



Evaluation of nixtamalization temperature and adding nejayote solids on textural and color properties of dough and tortillas

Evaluación de la temperatura de nixtamalización y adición de sólidos de nejayote sobre propiedades texturales y de color de masas y tortillas

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Abstract

Nixtamalization is a traditional preparation process in which dried corn kernels are cooked (~ 90 °C) and steeped (up to 12 h) in a lime solution. The kernels are drained and rinsed to remove the outer pericarp cover and milled and kneaded to produce dough, from which several products, among them, tortillas are obtained. Nixtamalization provides adequate texture to tortillas, but it requires a high energy input and releases a considerable volume of waste effluent (called nejayote). This work explored modifying the nixtamalization process temperature (60 °C instead of 90 °C) and the addition of nejayote solids to maize dough, and the effect on the textural and color properties of the dough and tortillas. It was observed that after nixtamalization at 60 °C the textural properties of corn products did not show a significant difference compared to those at 90 °C. Regarding the addition of nejayote solids, the textural and color properties of the dough and tortillas were not modified, neither at 60 nor at 90 °C, obtaining products with the same attributes as the commercial counterparts. This study may contribute to considerable energy savings in the process and to decrease in solids of the waste effluents.

Keywords: corn, dough, nejayote, nixtamalization, tortilla.

Resumen

La nixtamalización es un proceso tradicional en el que los granos de maíz secos se cuecen (~ 90 °C) y se remojan (hasta 12 h) en una solución de cal. Los granos se escurren y enjuagan para quitarles el pericarpio y se muelen y amasan para producir una masa, de la que se obtienen varios productos, entre ellos, las tortillas. La nixtamalización proporciona una textura adecuada a las tortillas, pero requiere un alto aporte de energía y libera un volumen considerable de efluente de desecho (llamado nejayote). Este trabajo exploró la modificación de la temperatura del proceso de nixtamalización (60 °C en lugar de 90 °C) y la adición de sólidos de nejayote a la masa de maíz, y el efecto sobre las propiedades de textura y color de la masa y las tortillas. Se encontró que después de la nixtamalización a 60 °C y la adición de sólidos de nejayote las propiedades texturales y color de la masa y tortillas no se modificaron, obteniendo productos con los mismos atributos que los productos comerciales. Este estudio puede contribuir a un ahorro considerable de energía en el proceso y a la disminución de sólidos de los efluentes residuales.

Palabras clave: maíz, masa, nejayote, nixtamalización, tortilla.

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1 Introduction

Corn-based foods, like tortilla, have been consumed in Mexico and throughout Mesoamerica since ancient times. For the elaboration of these products, corn kernels are subjected to a process called nixtamalization, which comes from the nahuatl word "nextli" which means ashes, and "tamalli" which means corn dough. This process consists in cooking the corn kernels in water with lime ($\text{Ca}(\text{OH})_2$), steeping in this alkaline liquor and subsequent washing to eliminate the cooking liquid. A suitable nixtamalization process achieves the gelatinization of the starch of the kernel obtaining a good performance of the resulting dough during rolling (Enríquez Castro *et al.*, 2018; García-Armenta *et al.*, 2020). According to Liu *et al.* (2019), gelatinization of most starches occurs in an interval between 60 and 80 °C. The loss of the crystalline structure of starch due to the absorption of water also contributes to the gelatinization process (Peña-Reyes *et al.*, 2017). When dough acquires a proper texture, it can slightly adhere to the laminating rollers of the tortilla machine and it can be easily separated, otherwise the dough sticks to the rollers and the machine cannot work freely (Gasca-Mancera and Casas-Alencáster, 2007). The color of the tortillas depends on the variety of corn and the amount of $\text{Ca}(\text{OH})_2$ used in nixtamalization (Manresa-González and Vicente, 2007). The textural characteristics of the dough (hardness, cohesiveness, elasticity and adhesiveness) and of tortillas (rolling force and extensibility), as well as the color, can be measured instrumentally (Enríquez-Castro *et al.*, 2018).

In the traditional process of nixtamalization, corn is first boiled in water in a proportion of 1: 2 to 1: 3, weight: volume, to which 1-3% lime has been added, reaching a pH from 11 to 13. The cooking time fluctuates between 20 and 40 minutes, depending on the varieties of corn. After boiling time, the corn is left to rest for 10 to 12 hours. This process requires a great energy input, due to the high temperatures employed (around 90 °C) and to the large processing times. To reduce the ecological impact of nixtamalization, some authors have proposed some modifications to the traditional method. Peña-Reyes *et al.* (2017) evaluated the hydration of maize and the textural properties of dough and tortillas produced under different nixtamalization conditions, including the cooking at 60 °C, which implies energy savings in the process.

On the other hand, nixtamalization process needs large amounts of water and produces a waste effluent, known as nejayote, which is considered highly polluting due to its alkaline pH (9-12) and its high concentration of organic matter (5-50 g / L) which causes a high demand of oxygen, both biological (8100 mg O_2 / L) and chemical (2500 mg O_2 / L) (Gutiérrez-Urbe *et al.*, 2010; Balderas-López *et al.*, 2018; Valderrama-Bravo *et al.*, 2020). It has been found that approximately 30.4% of nejayote solids are soluble fiber from pericarp tissue, and some other nutrients are lost, including vitamins, fat, protein, some minerals, carotenoids and phenolic compounds. Around 17% of niacin content of corn is lost in this effluent since this vitamin is closely linked to the pericarp (Campechano-Carrera *et al.*, 2011; García-Zamora *et al.*, 2015). The reinsertion of these solids into the corn dough could be an improvement to the process in order to reduce the ecological impact of waste effluent, as long as the quality attributes of the products are not adversely affected.

The objective of this work was to evaluate the effect of two nixtamalization temperatures (60 and 90 °C) and the addition of solids from nejayote on the textural properties and color of dough and tortillas, in comparison their counterparts traditionally produced from a local mills.

2 Materials and methods

2.1 Nixtamalization and dough elaboration

The nixtamalization was carried out according to Peña-Reyes *et al.* (2017) with some modifications. Batches of 1 kg of hybrid white dent corn kernels (cultivar Cargill® Sinaloa variety) were used. The dried kernels were cooked (at 60 or 90 °C) for 20 minutes in 2 L of water with 26 g of $\text{Ca}(\text{OH})_2$ (92% purity, Grupo Calidra, Michoacán, Mexico), then the pot was left to stand for 14 h. For the treatment at 60 °C, the pot was covered with elastomer during the steeping time. After that, the cooked kernels (nixtamal) were washed and ground in a stone mill (MN-80, Casa Matus, Tabasco, Mexico) adding the necessary water to obtain a dough with a water content of 55%. Dough and tortillas prepared by the traditional method at a local nixtamal mill (Iztapalapa, Mexico City) were used as controls.

2.2 Elaboration of dough with nejayote solids

In order to evaluate the effect of the addition of solids, nejayote was collected and set-in decantation cones for one hour. The supernatant was discarded and sedimented solids with a water content of 62.3% and 64.5% at 60 and 90 °C were obtained respectively. Nejayote solids were added during milling of the corn kernels to obtain a dough with a water content of 55%.

2.3 Elaboration of tortillas

Tortillas were made with a manual tortilla machine (González TM-G, Nuevo León, México). For each tortilla, 30.73 ± 0.93 g of dough was used. Tortillas were cooked on a griddle at 260 ± 10 °C for 40 s on one side, then for 30 s on the opposite side, and finally turned over again for 20 s. The resulting tortillas were flat discs with a diameter of 12 ± 0.3 cm and a thickness of 1.5 ± 0.08 mm. Subsequently, they were placed in polyethylene bags and stored at room temperature (25 ± 1 °C).

2.4 Texture analysis

Texture analysis for dough and tortillas were performed on a Brookfield CT3 texturometer. The dough was left to rest in polyethylene bags with airtight closure until they reached room temperature. Then, dough cylinders of 12 mm in diameter and 20 mm in height were obtained, which were left to rest for 15 min in a tightly closed container, to allow the dough to relax after handling. Subsequently, hardness, resilience, elasticity, adhesiveness and cohesiveness were measured as reported by Ruiz-Gutiérrez *et al.* (2012) with some modifications: texture profile analysis (TPA) consisted of two compressions per cycle, using a load cell of 10 kg, and a speed of 5 mm/s. An apparent relative deformation (ARD) of 40% was used. For each treatment ten repetitions were performed and averages were reported.

In tortillas, the extensibility and rollability were evaluated. For the extensibility test, tortilla was cut in a "dog-bone" shape (3.75 cm wide at the ends, 1.5 cm wide at the central part, 8.7 cm long) and a stress test with a TA-DGA probe was used at a speed of 2 mm/s with a tension distance of 4 mm (Peña-Reyes *et al.*, 2017). For the rollability test, a device that has a rotating roller was used, to which the tortilla is fixed; it is joined to the moving arm of the texturometer to turn the roller, causing the tortilla to roll over it. The

equipment was used in the tension mode at a speed of 3 mm/s, and a distance of 50 mm. For each treatment ten repetitions were performed when the tortilla reached room temperature (Suhendro *et al.*, 1999) and averages were reported.

In addition, dough and tortilla, from a traditional nixtamal mill, were tested with the above-mentioned methods to use them as a reference.

2.5 Color analysis

The color in the samples was determined using a colorimeter (NH310 Portable, Shenzhen 3NH Technology Co. Ltd., China). An 8 mm aperture was used and measurements were taken with the illuminant C, to obtain the values for L* (luminosity), a* and b* (chromaticity). The color difference between treatments (with and without solids addition) was also calculated with Equation (1) reported by Sánchez-Madriral *et al.* (2014).

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \quad (1)$$

ΔE = Color difference between sample and sampleSN

ΔL = Luminosity difference ($L_{sample} - L_{sampleSN}$)

Δa = Chromaticity difference a ($a_{sample} - a_{sampleSN}$)

Δb = Chromaticity difference b ($b_{sample} - b_{sampleSN}$)

where "sample" refers to dough or tortilla without the addition of solids from the nejayote at the different temperatures studied. And "sampleSN" refers to dough or tortilla with the addition of solids from the nejayote at the different temperatures studied.

2.6 Moisture determination

The moisture content of nixtamal, dough and tortillas were determined by drying in a convection oven at 130 °C for 1 h according to the AOAC 925.10 method (2005).

2.7 Physico-chemical analysis of nejayote

Nejayote and the solid sediment after its decantation, were physico-chemical analyzed. Carbohydrates according by Makoto *et al.* (2021). Protein by Bradford method (Sebastián-Nicolás *et al.*, 2020) method. Total soluble solid (°Brix) by using hand refractometer. Moisture as described in section 2.6.

2.8 Sensorial analysis

A sensory acceptability test was performed employing a 9-point hedonic scale to evaluate the consumers'

acceptability of tortillas elaborated with the addition of solids nejayote (at 60 °C and 90 °C), and of the commercial product. For this purpose, 100 tests were performed one day after making tortillas. Each panelist was given the three codified samples with a questionnaire. Each tortilla sample was given a different random three-digit number. The samples were given in different orders. The temperature of the samples was 25 °C when given to consumers. Participants were asked to eat the tortilla sample one at a time, and drink water before the evaluation and between samples. For each tortilla, the participants were requested to evaluate the attributes of flavor, texture, and overall acceptability using a 9-point hedonic scale ranging from 1 to 9 with the following meaning: 1="Dislike extremely", 2="Dislike very much," 3="Dislike moderately," 4="Dislike slightly," 5="Neither like nor dislike," 6="Like slightly," 7="Like moderately," 8="Like very much," and 9="Like extremely" (Gómez-Fernández *et al.*, 2021).

2.9 Statistical analysis

For the determination of moisture, protein, carbohydrate and total soluble solid three replicas were performed, and the standard deviations were estimated using the XLstat software (Addinsoft, USA). In the texture tests for dough and tortilla (hardness, cohesiveness, adhesiveness, resilience, elasticity, rollability and extensibility) a One-way analysis of variance was performed, using Statgraphics Centurion XVI software (Statpoint Technologies, Statistical Technologies, Statistical Technologies Inc., USA); Treatment means were obtained from 10 samples and compared using a Fisher test with an $\alpha = 0.05$.

3 Results and discussion

3.1 Effect of nixtamalization temperature

3.1.1 Moisture in nixtamal, dough and tortillas

As can be seen in Figure 1, there was no significant difference ($\alpha = 0.05$) in moisture values of the nixtamal (cooked corn kernels) obtained at the two studied temperatures. These results agree with those reported by Sierra-Macías *et al.* (2010), who evaluated the water content of nixtamal from different corn varieties and found values between 45-49%. Water

content of doughs were adjusted to approximately

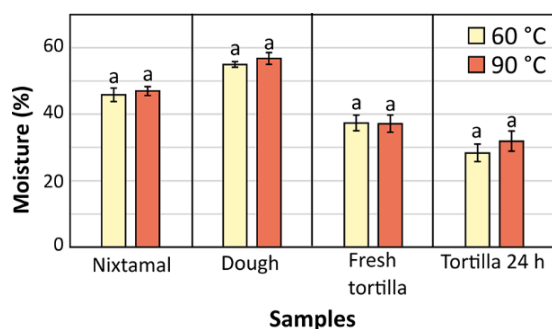


Figure 1. Moisture of nixtamal, dough and tortilla made at different nixtamalization temperatures. Values with the same superscript within each group were statistically equal when applying the Tukey test with a value of $\alpha = 0.05$.

55% as it has been reported for the proper preparation of tortillas (Contreras-Jiménez *et al.*, 2014). In freshly made tortillas there was no significant difference ($\alpha = 0.05$) in moisture, at the two studied temperatures, which was about an 18% lower than that of dough. Salinas-Moreno and Aguilar-Modesto (2010) reported moisture values for freshly made tortillas between 35-40%. Similar results were reported by Vernon-Carter *et al.* (2020). In tortillas stored for 24 h, there was a further significant reduction in moisture that was of about 9 and 6% for nixtamalization temperatures of 60 and 90 °C, respectively. Román-Brito *et al.* (2007) reported a moisture loss of 5% for corn tortillas with three days of storage, that was reflected in a hardening of the tortilla, due to the formation of a rigid structure in the tortilla, product of the loss of water.

3.1.2 Textural properties of dough and tortillas

The effect of the nixtamalization temperature on the texture of the dough is reported in Figure 2, and as can be seen, there was no significant difference ($\alpha = 0.05$) in any of the textural properties evaluated. With these results it can be confirmed that the use of nixtamalization temperatures of 60 or 90 °C, does not make a difference in the texture of the dough, compared with the traditional dough, which is important to make good quality tortillas.

In Figure 3A and 3B the effect of temperature on the textural properties of the tortillas, is shown. As it can be seen there was no significant difference ($\alpha = 0.05$) in the rollability of the freshly made tortillas, at both temperatures studied, but there was a significant difference ($\alpha = 0.05$) on the 24 h aged tortillas.

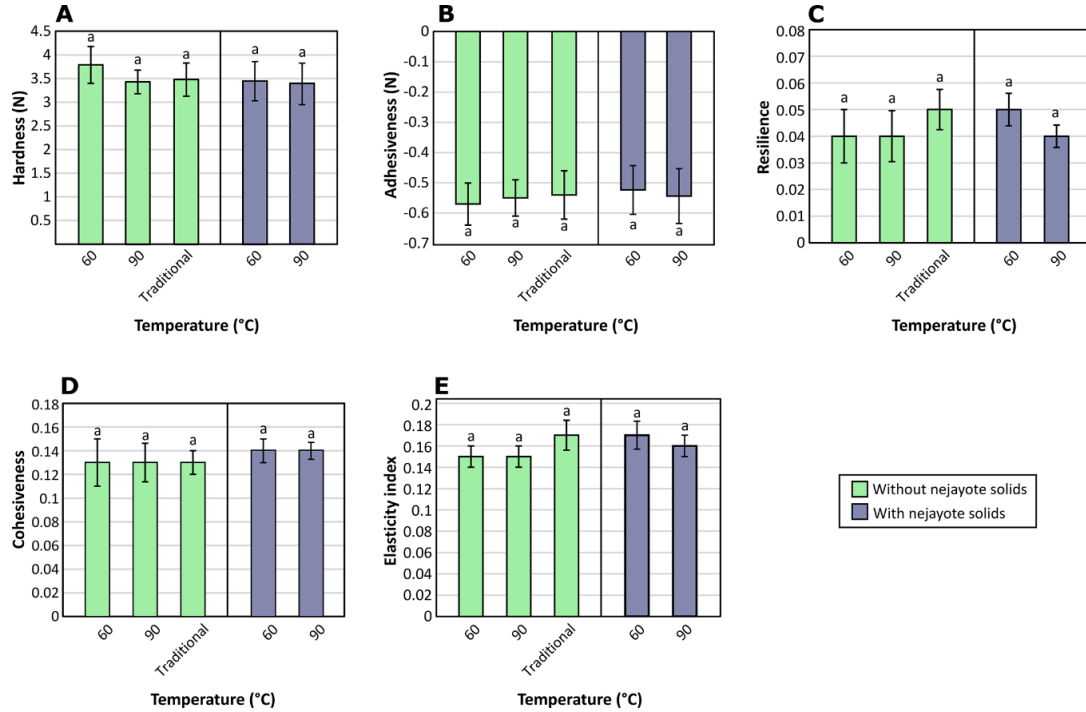


Figure 2. Textural properties of doughs made at different nixtamalization temperatures, without or with nejayote solids. Values with the same superscript within each group were statistically equal when applying the Tukey test with a value of $\alpha = 0.05$.

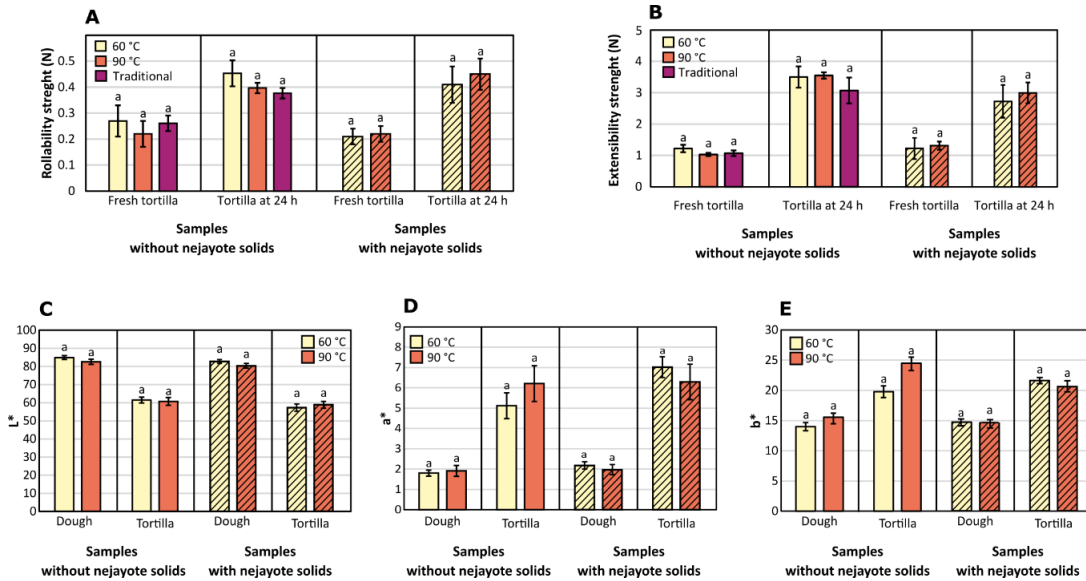


Figure 3. Textural and color properties of tortillas and color of dough and tortillas made at different nixtamalization temperatures, without or with nejayote solids. Rollability (A), Elasticity index (B), L*: luminosity (C), a*: green-red chromaticity (D), b*: blue-yellow chromaticity (E). Values with the same superscript within each group were statistically equal when applying the Tukey test with a value of $\alpha = 0.05$.

Suhendro *et al.* (1999) reported rollability values for fresh tortillas from 0.20 to 0.37 N, which upon aging for 24 h required twice the strength for achieving the same rollability. These results agree with our findings. This phenomenon is more evident in tortillas made with nixtamalized corn at 60 °C (with lower water content, prone to a greater retrogradation), acquiring a rigid form (Román-Brito *et al.*, 2007). Regarding the extensibility, it can be observed that there was no significant difference ($\alpha = 0.05$) between the freshly made tortillas and between the 24 h aged tortillas at both temperatures. However, the extensibility of the latter was halved, due to the hardening effect caused by the higher retrogradation suffered due to loss of water, becoming less flexible (Román-Brito *et al.*, 2007). The results obtained, coincide with those reported by Salinas-Moreno *et al.* (2011) since they observed the same trend in the increase of tensile strength of tortillas stored for 24 h. These results suggest that starch gelatinization reached at 60 °C is enough to obtain a dough with a suitable texture for making tortillas.

3.1.3 Color analysis of dough and tortillas

In Figure 3C to 3E, the color analysis variables (L^* , a^* and b^*) of the dough and tortillas are reported. It can be seen that there were no significant differences ($\alpha = 0.05$) in luminosity, as well as in chromaticity, both in dough and in tortillas elaborated with corn nixtamalized at different temperatures. We observe that the luminosity (L^*) and the chromaticity (b^*) values are similar to those reported by Ruiz-Gutiérrez *et al.* (2012); however, for chromaticity (a^*) differs with the value they report (-0.545) and the difference can be attributed to the different nixtamalization conditions employed such as temperature (80 °C), stirring and cooking time (60 min). This indicates that their samples have a shade that is in the range of green, while in this study the shade is in the range of reds. Sánchez-Madrigal *et al.* (2014) report similar results in variables L^* and b^* but in variable a^* the values are negative, which indicates a hue in the range of green and which can be attributed to the type of corn used in that investigation.

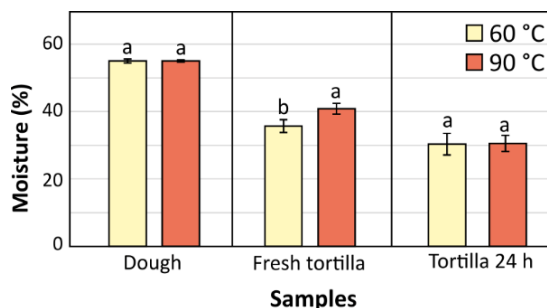


Figure 4. Moisture of dough and tortilla made at different nixtamalization temperatures, with nejayote solids. Values with the same superscript within each group were statistically equal when applying the Tukey test with a value of $\alpha = 0.05$.

3.2 Effect of adding nejayote solids in dough and tortilla

3.2.1 Dough and tortillas moisture

In Figure 4, it can be seen the effect of the addition of the nejayote solids on the moisture of dough and tortillas. Dough moisture was standardized to approximately 55% with the addition of solids from nejayote. In the freshly made tortillas, those made with the 60 °C nixtamal had a significant ($\alpha = 0.05$) lower moisture content (of about 20% less) than those made with 90 °C nixtamal. No significant differences were found in moisture content for the 24 h aged tortillas made from 60 and 90 °C nixtamal, but both exhibited lower moisture than the fresh tortillas. On the other hand, comparing the moisture of the tortillas with and without the addition of nejayote solids (Figure 1 and 4), it can be observed that for the tortillas stored 24 h there are no significant changes ($\alpha = 0.05$); while for freshly made tortillas (90 °C) it was observed that the addition of solids exerts a positive effect by helping to retain water.

3.2.2 Textural properties of dough and tortillas

In Figure 2 we can see the effect of the addition of nejayote solids on the dough texture at both temperatures of nixtamalization studied. In the same way as in the dough without addition of solids, there was no significant difference ($\alpha = 0.05$) in any of the textural properties studied.

Table 1. Physicochemical composition of nejayote and solid sediment after its decantation from nixtamalization at 60 °C.

Sample	pH	Total soluble solids (°Brix)	Carbohydrates (g/L)	Protein (g/L)
Nejayote	11.86 ±0.01	1.47 ±0.10	63.43 ±0.28	11.92 ±0.08
Solid sediment	12.61 ±0.01	18.24 ±0.05	318.60 ±0.35	49.51 ±0.04

Thus, the texture of the dough was not modified by adding the nejayote solids. Valderrama-Bravo *et al.* (2015) studied the textural properties of the dough with the addition of different percentages of nejayote solids, reporting that the cohesiveness, resilience and elasticity did not show differences; however, they report a difference in hardness, that they attributed to the calcium carbonate present in the nejayote.

Figure 3A and 3B report the textural properties of tortillas made with the addition of solids. As it can be seen, there was no significant difference ($\alpha = 0.05$) in the rollability and in the extensibility in both freshly made tortillas and tortillas stored for 24 h at the two nixtamalization temperatures. Comparing the results obtained for the tortillas made with and without the addition of solids we can see that there was no significant difference ($\alpha = 0.05$), so we can say that the addition of solids does not affect the textural properties evaluated. The results obtained coincide with those reported by Valderrama-Bravo *et al.* (2020), who evaluated the impact of the agronomic management of maize on the textural properties in freshly prepared tortillas.

3.2.3 Color analysis of dough and tortillas

In Figure 3C to 3E, the color analysis variables of the dough and tortillas made with addition of nejayote solids are reported. There was no significant difference ($\alpha = 0.05$) in the three variables studied (L *, a * and b *) both in dough and in tortillas elaborated with corn nixtamalized at both studied temperatures. When comparing with the values of the samples without addition of nejayote solids, no significant differences were observed ($\alpha = 0.05$), so we can say that the addition of solids does not affect the color of the products obtained.

The color difference (ΔE) between the treatments with and without the addition of solids from the nejayote, was calculated. For dough, ΔE at 60 and 90 °C were 2.3 and 2.5 respectively, meanwhile for tortilla, ΔE at 60 and 90 °C were 4.9 and 4.6 respectively, a more noticeable color difference compared to dough. However, both in dough and in

tortillas, color difference complies with acceptance standards (ISO 12647-2), which indicates that even though the addition of solids causes variation in color, this is acceptable, being considered for dough, as a normal variation, and in the case of tortillas, the quality is sufficient to achieve acceptance in color as reported by Manresa-González and Vicente (2007). This variation can be explained by the presence of calcium hydroxide, the carotenoid and flavonoid pigments present in the nejayote, which give it a yellow coloration.

Also, the nutritional value of the product may be enhanced. As reported by Acosta-Estrada *et al.* (2014), the incorporation of solids from nejayote into the product increases the content of fiber, calcium and phenolic compounds. For example, the addition of 9% nejayote solids increased the dietary fiber up to 54% in bread. Moreover, enriched bread contained about 745 times freer ferulic acid and increased approximately 70% of their antioxidant capacity, and provided 15% of the recommended calcium intake.

3.3 Sensory analysis

The sensory acceptability values of tortillas elaborated with nixtamalized corn at 60 and 90 °C and with the addition of solids from nejayote are reported in Table 2. As can be seen, the solids nejayote addition did not affect any of the parameters evaluated of the tortillas. This means that the addition of solids did not modify the acceptance of the consumer for the product. These results are agreed with the obtained the textural properties analysis reported in section 3.2.2. Moreover, the results obtained are similar with those reported by Valderrama-Bravo *et al.* (2020), who evaluated the textural properties in tortillas.

3.4 Physico-chemical composition of nejayote

The physico-chemical composition of nejayote and solid sediment are reported in Table 1. The pH value of nejayote agrees with the reported by Valderrama-Bravo *et al.* (2015).

Table 2. Sensory acceptability values of tortillas added with solids nejayote.

Parameter	Tortilla (60 °C)*	Tortilla (90 °C)*	Commercial Tortilla
Flavor	7.62 ±0.16	7.63 ±0.14	7.75 ±0.12
Texture	7.8 ±0.11	7.8 ±0.12	7.9 ±0.14
Overall acceptability	7.66 ±0.11	7.70 ±0.10	7.77 ±0.12

*Nixtamalization temperatures

The total soluble solids are in accordance with that informed by Castro-Muñoz and Yáñez-Fernández (2015). Carbohydrates and protein content agree with that communicated by Niño-Medina *et al.* (2009). Additionally, in the case of solid sediment, all parameters were higher than the nejayote as a result of the decanted process. Vacio-Muro *et al.* (2020) reported that 50% of the nejayote solids are suspended and these contained about 64% nonstarch polysaccharides, 20% of starch and 1.4% of protein. The reincorporation of solids from nejayote for the elaboration of corn dough, replaces the addition of water during the milling of the grain, which implies a direct saving in the consumption of water. In addition, the residual liquid from the decantation can be used to wash the corn kernels at the beginning of the nixtamalization. With these changes in the process, significant savings can be obtained, which would result in a more environmentally friendly nixtamalization proposal.

Conclusions

The nixtamalization temperature did not affect the textural properties of the dough and tortillas, managing to obtain at 60 °C, products with characteristics like commercial products obtained at higher temperatures (90 °C). Regarding the addition of solids from the nejayote, it was observed that textural properties of dough and tortillas were not modified, obtaining products with the same characteristics as commercial products. Color variables (L^* , a^* , and b^*) were also unchanged. With these results, a comprehensive process of dough and tortilla production can be proposed, in which the nejayote solids would be recovered, reducing the contamination that this effluent causes and fortified dough and tortillas with calcium and fiber would be obtained. In addition, the use of a nixtamalization temperature of 60 °C

would help to reduce energy costs in the traditional nixtamalization process.

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