<td>0.911</td>
</tr>
<tr>
<td></td>
<td>With</td>
<td>2.07$^a$</td>
<td>0.947</td>
<td>0.489$^a$</td>
<td>0.960</td>
</tr>
<tr>
<td>80</td>
<td>Without</td>
<td>2.39$^{ab}$</td>
<td>0.955</td>
<td>0.332$^a$</td>
<td>0.943</td>
</tr>
<tr>
<td></td>
<td>With</td>
<td>2.98$^{ab}$</td>
<td>0.967</td>
<td>0.417$^a$</td>
<td>0.844</td>
</tr>
<tr>
<td>90</td>
<td>Without</td>
<td>2.95$^{ab}$</td>
<td>0.969</td>
<td>0.482$^a$</td>
<td>0.942</td>
</tr>
<tr>
<td></td>
<td>With</td>
<td>2.9$^{ab}$</td>
<td>0.974</td>
<td>1.09$^a$</td>
<td>0.912</td>
</tr>
<tr>
<td>100</td>
<td>Without</td>
<td>3.52$^b$</td>
<td>0.987</td>
<td>1.43$^a$</td>
<td>0.888</td>
</tr>
<tr>
<td></td>
<td>With</td>
<td>2.96$^b$</td>
<td>0.971</td>
<td>0.719$^a$</td>
<td>0.941</td>
</tr>
</tbody>
</table>

* Values presented are the average of duplicate measurements. Average values in each column with different letters represent a significant difference, according to the Tukey test ($a = 0.05$).
out mechanical agitation. Increases in gelatinization temperatures \( (T_o, T_p \text{ and } T_e) \) and the broadening of the range \( (T_e-T_o) \) have been attributed to Ca\(^{2+}\) interaction with hydroxyl groups in the amyllose chain in starch (Robles et al., 1988; Jane, 1993) and the annealing of starch during the process, which reorganizes and stabilizes these chains (Mondragón et al., 2004b; Robles et al., 1988).

Gelatinization temperatures and enthalpies values are within previously reported ranges (Mondragón et al., 2004b; Loisel et al., 2006; Robles et al., 1988). Higher values have been reported by Mendez et al. (2006) and Li et al. (2007), which can be attributed to a series of factors: corn variety, starch’s crystalline properties, intermolecular bonds between starch chains and other grain components, and the heating rate of the starch suspension; which all affect corn starch’s thermal properties (Mendez et al., 2006; Huijbrchts et al., 2008).

### 4. Conclusion

Cooking temperature during the nixtamalization process had a significant effect on pericarp removal and also on water and calcium absorption. Nixtamalization with mechanical agitation at low temperatures (70 and 80 °C) increased pericarp removal significantly, producing twice as much elimination of pericarp, while at higher cooking temperatures the mechanical agitation had no significant effect. Pericarp removal values were over 90% for cooking temperatures above 70 °C, with and without mechanical agitation. Water absorption was significantly affected by increasing cooking temperature reaching values from 40 to 44% of water absorption while with steeping; the maximum absorption was reached at 8 h, with 45 and 59% of absorbed water. Calcium absorption is limited by the pericarp however; it increased 6–11 times its initial concentration. Calcium absorption is limited by the pericarp however; it increased 6–11 times its initial concentration. Fick’s model for a finite slab with parallelepiped-like geometry, satisfactorily predicts water and calcium absorption during nixtamalization with and without mechanical agitation, explaining 84–98% the variation of the experimental data. Significant differences in gelatinization temperatures were observed at 70 and 100 °C, for systems with and without mechanical agitation. However, enthalpy and range did not show significant changes for the different experimental conditions.

The information in this study can contribute to the optimization of the nixtamalization process, which will ultimately result in quality improvement of the final products.
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