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Evaluation of physicochemical changes in nixtamalized maize flours as a function of steeping time

J.L. Fernández-Muñoz *, E. San Martín-Martínez, J.A.I. Díaz-Góngora, A. Calderón, A. Alvarado-Escobar, H. Ortiz-Cárdenas

Centro de Investigación en Ciencia Aplicada y Tecnología Avanzada del Instituto Politécnico Nacional, Unidad Legaria 694, Col. Irrigación, México D.F., Z.P. 11500, Mexico

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Abstract

The physicochemical properties of nixtamalized corn flours for different steeping times were studied, evaluating the particle size distribution (PSD), calcium concentration, viscosity, and crystallinity. Nixtamalized maize kernels with different steeping times were milled, dehydrated, and pulverized, which resulted in a variation in the particle size of the corresponding flours. The PSD was separated into two groups according to their percentage share in the flours: Group I (40, 60 and 80 US mesh), and Group II (30 and 100 US mesh). The calcium content increased in a nonlinear way as a function of increases in the steeping time. The greatest values were for the 100 US particle size. The maximum viscosity of the flours increased with steeping time and with diminishing particle size, for particle size greater than 30 US. The crystallinity of our samples varied from 9% to 14%, as a function of steeping time and calcium concentration. For the samples with steeping times of 8 and 9 h, the crystallinity behavior is similar: between 9% and 10.7%, respectively. The results obtained indicate that steeping time significantly improves the physicochemical properties of nixtamalized maize flours, as well as the quality of the final product.

Keywords: Nixtamalized maize flour; Particle size distribution; Calcium content; Viscosity; Crystallinity

1. Introduction

The physicochemical state of different PSD fractions is considered to be an important criterion for nixtamalized maize flour applications in the production of tortillas or derived products according (Bedolla & Rooney, 1984; Gomez, Waniska, & Rooney, 1991). Mexican tortillas require flour with a particle size distribution consisting of fine particles for greater flexibility and cohesion, whereas fried Mexican tortillas require a coarse particle size distribution, which gives a crispy texture to the tortillas after frying (Montemayor & Rubio, 1983). When the particle size distribution properties of the flour are similar, this promotes the formation of better quality masa, and hence reduces blistering and diminishes oil absorption during the frying process (Gomez et al., 1991; Khan, Des Rosiers, Rooney, Morgan, & Sweat, 1982), developed a methodology for measuring the particle size index of instant corn flours for making tortillas, correlated with the quality of the product. According to the work of Pflugfelder, Rooney, and Waniska (1988), corn masa cannot be considered to have a homogeneous composition, due to differences in the physicochemical properties because of the particle size distribution. Rather, it must be considered to be a complex, heterogeneous mixture of corn kernel fractions whose reactions and interactions determine the behavior of the masa during the baking and frying processes. Although the differences in the physicochemical and functional properties attributed to different flour particle size distributions have been observed, little information exists about how such

* Corresponding author. Tel.: +52 5557296000x67705; fax: +52 5553954147.
E-mail address: jlfernandez@ipn.mx (J.L. Fernández-Muñoz).

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differences cause modifications in the texture and other characteristics of the product. More recently, Sahai, Buendía, and Jackson (2001) reported that commercial nixtamalized maize flours are a mixture of different-sized particles that contribute different functionality during a single alkaline thermal treatment and steeping time. In order to produce a specific kind of flour recommended for applications involving a particular kind of food, the flour must first be separated by means of sifting into its different particle sizes in order to then make a mixture containing a particular particle size distribution that has the desired functionality for the specific application. This functional change is probably related to differences in the intrinsic characteristics of the polymers that make up the starch of these fractions. In their studies, Aranbula-Villa, Barron-Avila, Gonzalez-Hernández, Moreno-Martinez, and Luna-Barcenas (2001) discovered some physicochemical changes that take place in the maize kernel during the alkaline cooking phase and steeping time which modify the properties of the nixtamalized maize flour and are reflected in better quality tortillas. Fernández-Muñoz et al. (2002) studied the physicochemical and rheological changes in nixtamalized maize flours as a function of steeping times from 0 to 24 h. They found that there are variations in the calcium content and maximum viscosity, as well as fluctuations in the crystallinity and pH value as steeping time increases. Some of the main physicochemical properties associated with the functionality of nixtamalized maize flours are: particle size distribution, pH, water absorption capacity, and the rheology of the masa according (Campus-Baypoli, Rosas-Burgos, Torres-Chavez, Ramírez-Wong, & Serna-Saldivar, 1999). The objective of the present research project was to evaluate the physicochemical, rheological, and structural properties of different particle size distribution fractions of nixtamalized maize flour, produced by a single cooking process and different steeping times from 1 to 24 h. Particle size distribution, calcium content, viscosity and crystallinity were evaluated.

2. Materials and methods

2.1. Preparation of nixtamalized samples

In the initial cooking step of the nixtamalization process, each sample was prepared by cooking 3 kg of whole maize kernels in a suspension of 6 L of water and 60 g of calcium hydroxide (reagent powder, Fermont, Monterrey, NL, Mexico); with obtained calcium hydroxide at 2% of weight in relation of the maize. 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, and 24 h. After steeping each sample, the cooking liquor (nejayote) was drained off and the nixtamal sample were washed twice in water using a 2:1 (v/w) ration by stirring the kernels in the wash water for 2 min. After washing and draining, each sample was ground into corn masa and then dehydrated using a flash type dryer. 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, D.F., Mexico) equipped with a 0.8 mm screen.

2.2. Particle size distribution

For fractionation, corn flour was sieved in a Ro-TAP RXZ9WH349 equipment for 15 min and US standard sieves no. 30, 40, 60, 80, and 100 were used to obtain five fractions. Each fraction trial was performed using 150 g of corn flour, and fraction yields as well as total recovery were calculated. The fractionation process was performed in triplicate. Means with standard deviation are reported (Sahai et al., 2001).

2.3. Atomic absorption spectroscopy

The calcium content of corn flour was obtained by mineralizing the sample using the dry-ashing method 968.08 (AOAC, 1998), following by calcium ion determination with a double-bean atomic absorption spectrometer (Analyst 300, Perkin–Elmer), equipped with a deuterium lamp, background corrector, and hollow cathode lamp, operated with 12 psi of dry air, 70 psi acetylene, 422.7 nm flame, and 10 mA lamp current, 0.7 nm slit width.

2.4. Relative viscosity

The relative viscosities of the water suspensions of the corn flour dough were determined using a pasting viscometer (Rapid Visco Analyser [RVA] Newport Scientific, Narabee, NSW Australia). Dough samples were adjusted to a 14% moisture content, and distilled water was added to keep the total weight of water and sample constant at 28 g. Dry base material (4 g) retained on each mesh (30, 40, 60, 80 and 100 US) was suspended in 24 mL of water. The each sample with a speed of 160 rpm was heated to 50 °C, and then heated to 90 °C at a heating rate of 5.6 °C/min, and held constant at 90 °C for 5 min, and finally cooled to 50 °C at a rate of 5.6 °C/min.

2.5. X-ray diffraction

The analysis was done using the method Rodriguez et al. (1996). The nixtamalized product was ground into a fine powder and passed through No. 30, 40, 60, 80 and 100 US screen. The samples in powder form were then densely packed into an Al frame. The X-ray diffraction patterns of the samples were recorded on a diffractometer (Simens D5000) operating at 35 KV and 15 mA with a Cu Kα radiation of wavelength $\lambda = 1.5406$ Å. Data was collected from 4° to 30° on 2θ scale with steps of 0.05°. The crystalline percentages were calculated by normalizing the integrated diffracted intensity over the 2θ ranges measure in relation to the integrated noncoherent intensity. The noncoherent intensity was obtained by subtraction the sharp diffracted

peaks from the total diffracted intensity. The same procedure was used for all the samples. The spectrum analysis software (Diffract/TA. Socavin VI.2) was used for three measurements for each sample, and average values were reported.

2.6. Statistical analysis

All treatments were performed randomly and the data of particle size distribution, calcium content, peak viscosity and crystallinity values were statistically analyzed using the SAS Systems for Windows software, version 6.12 TS020 (SAS, Statistical Analysis System, Institute Inc., Cary, NC, 1996). Analysis of variance and *F*-tests were used to determine significant differences among the treatment means at $P \leqslant 0.05$. Multiple of mean comparisons were made by least significant difference (LSD).

3. Results and discussion

3.1. Particle size distribution

Fig. 1 shows the percentage particle size distribution (PSD) for each one of the instant nixtamalized maize flours as a function of steeping times from 0 to 24 h. These values represent the average of three measurements per sample. According to the results shown in this figure, we can differentiate two particle size distribution groups. The first PSD group retained in the 30 and 100 US mesh screens varied between 4% and 12%; the second PSD group retained in the 40, 60, and 80 US mesh screens varied from 80% to 90%. The PSD behavior of nixtamalized maize flour is associated with the alkaline thermal process that modifies the components of the grain, obtaining a greater percentage of particles in the 40, 60, and 80 US mesh screens (Group I) and consequently a smaller percentage in the 30 and 100 US mesh screens (Group II). This behavior was not significantly affected ($P \leqslant 0.05$) by steeping time of 6–8 h, but it was observed that with a steeping time of more than 9 h, percentage fluctuations appear in the two PSD fractions, increased that significantly differences ($P \leqslant 0.05$) among this samples. This PSD behavior of nixtamalized maize flours may be related to the inhibition of starch gelatinization, due to the incorporation of calcium into the maize kernel during the nixtamalization process (Bryan & Hamaker, 1997; Robles, Murray, & Paredes-Lopez, 1988). Herrera-Gomez, Canonico-Franco, Ramos, and Pless (2002) indicate that starch granule aggregation is dependent on cooking temperature. Therefore, the PSD of the nixtamalized maize flour is probably related to starch granule aggregation as a consequence of the cooking step of the nixtamalization process. The fluctuations that were observed in both PSD groups are probably related to the steeping time and the alkaline solution in which the maize was steeped.

3.2. Calcium content

Fig. 2 shows the calcium content of the PSD of the nixtamalized maize flours as a function of steeping time. These values represent the average of three measurements per sample, we can observe that the smaller particles (80 and 100 US mesh) contain more calcium content that coarse particle size (30, 40 and 60 US mesh), this result was significant differences with $P \leqslant 0.05$. As we can see in this figure, the calcium content of the different PSD fractions of the flours does not increase in a linear relation to the increase in steeping time. These results are in agreement with the research published previously by Trejo-González, Feria-Morales, and Wild-Altamirano (1982), Morad, Iskander, Rooney, and Earp (1986), Fernández-Muñoz et al. (2002,

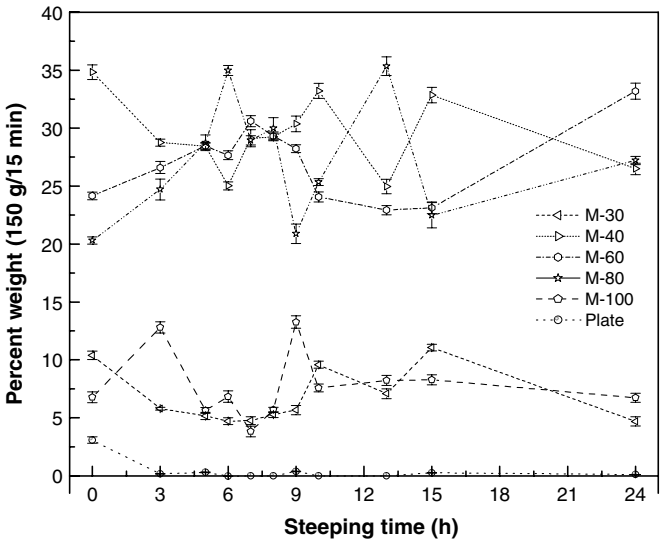

Fig. 1. Percentage particle size distributions of instant nixtamalized maize flour as a function of steeping time.

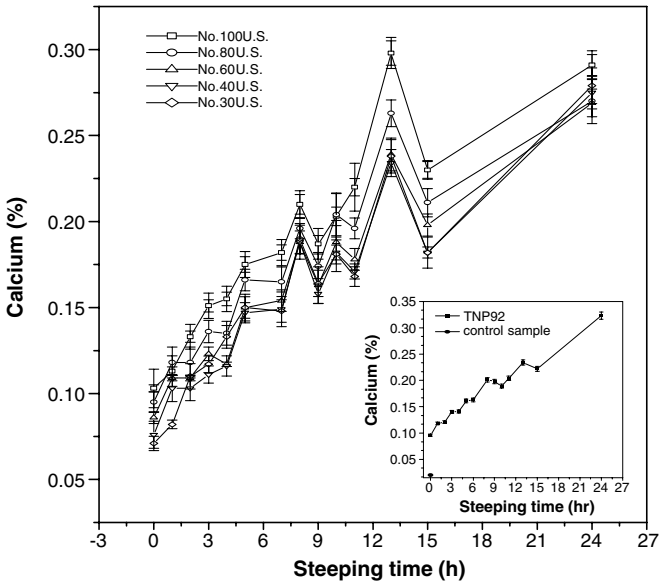

Fig. 2. Calcium content of particle size distributions of instant nixtamalized maize flour as a function of steeping time.

2004), Zazueta et al. (2002), González, Reguera, Mendoza, Figueroa, and Sánchez-Sinencio (2004), which demonstrated that the incorporation of calcium into the maize kernel during the nixtamalization process follows a nonlinear process in proportion to the increase in steeping time. Similarly, in Fig. 2, it can be seen that the smaller particles (100 US mesh) contain more calcium, probably corresponding to the outer layers of endosperm, where there is greater calcium diffusion during the nixtamalization process (González et al., 2004). These parts of the kernel may possibly be more prone to be broken down during the milling process, leading to a PSD with finer particles. These results also are in agreement with (Pflugfelder et al., 1988), who found that nixtamalized maize masa with greater calcium content contained a higher percentage of fine particles. At the same time, it was mentioned that large particles had lower calcium content. Therefore, it may be said that calcium diffusion takes place in a radial manner inside the maize kernel during the nixtamalization process, and that the particle size is smaller in the outer layers of the kernel (corny endosperm), and increases in the innermost part of the kernel (floury endosperm) White and Johnson (2003). We can see a significant jump in the calcium content of the instant flour samples between 4 and 5 h of steeping time, which is related to the permeability of the pericarp to calcium ions (Fernández-Muñoz et al., 2002, 2004). The fluctuation in the calcium content of the PSD samples with more than 8 h steeping time is due to the dissolution of the cell wall and part of the endosperm, which facilitates the loss of pericarp during the washing process (Gomez, McDonough, Rooney, & Waniska, 1989).

3.3. Viscosity of nixtamalized maize flours

Fig. 3 shows the maximum viscosity values of the PSD samples of instant nixtamalized maize flours as a function of steeping time. These values represent the average of three measurements per sample. The results show that maximum viscosity increases in proportion to calcium content for the different steeping times, expressed as an ascending curve as a function of steeping time. That is to say, the calcium content of the different fractions (30, 40, 60, 80, and 100 US) is lower (700–1100 cP) for a steeping time of zero, and maximum viscosity increases with increased calcium content. At 1 h of steeping time, the calcium concentration increases substantially (1100–1650 cP), causing high viscosity values for the different fractions. This tendency is also the case with longer steeping times, and we can observe that greater (1300–1600 cP) viscosity values occur for high concentrations of calcium and steeping times of over 13 h. Also, we can see that the difference viscosity of the samples that were steeped for 8 and 9 h diminished in comparison to that of the flours with other steeping times ($P \leqslant 0.05$), although the calcium concentration did not change substantially in these samples. Maximum viscosity values are related to calcium content. Steeping time is associated with calcium content, preserving the starch granules and promoting aggregations and crosslink connections, resulting in a greater viscosity (San Martín-Martínez, Jaime-Fonseca, Martínez-Bustos, & Martínez-Montes, 2003; Bryan & Hamaker, 1997).

3.4. Crystallinity of nixtamalized maize flours

The relative crystallinity of the flours, processed at different steeping times, can be seen in Fig. 4. Particle size distribution with different calcium content produces fluctuations in crystallinity; we obtained significantly difference with $P \leqslant 0.05$ in the all samples, except for 8 and 9 h of steeping times. These values represent the average of three measurements per sample. At zero steeping time, crystallinity decreases with smaller particle size. For different

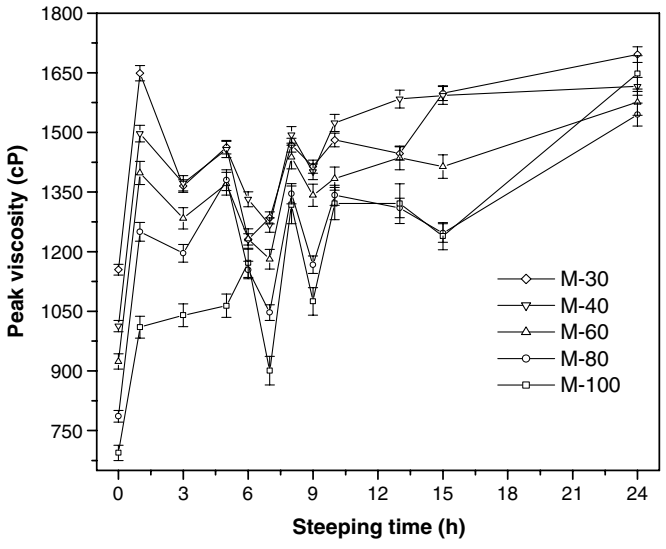

Fig. 3. Peak viscosity of particle size distributions of instant nixtamalized maize flour as a function of steeping time.

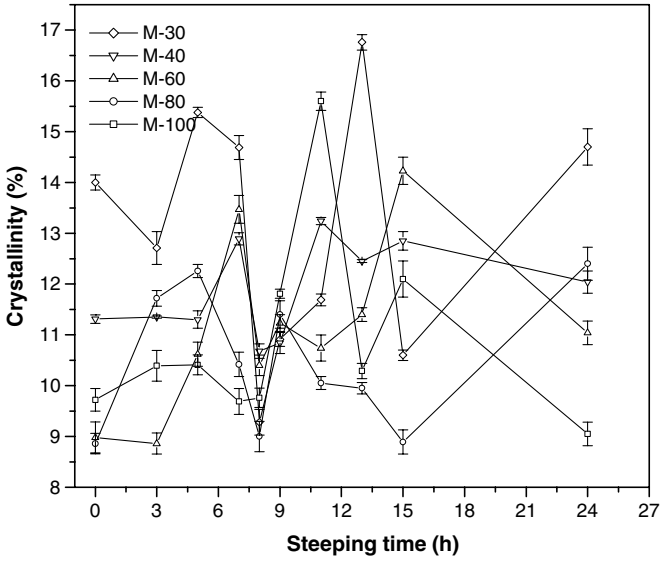

Fig. 4. Crystallinity of particle size distributions of instant nixtamalized maize flour as a function of steeping time.

steeping times, there are fluctuations in the crystallinity values, but in general, the crystallinity of all the samples are in the range from 9% to 14%. The samples with a steeping time of 8 and 9 h display less fluctuation and have crystallinity values between 10.5% and 11.5%. The samples with 8 and 9 h of steeping have a value of 10%. The sample with 8 h of steeping time also has high viscosity values and less fluctuation in its calcium content. Other particle sizes are very similar in terms of their crystallinity behavior. Data from (Rodriguez et al., 1996; Bryan & Hamaker, 1997) indicates that when a concentration of 0.1–0.2% of calcium hydroxide is added starch crystallinity increases. This change in the crystallinity of the starches is caused by the calcium diffusion process in the maize kernel during cooking and steeping, since a recrystallization or annealing of the starches occurs in the steeping stage, as (Gomez et al., 1991; Campus-Baypoli et al., 1999) have mentioned. Therefore, it is important to mention the recrystallization of the starch in the maize kernel, which depends on steeping time in the nixtamalization process. In this case, the similar value was observed in the case of steeping times between 8 and 9 h.

4. Conclusions

In general, we observed that the smallest particles contain a greater percentage of calcium for the different steeping times. It appears that they are fragments of the outer parts of the maize kernel, such as the pericarp or husk, and the outer parts of the endosperm, where greater calcium diffusion takes place. Instant nixtamalized maize flour with a more homogeneous particle size distribution has very similar crystallinity and viscosity. Tortillas produced from these types of flours probability will be more homogeneous in terms of their physical mechanics and sensorial properties. The increase in calcium content in nixtamalized maize flours, and the increase in maximum viscosity, will be affected as the size of the particles diminishes. Therefore, it can be assumed that calcium inhibits the gelatinization process during the nixtamalization process, promoting the aggregations and possible crosslink connections, which increase viscosity.

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