Study of Calcium Ion Diffusion in Components of Maize Kernels During Traditional Nixtamalization Process

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ABSTRACT

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Our report shows the calcium ion diffusion process through the different parts of maize kernels (pericarp, endosperm, and germ) during the traditional nixtamalization process as a function of steeping time (t) 0–24 hr. The cooking step of the nixtamalization process used 3 kg of maize kernels in 6L of water and 2% calcium hydroxide (w/w). The cooking temperature was 92°C for 40 min. The calcium content of the samples was measured using atomic absorption spectroscopy. We found that the whole instant corn flour, pericarp, endosperm, and germ, had a nonlinear relationship to steeping time, showing a local maximum at 9 hr. Analysis of the different parts of the nixtamalized kernels showed that in short steeping times (0–5 hr) calcium diffusion took place mainly in the pericarp. Calcium diffusion in the endosperm and germ occurred gradually over longer steeping times. However, the physical state of the kernels (broken kernels) accelerated the diffusion process. Calcium diffusion occurred first in the pericarp, followed by the endosperm and germ. Immediately after cooking (t = 0 hr), we found a 1.148% calcium content in the pericarp, 0.007% in the germ, and 0.028% in the endosperm. After 24 hr of steeping, the calcium contents were 2.714% in the pericarp, 0.776% in the germ, and 0.181% in the endosperm. In another study, the calcium content in the endosperm was measured by first separating the 10% from the outermost, followed by another 10% from the next endosperm tissue, and concluding with the remaining 80%. Calcium ions were present mainly in the outermost layers of the endosperm. The damaged kernels steeped for more than 5 hr showed greater calcium concentrations than the undamaged counterparts.

The nixtamalization is a traditional Mexican process that consists of alkaline cooking and steeping of maize using calcium hydroxide. This process is increasing in the United States and all over the world with the spread of Mexican and Mesoamerican foods and nixtamalized products such as corn flour, tortillas, tacos, and tortilla chips. In the traditional nixtamalization process, nixtamal is steeped in the cooking water and then washed at least twice to remove the remains of organic components (pericarp, germ, and endosperm fractions) and excess calcium. After this step, the nixtamal is ready to produce masa or dehydrated masa for the production of instant corn flour. This process continues to be highly relevant for the tortilla industry because the homemade tortillas are the standard of quality.

The amount of calcium incorporated during the nixtamalization process in the whole kernels as well as in pericarp, endosperm, and germ is very important because the interaction between calcium hydroxide and the different components of the kernels determined the physicochemical and sensorial characteristics of the products (masa, instant corn flour, or tortillas). Paredez-Lopez and Saharopulos (1982) reported microstructural changes that occurred during alkaline cooking and steeping of corn, whereas Trejo-Gonzalez et al (1982) studied the nixtamal chemical changes (minerals, vitamins, amino acids, and starch) as a function of different steeping times (0 and 12 hr). They found that lime treatment improved the nutritive value of the corn by increasing the availability of lysine in the glutelin fraction and the gelatinization of starch. In addition, authors found an increment in the calcium content as a function of steeping time. Rodriguez et al (1995, 1996) found changes in the thermal diffusivity, texture, X-ray patterns (relative crystallinity quality), and infrared spectra properties as a function of the lime concentration at 0–0.5% (w/w). For products containing calcium at 0–0.25% (w/w), the thermal diffusivity, peak viscosity, and crystallinity increased when the calcium content increased and an opposite behavior was observed for calcium concentrations >0.25%. Bryant et al (1997), studied the swelling power, water retention capacity, and degree of gelatinization of corn flour cooked in water with and without lime; at low concentrations, lime increased swelling and starch digestibility and then decreased at higher lime concentrations. Fernandez-Muñoz et al (2001), found that calcium contents of 0.25% (w/w) increased the starch thermal diffusivity and crystallinity quality. This improvement in the thermal and structural properties is related to the cross-linking of the polymers chains. More recently, Fernandez-Muñoz et al (2002), demonstrated that the variation in calcium content in instant corn flour prepared at different steeping times is a result of a simultaneous kinetic diffusion process taking place in the pericarp, endosperm, and germ, but that this calcium diffusion during steeping is not a linear process and that the changes in the structural and rheological properties as a function of the steeping time nonlinear. A more detailed qualitative description of the diffusion of calcium into the kernel during the nixtamalization process has been reported by Zazueta et al (2002), who used a calcium-45-based technique to give a detailed picture of calcium absorption in corn kernels as a function of steeping time for kernels with and without damage. They found that there is quasi-simultaneous calcium ion diffusion in the pericarp, endosperm, and germ during the nixtamalization process as a function of steeping time.

This study showed that the incorporation of calcium does not proceed at the same rate for all grains and can occur abruptly with significant kernel damage. McDonough et al (1987, 2001) studied the distribution of calcium in maize kernels at different stages of the nixtamalization process. They concluded that, for short steeping times, calcium ions are mainly deposited in the pericarp and the germ, with some penetration up to and into the aleurone layer, and perhaps even into the innermost endosperm layers.

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We present a quantitative analysis of calcium ion diffusion in the anatomical parts of maize kernels (pericarp, endosperm, and germ) during the traditional nixtamalization process as a function of steeping time (0–24 hr). Because calcium ion diffusion occurs at different rates in the pericarp, endosperm, and germ (Zazueta et al 2002), we measured the calcium concentration in these three main parts of the corn kernel, with particular attention to the endosperm. Differences in calcium content between damaged and undamaged kernels were also examined.

MATERIALS AND METHODS

Sample Preparation

Commercially available Toluca maize was obtained from a local market in Querétaro City. In the initial cooking step of the nixtamalization process, each sample of this series (TNP92) was prepared by cooking 3 kg of whole maize kernels in a solution of 6L of water and 60 g of calcium hydroxide (reagent powder, Fermont, Monterrey, NL, Mexico); with calcium hydroxide at 2% of the weight of the maize. This lime concentration (normal saturation) is in the range reported previously by Trejo-Gonzalez et al (1982), and Fernandez-Muñoz et al (2002). Kernel properties were dervied from 1,000 kernel weights taken on randomly selected subsamples of 200 kernels (368.32 g ± 6.82): crude protein (N × 6.25, 8.61%) measured by the micro-Kjeldahl method (Approved Method 40-70, AACC 2000), moisture 10.59% (Approved Method 30-20), total ether extract 4.46% (Approved Method 44-15A), insoluble dietary fiber 8.58%, soluble dietary fiber 1.94%, total dietary fiber 10.52% (method 925.10, AOAC 1995), ash 1.05%, and total damaged kernels 5.86%.

Maize kernels were cooked for 40 min at 92°C. After cooking, the maize was steeped for 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 13, 15, 18, and 24 hr. After steeping each sample, the cooking liquor (nejayote) was drained off and the nixtamal samples were washed twice in water using a 2:1 (v/w) ratio by stirring the kernels in the wash water for 2 min. After washing and draining, 2.6 kg was ground (FUMASA, M100, Querétaro) into corn masa and then dehydrated using a flash type dryer (Cinvestav-AV, M2000, Querétaro). The dryer conditions were adjusted to have 250°C inlet air temperature and 90°C to the exhaust air to avoid burning the material. Then the material was remilled using a hammer mill (PULVEX 200, Mexico DF, Mexico) equipped with a 0.8-mm screen. The remaining 0.4 kg of each sample was used to determine calcium content of the different anatomical parts of the corn kernels.

The pericarp, endosperm, and germ of each soaked kernel were separated manually and dried in an air oven at 40°C for 6 hr (each component part reached a relative moisture content of ≈10%). Because the endosperm constitutes ≈80% of the whole kernel, the outermost 10% and next 10% of the dry undamaged endosperm were removed manually by sanding all its faces. The first sanding (10%), second sanding (10%), and the 80% remaining of the endosperm were then tested for calcium contents to achieve a more detailed picture of calcium diffusion in the endosperm. Dry endosperm with physical damage was selected by optical inspections to quantify the calcium content.

Dry Matter Loss During Cooking and Steeping

After the indicated steeping times (0–24 hr), the samples were washed twice according to the process described above and the water from the first and second washes as well as the steeping water (nejayote) was evaporated until dryness. The total remaining solids were used to calculate the total dry matter lost as a function of steeping time.

Atomic Absorption Spectroscopy

The calcium content of the instant corn flours as well as the different parts of the kernels (pericarp, endosperm, and germ), damaged endosperm, different layers of the undamaged endosperm, and unwashed nixtamalized kernels, washed nixtamal samples with one or two washings, and samples steeped for different times were determined by the dry-ashing method 968.08 (AOAC 1998).

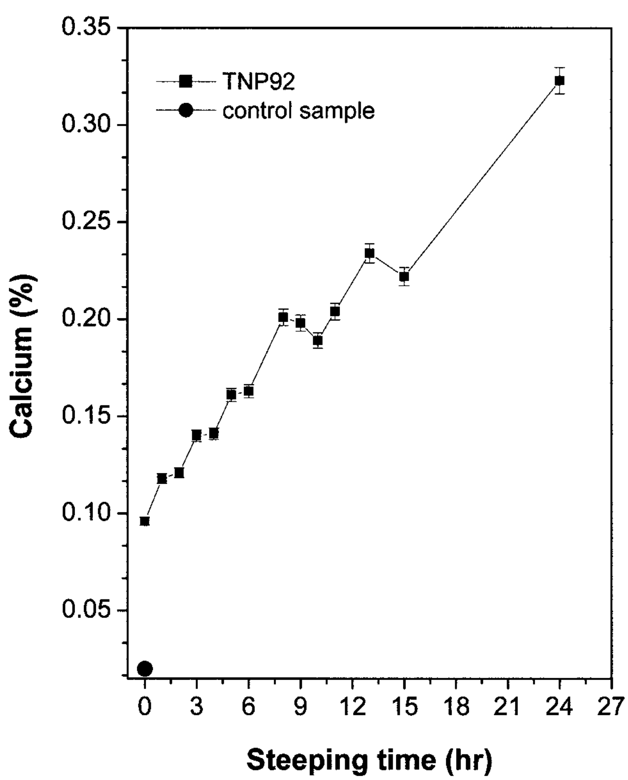

Fig. 1. Calcium content of instant corn flour prepared by traditional nixtamalization process at 92°C as a function of steeping time. Error bars indicate standard deviation.

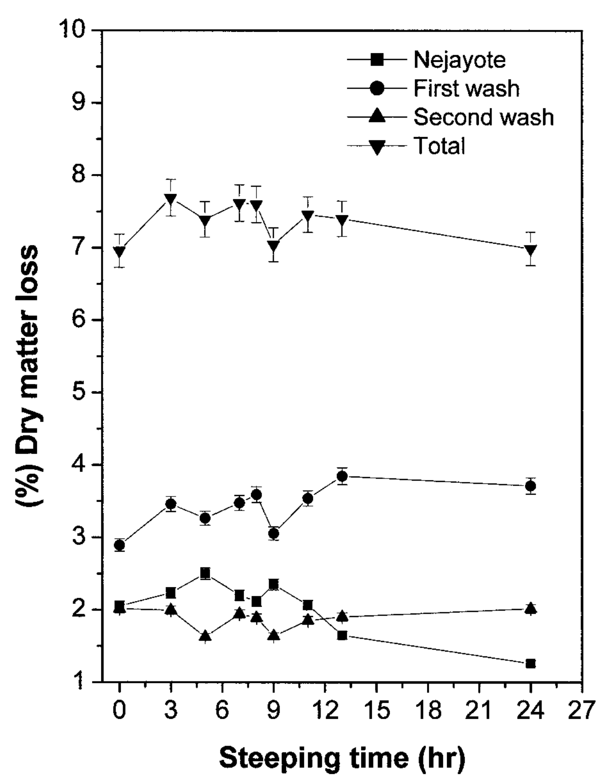

Fig. 2. Effects of steeping time on loss of corn dry matter for traditional nixtamalization process at 92°C (TNP92). Error bars indicate standard deviation.

The calcium ion concentration was measured with a double-beam atomic absorption spectrometer (Analyst 300 Perkin Elmer) equipped with a deuterium lamp, background corrector, and a hollow cathode lamp. The equipment was operated with 12 psi of dry air, 70 psi of acetylene, 422.7 nm flame, a 10 mA lamp current, and 0.7 nm slit width.

RESULTS AND DISCUSION

Calcium Content in the Traditional Nixtamalization Process

Figure 1 shows the calcium content of the instant maize flours as a function of steeping time for samples cooked at 92°C using the traditional nixtamalization process (TMN92) and a control sample cooked at 92°C without calcium. The calcium content values in Fig. 1 represent the average of three measurements; the calcium content of the control sample after cooking (without calcium) was ≈0.024% (w/w). In comparison, the calcium content of the sample with 0 hr of steeping time was ≈0.092% (w/w). This means that the kernels absorbed a significant amount of calcium during cooking (an increase of ≈400% in the calcium content). This can be explained using two time frames as reported recently by Fernandez et al (2002) for samples cooked at 72°C. The first time frame was $0 < t < 9$ hr, and the second was $t > 10$ hr. The first time frame was subdivided into one at $0 < t < 5$ hr, in which the calcium ion diffusion probably took place mainly in the pericarp, and a second at $5 > t > 9$ hr, in which the calcium diffusion process also included the endosperm and germ. The calcium content of the TNP92 instant corn flours followed an S-shaped curve for steeping times from 0 to 9 hr. S-shaped curves, known as logistic curves (Silva and Miranda et al 1994; Fernandez et al 2002), describe second-order kinetics in which a given system undergoes a transition between two saturation values. This transition occurs in a somewhat localized manner around a given value. Mathematically, the logistic curve can be written as $f = f_o + \Delta f[\Theta/(1+\Theta)]$, where $\Theta = \exp[(t - t_c)\ \Delta t]$. Here, f represents the calcium content, f_o represents the initial value of f ($t = 0$ hr), Δf is the overall change in f going from low to high saturation values, and t_c is the value of t when f reaches the halfway point, changing its curvature in an abrupt manner during a characteristic interval Δt around t_c. Fitting the calcium content data as a function of the steeping time for $0 < t < 9$ hr for the TNP92 samples, the values were $f_o = 0.091\%$, $\Delta f = 0.134\%$, $t_c = 5.21$ hr, $\Delta t = 2.51$ hr, and chi-square $= 5 \times 10^5$. The t_c values are related to the partial permeability and degradation of the endosperm. According to Fernandez-Muñoz et al (2002), the value for t_c for the corn cooked at 72°C for 100 min (TNS and OS) was 3.45–3.73 hr, but this value changed to 5.21 hr for corn kernels cooked at 92°C for 40 min. This means that the temperature of the alkaline solution had a strong influence in the pericarp degradation. According to Zazueta et al (2002), the solubility of calcium hydroxide in water changes from 0.185% (w/w) at 0°C to 0.080% (w/w) at 100°C and this fact could have influenced the calcium content in the instant flours. For $t > 9$ hr, the calcium content of the instant corn flour behaved differently, the decrease in the calcium content could be related to the loss of dry matter during the washing process. It is well known (Pflugfelder et al 1988; Sahai et al 2000) that dry matter is lost when nixtamalized corn is washed. This loss may have affected the total calcium content of the instant corn flour. The increased calcium content for steeping times >12 hr is probably related to calcium diffusion in the internal layers of the endosperm and the germ.

Dry Matter Loss

Figure 2 shows the dry matter lost during the washing process for the TNP92 samples. The total dry matter lost is the result of solids lost during the first and second washes as well as in the nejayote. The first wash produced a dry matter loss of 2.89–3.84%, while the second wash produced a dry matter loss of 1.62–2.01%. It is very important to note that the first wash produced a greater dry matter loss. Solid losses in the nejayote were 1.26–2.23%. The

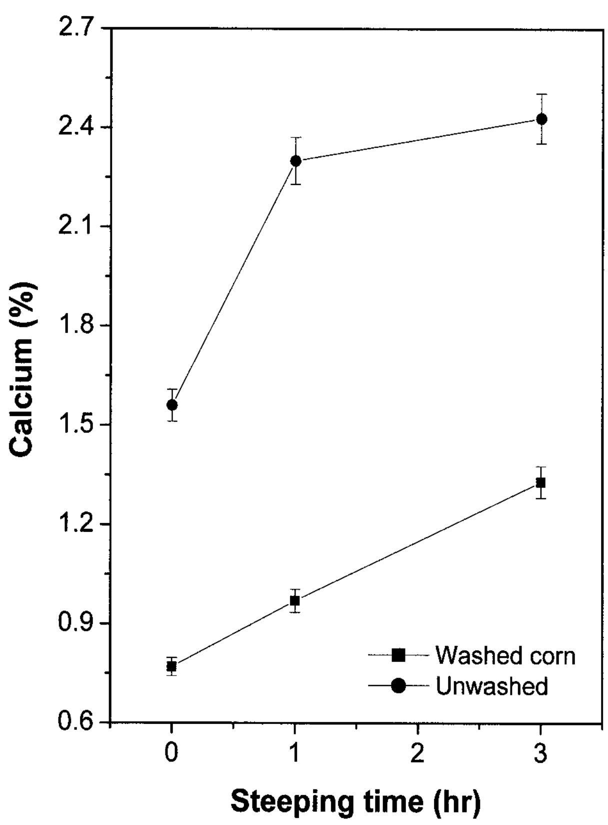

Fig. 3. Difference in calcium content between washed and unwashed corn kernels for steeping times of 0–3 hr.

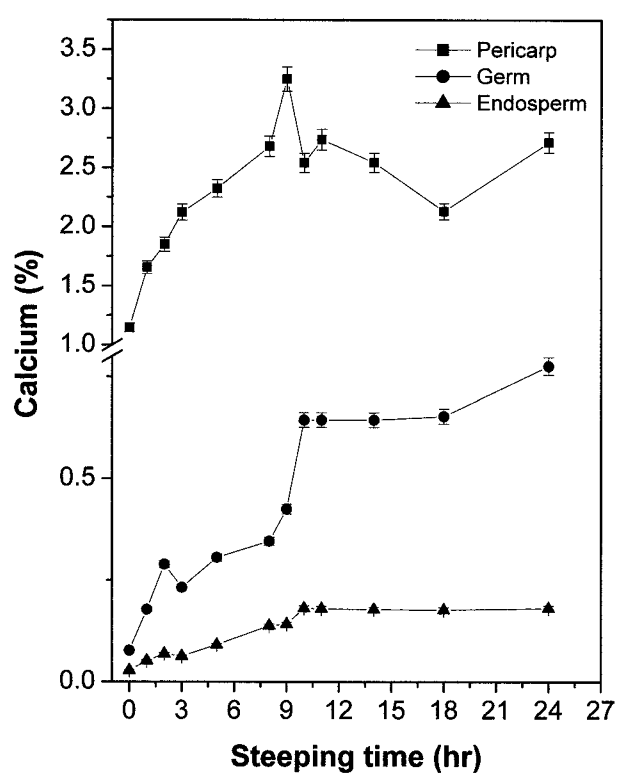

Fig. 4. Calcium content of different components of corn kernel (pericarp, endosperm, and germ) as a function of steeping time. Error bars indicate standard deviation.

total dry matter loss as a function of steeping time for the traditional nixtamalization process was 6.98 and 7.68%. Previously, Katz et al (1974) reported a 5–14% loss of dry matter during the conversion of raw corn into nixtamal using the traditional process. According to Pflugfelder et al (1988), the dry matter loss during nixtamalization was 4% for nixtamal steeped for 0 hr and 14% for counterparts steeped for 24 hr, in this case nixtamal was washed once. Figure 3 shows the difference in the calcium content between washed and unwashed corn kernels for steeping times of 0–3 hr. This shows that the main effect of the washing process is to remove excess calcium ions located in the external layers of the pericarp and endosperm and this process is time-dependent (steeping time). According to Martinez Bustos et al (2001), excessive washing has a negative effect on the rheological and textural properties of masa and tortillas due to the solubilization of noncellulose wall polysaccharides from the germ, pericarp, and tip cap fractions.

Calcium Content in Grain Components

Figure 4 shows the calcium content of the pericarp, endosperm, and germ as a function of steeping time (0–24 hr) for TNP92. Two different time frames were observed in the pericarp: one at 0–9 hr and one at $t > 10$ hr. In the first time frame, the calcium content increased with increased steeping time, showing a change in the slope at ≈5 hr and a saturation process at ≈9 hr. The sharp decline in the calcium content observed for $9 < t < 12$ hr reflects the loss of a large part of the pericarp when these long-treated grains were washed. This result is in agreement with experiments performed by Pflugfelder et al (1988), in which the pericarp constituted most of the dry matter lost during alkaline processing. Calcium ion diffusion in the germ followed almost the same behavior as that of the pericarp at 0–9 hr, reaching a saturation point at ≈9 hr. The rate of calcium diffusion in the germ for the interval of 5–10 hr is greater than the rate for 0–5 hr. Results obtained by fitting the experimental data to the logistic curve suggests that this increment could be related to the permeability of the pericarp. A decrease in the loss of dry matter but an increase in the total calcium content was observed in products steeped for 13–24 hr. Calcium ion diffusion in the germ and endosperm did not follow linear curves at least for $0 < t < 10$ hr; in both cases a saturation process existed.

Distribution of Calcium in Endosperm

Figure 5 shows the calcium ion content of the outermost 10% (first sanding), the next 10% (second sanding), and the remaining 80% of the endosperm. The calcium content increased in the outermost 10% of the endosperm from 0 to 24 hr of steeping time. This increment is ≈1,600%. In the next 10% (second sanding), the calcium content steeped from 0 to 24 hr increased ≈600%, while in the remaining 80% for the same steeping period, the calcium content increased ≈260%. For steeping times at 0–5 hr, calcium ion diffusion in the endosperm took place only in the outermost layers due to the fact that calcium ion diffusion at the beginning was taking place mainly in the pericarp. The calcium content of the whole endosperm began to increase at ≈5 hr due to the partial permeability of the pericarp (Fig. 3). This result was confirmed by fitting the experimental data to a logistic curve in which we found a characteristic t_c at 5.21 hr. This t_c represents the partial permeability of the pericarp, allowing greater calcium penetration of the endosperm.

The foregoing results confirm that the calcium ion distribution in the endosperm is also a kinetic process taking place from the external to the internal layers. A saturation process took place after 10 hr for the calcium ion diffusion into the endosperm taken as a whole (Fig. 3).

Calcium Content in Damaged Kernels

The traditional nixtamalization process always includes both whole kernels and partially damaged ones. This means that some kernels (in this study 5.86%) show some kind of physical damage (fissured, completely broken, without germ, etc.). The total calcium

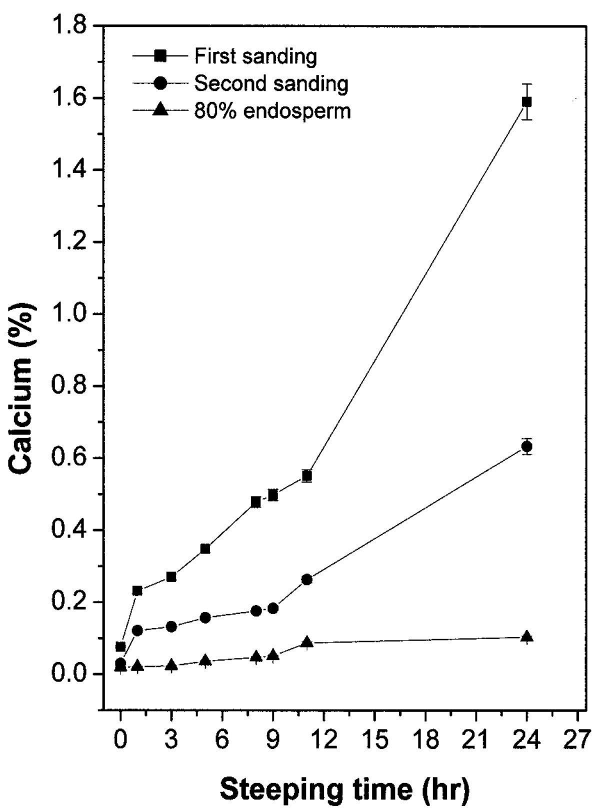

Fig. 5. Calcium content of outermost 10%, next 10%, and remaining endosperm as a function of steeping time.

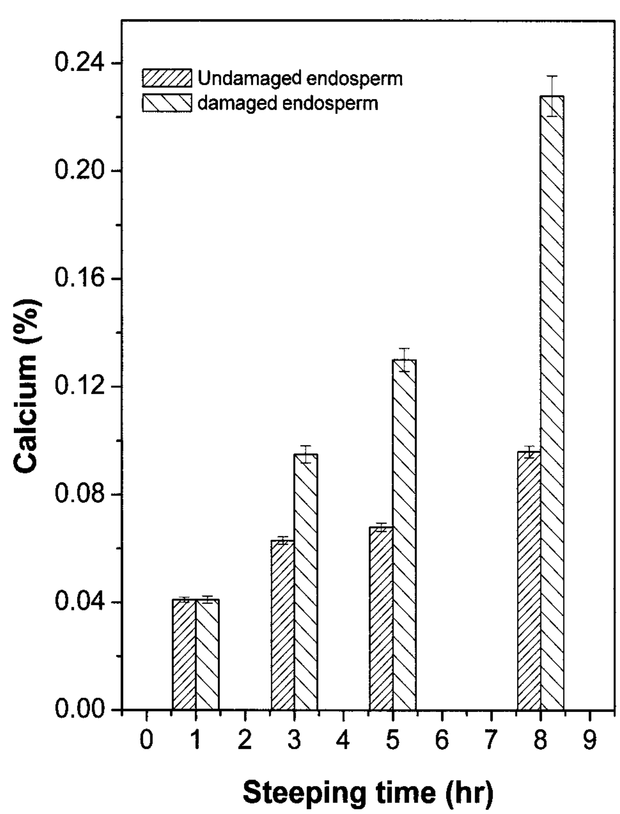

Fig. 6. Calcium content of whole and damaged endosperm as a function of steeping time.

content of the final corn flour produced is determined by the calcium ion diffusion in both the whole and damaged kernels. Zazueta et al (2002) showed that the calcium absorbed by the kernels varies considerably from kernel to kernel and increases abruptly in kernels with significant damage. Figure 6 shows the calcium content of damaged and undamaged nixtamalized corn kernels as a function of steeping times at 0–8 hr. Early on in the calcium ion diffusion process, there were no significant differences in the calcium content when damaged and undamaged kernels were compared. The calcium ion diffusion for damaged kernels for 0–8 hr followed a linear relationship. The calcium content increased more rapidly in the damaged kernels due to greater effective area for calcium diffusion. In addition, after 5 hr, the calcium content in the damaged kernels increased dramatically. This may be due to pericarp solubilization.

CONCLUSIONS

The calcium ion diffusion in the whole corn kernel was not a linear process due to the structure of the grain; the diffusion first took place mainly in the pericarp at 0–5 hr, and then in the rest of the kernel. These results are in agreement with Zazueta et al (2002), who showed that calcium ion diffusion becomes important in the internal parts of the kernel (endosperm and germ) only after long or prolonged steeping times. Results showed that the calcium ion diffusion process in the corn kernel fit a logistic curve. This result is not in agreement with a first-order dependence in a previous work (Trejo-Gonzalez et al 1982). Calcium ion diffusion is a quasi-simultaneous kinetic process taking place first in the pericarp and then into the germ and endosperm with different rates of incorporation. A detailed structural analysis of the endosperm showed that the calcium ions were mainly located in the outermost layers of the endosperm. For short steeping times, the calcium ions were present only in the external layers but when the steeping times increased, they penetrated the entire endosperm. The entry of calcium into the interior of the endosperm depended on the physical state of the grains; broken and cracked kernels had a more effective area for calcium migration. The main effect of the washing procedure for short steeping times was to remove excess calcium ions present in the external part of the pericarp and, for longer steeping times, the removal from the outer layers of the endosperm. Calcium ion diffusion in the corn kernel (pericarp, germ, and endosperm) during the traditional nixtamalization process is governed by cooking temperature, steeping time, initial level of calcium hydroxide, and water content.

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