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The Physicochemical, Rheological And Morphological Properties Of Products Obtained From Traditional Nixtamalization-Extrusion Methods And Their Influence On Starch.

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Abstract: Nixtamalization is an ancient method of processing corn that removes its husk or skin, improving nutritional value and protein quality. The objective of this study was to compare the physicochemical, rheological and morphological characteristics of corn flour without and nixtamalized, products such as dough and tortillas from the traditional nixtamalization process (TNP) and the extrusion nixtamalization process (ENP) and their relationship with starch. To compare the effects of the traditional nixtamalization and extrusion processes on the same variety of corn, samples of ground corn, nixtamalized flour, dough and tortillas were obtained. The extrusion process produced corn flour with smaller particle sizes (particle size index, PSI = 51) than the flour produced by the traditional nixtamalization process (PSI = 44). The PNT mass showed higher values of elastic modulus (G') and viscosity (G") than the ENP mass. Furthermore, in a temperature scan test, the PNT mass showed a peak at G' and G", while the ENP mass did not show these peaks. The tortillas produced by ENP obtained higher contents of resistant starch and firmness and rolling capacity comparable to those of TNP, but lower quality parameter values. Comparison of the physicochemical properties of the products produced by the two methods shows the quality of recording starch damage during milling and extrusion methods to produce higher quality tortillas. For the first time, the measurement of the viscoelastic parameters G' and G" in temperature sweep mode is proposed to monitor changes in the degree of starch damage.
Keywords: Corn, nixtamalization, extrusion, flour, dough, tortillas, starch.

1 INTRODUCTION

The processing of corn by nixtamalization has allowed the development of traditional products, such as tortillas and other innovative products with high consumer acceptance. Currently, through this process, corn is cooked in a Ca(OH)2 solution, then soaked, washed, and ground into flour, dough, tortillas, and other widely accepted products.(Tejeda, 2021)(Galindo, 2021)

During traditional nixtamalization, in addition to structural changes in the grain, there are modifications in the rheological, functional and textural properties that determine the acceptability of the final product. Among the most important structural changes is the gelatinization of starch, which is affected by factors such as temperature, cooking times, and wet milling operations. In the traditional nixtamalization process (TNP), wet grinding is used to highlight two factors: separation of starch granules by excess water and reduction of damaged starch content. These factors make it possible to gradually gelatinize the starch, improving the viscoelastic behavior of the dough obtained, as well as the flexibility, rollability, firmness, structural uniformity, color and shelf life of the tortillas; Sensory attributes are enhanced and appreciated by the consumer. The lack of control of cooking conditions, grain moisture content, and grinding particle size in TNP influence the quality of nixtamalized products. Despite the benefits of traditional nixtamalization, this process involves high energy and water consumption, which leads to environmental pollution. That is why alternative methods, such as extrusion cooking, have been studied to obtain flour, dough and tortillas. (Arriola, 2020) (López, 2020)(Morales F. , 2023)(Cuevas, 2023)(Olaechea, 2020)(Palacios, 2023)

Several research studies have been dedicated to the process of nixtamalization by extrusion (ENP) to obtain nixtamalized corn flours, considering certain conditions of the process such as: temperature, moisture content, percentage of calcium hydroxide, screw speed, among the main ones. Likewise, the changes in the chemical properties of flour, dough and tortilla have been evaluated, obtaining good approximations to the traditional product. However, adapting the ENP to obtain quality nixtamalized products entails technological limitations due to costs to control moisture and % (CaO) at low levels that directly affect the behavior of corn starch, due to an exhaustive mechanical force applied several times; which causes higher contents of damaged starch.(Zagaceta, 2020)(Morales J. , 2023)

During the extrusion-cooking process that is applied in the ENP, considerable damage occurs to the starch granules, causing a certain degree of dextrinization. Thus, the viscoelastic properties are affected, being lower than those observed in the

NPT, negatively influencing the quality of the dough and tortillas obtained in the ENP. Despite these disadvantages of extruded nixtamalized flours, it has been claimed that tortillas are comparable in quality to those made with the NPT, although texture and shelf life issues still exist. Currently, improvements have focused on decreasing the firmness and increasing the rollability of tortillas.(Váquez, 2020)(García A. , Actividad antihipertensiva de péptidos de zeína extraídos de maíz (Zea mays L.) criollo (azul y rojo) del Estado de México, 2020)(Hernández, 2023)

The structural properties in flours, doughs and tortillas are related to the changes that occur in starch, influencing quality, therefore, it is important to determine which operations are critical in the NPT with respect to the NPT. This has led to the identification of the extrusion conditions needed to reduce starch damage to a level comparable to that of the NPT.(Cuevas, 2023)(Galindo, 2021)

The objective of this study is to evaluate and compare the physicochemical, rheological and morphological characteristics of products from the traditional nixtamalization and extrusion processes and their relationship with changes in corn starch granules to improve tortillas obtained with extruded flour.(Amores, 2022)

2 FUNDAMENTALS OF NIXTAMALIZATION

2.1 Raw Material)

Maize (Zea mays) is the main component used in the nixtamalization process. According to , it states that commercial hybrids of white maize (Zea mays L.) H-430 and H-431; This grain is resistant to high temperatures (up to 40°C) in the northwest area of Mexico, which was obtained in a local market in Hermosillo, Sonora, Mexico. Using a vibrating cleaner (Clipper, Model V230, Clipper Products, Bluffton, IN, USA), they cleaned the corn kernels; determining the chemical composition of the corn kernel by the methods internationally approved by the American Association of Cereal Chemists (AACC), obtained the following results: 11.2% moisture content (44 - 15.02), 71.7% total starch content (dry basis) (76 - 13.01); 4.88% ethereal extract (dry basis) (30 - 20.01); 1.14% ash content (dry basis) (08 - 01.01); pH = 6.5 (02 - 52.01) and 9.17% protein content (dry basis) using the AOAC 990.03 method.(Campos, 2023)(García R. , 2022)(Montoya, 2020)

2.2 Extrusion Nixtamalization (ENP) Process

2.2.1 Milling

Grinding is an operation that is performed according to the procedure used by , and . It relates the particle size index to the effect of starch damage during milling. indicates that, they individually ground three 2 kg maize samples with different meshes (0.5 mm, 0.8 mm and 1.0 mm) in a hammer mill (Model FT2, Armfield Limited, Bridge House, West Street Ringwood, England). They prepared a compound mixture of ground corn by mixing 0.45 g/g of ground corn with 0.5 mm mesh, 0.40 g/g with 0.8 mm mesh, and 0.15 g/g with 1.0 mm mesh. In the mixture there was an appropriate change (damage) in the starch granules to obtain a specific particle size for the maize flour subjected to the extrusion process, this change is due to the manipulation of the process factors in the extrusion experiment, including the type of mesh they used, to grind the grain, the moisture content of the feed material, the compression ratio, and the screw speed. (Curl, 2024) (Cuevas, 2023)(López, 2020)(Mier, 2022)(Huchin, 2021)

2.2.2 Conditioning

The extrusion process is done by grinding the whole corn kernel and conditioning it with lime and water before entering the extruder. This step helps water and lime diffuse into the internal structures of the corn kernel. According to , the ground corn mixture was supplemented with 0.3 g of lime/100 g of flour (Nixtacal Calhidra de Sonora; Hermosillo, Mexico) and distilled water, adjusting it to 25 g water/100 g flour; mixing for 3 minutes in a horizontal mixer (Hobart model AS200; Troy, OH) to be stored in a refrigerator for 12 hours at 5°C.(Aguilar, 2022)(Magaña, 2020)(Magaña, 2020)

2.2.3 Extrusion

To obtain extruded nixtamalized corn flour (ENCF), they performed the extrusion experiment according to the procedure reported by ; using a single-screw laboratory extruder (Model E 19/25 D, OHG Duisburg, Germany), but under different conditions. They used a screw diameter of 19 mm, with a length-to-diameter ratio of 20:1 and a nominal compression ratio of 1:1, and a die opening of 3.0 mm with four heating and cooling zones (1300 W each); The feeding speed of the conditioned sample was established, which was 45 rpm. The screw has a speed of 145 rpm, with an average residence time of 13 min and an operating pressure of 5.68 atm (83.6 psi). Extrusion temperatures were 60°C, 70°C, 80°C and 90°C in the first, second, third and fourth heating/cooling zones, respectively. The extruded ones were collected in aluminum trays for subsequent drying.(Cancino, 2021)(García A. , Actividad antihipertensiva de péptidos de zeína extraídos de maíz (Zea mays L.) criollo (azul y rojo) del Estado de México, 2020)(Jiménez, 2021)

2.2.4 Extrusion

According to the procedure reported by , they dried the extruded ones at 60°C for 1 h, they used a custom-made tunnel dryer (unbranded). The main objective of using the drying process was to reduce the moisture content in the extruded products coming out of the extruder. They decreased the humidity value from 18% to 11%. Next, the dry extrudes were hammer ground, started using a 0.8 mm mesh and then ground again by the mill with a 0.5 mm mesh so that they get the extruded nixtamalized corn meal.(Mejía, 2022)(Acosta, 2023)

2.2.5 Preparation of tortillas

They made the tortilla in different ways depending on the nixtamalization process. In both nixtamalization processes, they prepared the tortillas in a commercial plant (Tortillería Pimentel, Hermosillo Sonora, Mexico).(García R. , 2022)

For the ENP, once they obtained the extruded nixtamalized flour (ENCF), they prepared the tortillas according to the procedure reported by . They mixed a 2.5 kg sample of ENCF with water (2500 ml) in a horizontal mixer (Manufacturas Lenin Model 25, San Luis Potosi, Mexico) for 3 min to form 5.0 kg of corn dough as reported by . They obtained a minimal amount of dough in the mixer (according to the operator) which was enough to be made, run and processed on the roller of the tortilla maker. They wrapped the dough in a plastic bag and let it rest for 20 minutes before processing. They formed dough discs (25 g) on a tortilla forming machine (Lenin Manufactures, Model MLR 30, San Luis Potosi, Mexico) and baked for 56 s in a three-step oven at temperatures of 221°C ± 10°C; 248°C ± 10°C and 280°C ± 10°C. The corn tortillas were cooled and transported to the laboratory, where they were stored at room temperature (25°C) for later analysis.(Hernández, 2023)(Rizo, 2024)(Escobedo, 2023)

2.3 Traditional Nixtamalization Process (TNP)

2.3.1 Cooking & Soaking

(Ramírez, 2021) He mentions that samples of maize kernels (3 kg) were cooked with water (1:3) and 1 g lime/100 g, boiled the grain at (96°C) for 20 min. They let the cooked corn macerate for 14 hours, and then separated the cooking liquor (nejayote); They washed cooked grains (nixtamal) with tap water to remove excess calcium and dissolved solids (Dominguez, 2020)

2.3.2 Grinding & Mixing

They ground nixtamal to obtain fresh corn dough in a 1HP, 5" diameter spiral volcanic stone mill (Maquinaria del Río SA de CV, Michoacán, Mexico). They subjectively added water to the fresh dough and mixed for 3 minutes so that they get a proper dough consistency. Some of the fresh dough was freeze-dried and ground to obtain nixtamalized corn flour (FNC). The other part of the masa (fresh dough) was used to make tortillas.(Rojas, 2020)(Song, 2023)

2.3.3 Preparation of tortillas

They made the corn tortillas of the Natural Tortilleria Product (PNT) with fresh masa in the same commercial plant described with the same processing conditions previously described for the ENP. Then, they cooled the tortillas and transported them to the laboratory which were stored at room temperature (25°C) for further analysis.(Carrillo, 2023)(Mejía, 2022)

2.4 Analytical Evaluations

The moisture content, pH, and resistant starch (RS) content of maize flour, dough, and tortilla were measured in both processes following AACC approved international methods 44–19.01, 02–52.01, 32–40.0. The content of RS (Megazyme, K-RSTAR 08/15) reported as RS/100 g g. All determinations were made in triplicate.(Acosta, 2023)(Hernández, 2023)

2.4.1 Scanning electron microscopy (SEM)

They evaluated the morphology of the different samples according to the procedure reported by , below: they included photographs of the samples of the ground corn kernels, flour, masa and finally the tortilla of both nixtamalization methods with the help of a scanning electron microscope (JSM-5800LV, JEOL, Akishima, Japan) and exposed an acceleration rate of 10 kilovolts. They fixed samples with particle sizes <0.15 mm and a moisture content of 1% and coated with a layer of gold in a vacuum evaporator (Denton Desk II) at a pressure of 7.03 × 10 –2 kg/cm2 using a 1000X magnification.(Orwa, 2023)(Escobedo, 2023)

2.5 Analytical Evaluations

2.5.1 Particle Size Distribution (PSD)

The PSD determination of the fresh dough of the SOP and the extruded nixtamalized corn flour of the ENP was carried out on a set of sieves, but on a wet basis. This is due to a sample of flour (ENCF) and a semi-solid sample (fresh dough). In this case, they used the mass fractionation technique reported by . Briefly, for the NPT, they gently mixed 50 g of fresh dough with 100 ml of distilled water in a flask. They suspended and washed with water through standard US No. 20 (850 µm), 30 (600 µm), 40 (425 µm), 60 (250 µm), 80 (180 µm) and bottom sieves. They performed fraction separation in a vibrating stirrer (AS 200, Retsch GmbH, Haan, Mettmann; Germany) for 5 min. They placed each sieve fraction on an aluminum weighing plate and dried in an oven at 110°C for 24 h. They calculated the percentage of material retained in each sieve.(Preciado, 2022)(Campos, 2023)(Galindo, 2021)

Regarding the ENP, water was added to a sample of extruded nixtamalized corn flour to make dough with a moisture content to prepare tortillas (53.1%). Then they took 50 g of dough and performed the fractionation of the dough in the same way as it is done to fractionate the fresh dough of the SOP.(Tejeda, 2021)

2.5.2 Particle Size Index (PSI)

They calculated the PSI of the methodology reported by and performed in triplicate using the following formula:(Abou, 2022)

$$PSI = \sum \left(\left(\#SF_i\right) \left(\%RM_i\right) + \cdots + \left(\#SF_n\right) \left(\%RM_n\right) \right),$$

where #SF is the number of the sieve factor and is the material retained in each sieve.

The number of the factor depends on the number of the U.S. sieve (0.2, sieve No. 20; 0.3, sieve No. 30; 0.4, sieve No. 40; 0.6, sieve No. 60; 0.8, sieve No. 80; and 1.0, bottom).(Arriola, 2020)

2.5.3 Water Absorption Capacity (WAC)

They calculated the WAC according to the criteria of and reported as ml of water/100 g of flour and in triplicate.(Cabral, 2024)

2.5.4 Water Absorption Index (WAI)

They calculated the WAI from both maize flours according to the procedure of , with slight modifications as follows: in a 1 g sample (previously freeze-dried and ground) they mixed 15 ml of distilled water in a 50 ml centrifuge tube at 25 °C. They shook the suspension for 30 min and centrifuged at 5000 rpm for 30 min. They placed the supernatant on a tarred aluminum plate and evaporated it in a convection oven at 105°C for 12 h. They recorded gel weight, as well as precipitate, and reported WAI as g gel/g dry sample and in triplicate.(Roldán, 2020)(Rojas, 2020)(García A. , Actividad antihipertensiva de péptidos de zeína extraídos de maíz (Zea mays L.) criollo (azul y rojo) del Estado de México, 2020)

2.5.5 Water Solubility Index (WSI)

They determined the WSI using the methodology reported by ,in triplicate using the formula:(Rashwan, 2021)

$$WSI = \left(\frac{WSMS}{ISW} \right) * 100,$$

WSMS is the weight of the soluble material in the supernatant and ISW is the initial weight of the sample.

2.6 Rheological Mass Evaluations

2.6.1 Frequency Sweep Test

They performed the dynamic frequency sweep tests to measure the viscoelastic modulus of the mass of both nixtamalization processes (Rheometrics Model RSF III, Piscataway, NJ, USA). The elastic (G') and viscous (G") moduli of the mass of both processes were obtained according to the procedure reported by . Briefly, they placed a sample (3.0 g) between two parallel plates with a diameter of 25 mm and a gap of 2.5 mm. Part of the dough was exposed to the environment where it was coated with petroleum jelly to prevent moisture loss. They ran the frequency sweep test using a strain of 0.04% (linear viscoelastic region) at 25°C and a frequency range between 0.1 and 100 rad/s. G'G."(Olšaníková, 2022)(Tejeda, 2021)

2.6.2 Temperature Sweep Test

Dynamic temperature scanning tests were performed to evaluate changes in the viscoelastic properties of the mass of both nixtamalization processes (Rheometrics Model RSF III, Piscataway, NJ, USA). The elastic modulus (G') and the viscous modulus (G") of the mass of both processes obtained according to the procedure reported by . Briefly, they placed a sample (3.0 g) between two parallel plates with a diameter of 25 mm and a gap of 2.5 mm. They exposed some of the dough to the environment, after which they placed silicone oil (Sigma Aldrich, UK) on the edges of the dough to help prevent moisture loss. They ran the temperature sweep test in the temperature range of 25°C to 120°C with a frequency of 5 rad/s and a strain of 0.04% (linear viscoelastic region). Viscoelastic parameters determined the modulus and G'G" G'G".(Magaña, 2020)(Martinez, 2023)(Mejía, 2022)

2.7 Tortilla Evaluations

2.7.1 Physical Properties

They sampled ten tortillas from each process to assess weight (g), diameter (cm) and thickness (mm). They measured the weight of the tortillas on an analytical balance (Sartorius Research R300S), while they evaluated the diameter and thickness with a digital Vernier (Model CD6"C Mitutoyo Corporation, Kanawa, Japan). They reported the mean values and standard deviation of each physical property.(Maldonado, 2021)(Olaechea, 2020)

2.7.2 Firmness

They evaluated firmness and rollability at 2, 24 and 48 h of storage at room temperature, using a procedure suggested by the cornmeal industry, which indicates that the texture of a tortilla evaluated as a hot tortilla rather than room temperature (personal communication). They preheated the tortillas as follows: they placed the individual pieces of tortilla in a poly bag and then heated in a microwave oven (Samsung Co., Mexico) for 15 s at 100% electric power. They cut immediately after heating the tortilla into a 41.47 cm2 rectangle and placed on the bottom plate of the Kramer cell (part code HDP/K55 Stable Micro Systems, Surrey, England). They assessed the temperature of the tortilla reaching 30°C, cut the tortilla piece with a five-blade Kramer cell (top), and then connected to a texture analyzer (TA-XT-Plus Stable Micro Systems, Surrey, England). The spindle speed was set at 2 mm/s and the firmness was reported in kPa. (Aguilar, 2022) (Ramírez, 2021)(Cancino, 2021)(Song, 2023)

2.7.3 Rollability

The subjective method reported by , used to assign a nominal score for the measurement of tortilla rollability. A score of 5 indicates no tortilla breakage (best rolling ability), a score of 3 reflects 50% breakage in the tortilla structure, and a score of 1 represents 100% tortilla breakage. They prepared the tortilla which was identical to the reported procedure for assessing the firmness of the tortilla. They measured rollability at 2, 24 and 48 h of storage.(Ungureanu, 2022)(Morales J. , 2023)

2.8 Experimental design and statistical analysis.

They used a completely randomized experiment, where the factor was the type of nixtamalization process (TNP or ENP) applied to each product (corn flour, dough, and tortilla). They performed an analysis of variance (ANOVA) on all the data they collected from the different evaluations using a significance level of 95%. Evaluated to significant differences between the specific means, they performed the Tukey test with 95% significance. They performed ANOVA analysis using statistical analysis software.(Rodríguez, 2023)(Gastelum, 2023)

3 Fundamentals of Nixtamalization

3.1 Raw material

Table 1 shows the physicochemical properties of flours for the extrusion nixtamalization process and traditional nixtamalization processes. He mentions that both procedures are products with different characteristics. According to the company, TNP produced dough with a moisture content of 53.1% (g/100 g of flour) and also produced ENP in corn flour with a moisture content of 8.8% (g/100 g of flour). They performed the same assessments, the moisture content of the mass and decreased to a final value of 3.4%. According to this behavior, ANOVA shows very significant differences ($p<0.01$) between the moisture contents of both types of flour.(Arriola, 2020)(Cancino, 2021)(Tejeda, 2021)

Table 1

3.6 ± 0.06b

Características	PEV	PNT

pH	8,2 1 ± 0,11a 2	8,4 ± 0,06b
Contenido de Humedad (%)	8,2 1 ± 0,11a 2	8,4 ± 0,06b
Índice de tamaño de partícula	8,83 ± 0,39[a]	3,42 ± 0,06b
WAC^3 (mL agua/100g harina)	51,01 ± 0,57[a]	48,7 ± 0,32b
WAI^4 (g gel/g materia seca)	104,2 ± 0,29[a]	108 ± 0,5b
WSI^5 (%)	3,7 ± 0,08[a]	3,6 ± 0,06b
Almidón resistente (g/100g de muestra	5,8 ± 0,01[a]	3,6 ± 0,06b

Note: 1 means standard deviation. 2 Averages with the same font are not significantly different (p>0.05). 3 WAC Water Absorption Capacity. 4 WAI Water Absorption Index. 5 WSI water solubility index.

They determined that the pH of the corn flours of both processes resulted in an alkaline pH, which was slight, but significantly (p<0.05) higher in TNP (Table 1). This pH is desirable in tortillas due to the traditional alkaline flavor identified by consumers (Cabral, 2024)

Resistant starch content values were higher (p<0.05) for extruded flour (Table 1) than for lyophilized nixtamalized flour. As a result, the highest amount of fiber contained in whole wheat cornmeal comes from dry milling. The reduced water content, the temperature inside the extruder, and subsequent periods of heating and cooling prevented complete gelatinization. Studying macromolecules such as amylopectin partially alter and short linear chains appear, which promotes low enzymatic digestion of starch(Preciado, 2022) (Magaña, 2020)

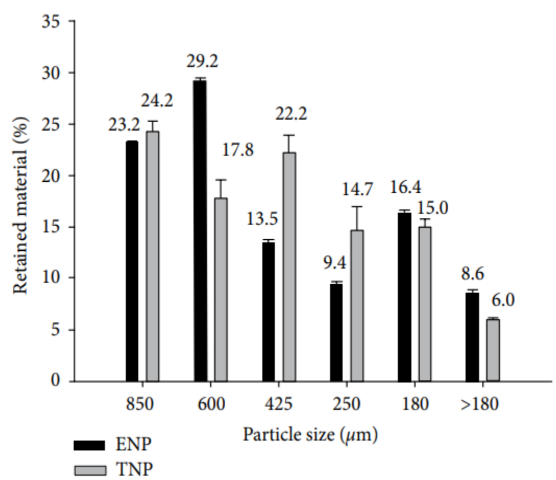
They observed a stronger interaction between starch and calcium in the thermoalkaline process, decreased RS content due to pericarp losses in the nejayote. Amylose and gradual gelatinization results in a stable, longer-required crystal structure. Therefore, the gelatinization temperature and enthalpy are higher and they observed higher crystallinity. The RS values for both flours in the studies carried out are consistent with . (Martinez, 2023) (Escobedo, 2023)(Cuevas, 2023)

3.1.1 Particle Size Distribution

Figure 1 shows the fraction of material as a percentage of mass of the SOP and ENP retained in each mesh. indicates that the results of the ANOVA, of the significant differences (p<0.05) between the nixtamalization processes in each fraction of retained material. In both mass types, the cumulative material retained between US No. 20 sieve (850 μ m) and US No. 80 sieve (180 μ m) was 92.3% and 83.1% for ENP and TNP, respectively. They mentioned that Mexican regulations related to the production of nixtamalized corn flours complied with up to the US No. 80 sieve (180 μ m) retain 75% of the cumulative material. This indicates that this PSD has a coarse to medium particle distribution, which is appropriate for making tortillas. Allowing the mass fractionation method to make an efficient comparison between both processes. (Preciado, 2022) (Intriago, 2020)(Carrillo, 2023)(Zagaceta, 2020)

Figure 1

Mass particle size distribution of ENP and TNP. The bars indicate the standard deviation.



PSD is directly related to the milling process used. Dry grinding is a fine granulometry, modifying the physicochemical characteristics and drastically changing the granular structure and surface of the starch. Wet milling into coarse and fine particles where cornmeal is produced, but these differences are less pronounced than dry milling.(Song, 2023)

According to , and , it is possible to measure the degree of gelatinization by monitoring the presence of native and fragmented starch granules at each step of both processes. SEM micrographs show the number of damaged starch granules in the corn kernel, flour, dough, and tortilla. As operations progress in both nixtamalization processes, starch damage became more critical during dough-to-tortilla baking. This was demonstrated in a scanning electron micrograph. (Lopez, 2020)(Galindo, 2021)(Tejeda, 2021)(Huchin, 2021)

Table 1 shows the particle size index (PSI) of corn flours from the ENP and TNP. , the higher the PSI value, the finer the corn flour. The PSI in ENCF was significantly ($p<0.05$) higher than the NCF. This is probably due to the grinding process they used. Dry grinding is more abrasive and produces a finer particle distribution, which creates more damaged starch granules. In contrast, wet grinding is responsible for creating a coarse grain size, where more native, fragmented starch granules exist at each stage of the process. Therefore, they increased the cohesiveness of the dough and improved the machinability of the tortillas. They chose a suitable PSI to produce flour and allow to improve the conditions of the extrusion process.(Rojas, 2020)(Carrillo, 2023)(Rizo, 2024)

3.1.2 Water Absorption Capacity

They measured the WAC (L water/kg flour) subjectively the amount of water absorbed by the flour during the preparation of the dough; It mentions that it is the amount of starch degraded in the extrusion process. They exhibited lower ($p<0.05$) extruded nixtamalized corn flour in WAC than nixtamalized corn flour (Table 1). I assert that the higher degree of cooking and compaction of the extruded flour as a result of the smaller intermolecular spaces.(Acosta, 2023)(Escobedo, 2023)

The dry grinding and thermal gelatinization conditions are more severe than those observed in wet grinding. It indicates that thermomechanical processes force gelatinization in an accelerated way, decreasing the water absorption capacity of extruded flours. considers that the particle size, the degree of gelatinisation and the damaged starch content; as the most important factors for increasing WAC in extruded flours.(Intriago, 2020)(Cancino, 2021)

However, wet grinding with plenty of water is what protects the structure of the starch granules. The maceration period of the corn kernel during the cooking liquor promotes the gradual gelatinization of the starch granules and a greater absorption of calcium, hardening the cell wall. For that reason, they claim that water enters the protein matrix more easily.(García A. , Actividad antihipertensiva de péptidos de zeína extraídos de maíz (Zea mays L.) criollo (azul y rojo) del Estado de México, 2020)(Dominguez, 2020)

They found it necessary to develop extrusion conditions that do not damage starch as much by increasing the water absorption capacity; Consequently, the textural and sensory characteristics of the dough and tortillas more closely resemble those of the traditional process.(Martinez, 2023)

3.1.3 Water Absorption Rate

The WAI value relates to grinding and extrusion parameters, such as barrel temperature and moisture content. Extruded nixtamalized maize flour with a slight but higher water absorption rate ($p<0.05$) than nixtamalized maize flour (Table 1). This is likely due to the limited water content used in dry milling, which results in further fragmentation of the starch granule and dextrinization. In such a way, the breaking of inter- and intramolecular hydrogen bonds allowed the release of hydroxyl groups and increased the ability to form more hydrogen bonds with water.(Cuevas, 2023)(Váquez, 2020)(Aguilar, 2022)

Comparing both dry grind and wet grind, they required a high WAI to promote greater flexibility and better reheat ability in tortillas, but overheating contributes to forming an amorphous, sticky dough; where starch granules lost structure and integrity. Therefore, the right conditions in both processes help to decrease the damaged starch content. Similar WAI values previously reported by .(Abou, 2022)(Roldán, 2020)(Zagaceta, 2020)

3.1.4 Water Solubility Index

The WAI value with milling and extrusion parameters, such as barrel temperature and moisture content, showed extruded nixtamalized corn flour with a slightly higher water absorption index ($p<0.05$) than nixtamalized corn flour (Table 1). This is likely due to the limited water content used in dry milling, which results in increased fragmentation and dextrinization of the starch granules. In this way, the breaking of inter- and intramolecular hydrogen bonds allowed the release of hydroxyl groups and increased the ability to form more hydrogen bonds with water.(Campos, 2023)(Escobedo, 2023)

Comparing dry milling and wet grinding, they required a high WAI to promote greater flexibility and better reheating of tortillas, but overheating contributes to the formation of amorphous, sticky dough, in which starch granules lose their structure and integrity. Therefore, processing under appropriate conditions in both processes helps to decrease the damaged starch content. Similar WAI values have previously been reported by .(Abou, 2022)(Zagaceta, 2020)(Palacios, 2023)

3.2 *Mass Physicochemical Evaluations*

3.2.1 *Moisture content, resistant starch and pH*

The moisture contents of the dough were significantly higher ($p<0.05$) than those of the dough prepared with extruded flour (Table 2).

Table 2

8.4b

Características	PEV	PNT
Contenido de humedad (%)	53,1 ± 0,13a 1,2	57,4 ± 0,68b
Almidón resistente (g/100 g de muestra)	1,43 ± 0,053a	0,85 ± 0,073b
pH	8.2a	8.4b

Note: 1 Means ± standard deviation. 2 Stockings with the same font are not significantly different ($p<0.05$).

They analyzed that this occurs because extruded flour absorbs less water than dough. The extreme conditions inside the extruder are probably the main causes why the starch granules do not increase in volume as much as during traditional nixtamalization. In dry grinding, the thermal and mechanical damage is greater. Starch granules do not expand properly, resulting in a higher density of the flour, leading to a more compact product.(Mera, 2020)(Jiménez, 2021)

Wet milling is the release of starch granules from the protein matrix. This makes it possible to increase the water content due to the swelling of the starch granules, which resulted in a volume increase of up to 30-40%. Another point they considered is that annealing favors the interaction of starch granules with calcium during the maceration of cooked corn, which is between 12 and 14 hours. Annealing in a range between glass transition temperature and gelatinization temperature; This mechanism promotes an increase in the water content in the dough to approximately 45-60% (w/w) and improves the pasty, rheological and textural properties of the dough.(Ungureanu, 2022)(Preciado, 2022)(Campos, 2023)

They indicated that Table 2 shows the resistance of the starch contents of both nixtamalization processes. They analysed the SR and it was higher in extruded nixtamalized maize flour, although no significant differences were observed. He mentions that the increase is related to the milling method used to obtain corn flour. They include that dry milling of the whole grain of corn from the pericarp prevents the release of lipids and proteins from the starch granules; Therefore, they increased the dietary fiber content, producing gelatinization even though there is a reduced amount of water present.(Orellana, 2023)(Madrigal, 2022)

Wet milling refers to the transformation of nixtamalized corn kernel into fresh masa considering the loss of fiber and lipids during the drainage of the nejayote. This reduces the formation of amylose-lipid complexes. They found similar RS contents in extruded flour, which is consistent with the behavior observed in this study.(López, 2020)(Carrillo, 2023)

They measured the pH in both processes and were significantly different ($p<0.05$), in both alkaline values meaning that a specific lime content is necessary to promote the correct flavor when producing the tortilla.(Medina, 2023)

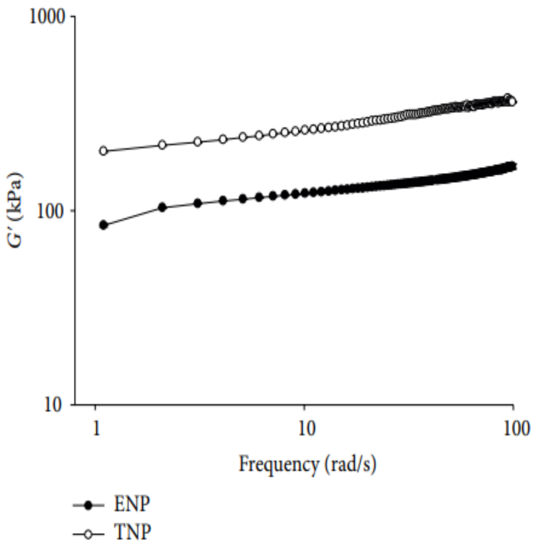
3.3 *Rheological Mass Evaluations*

3.3.1 *Frequency Sweep Test*

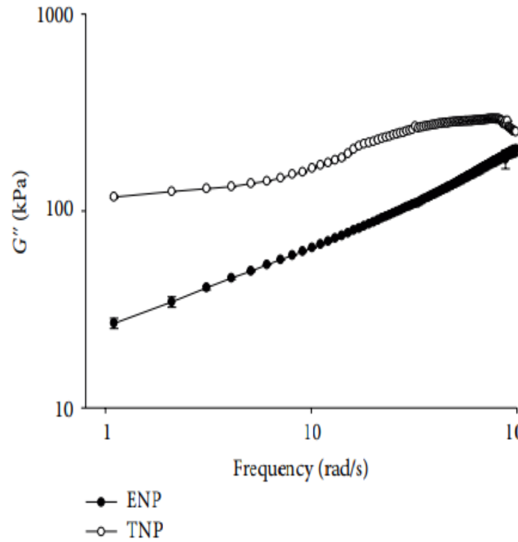
Figure 2 shows the viscoelastic (G') and frequency-function (G) parameters. According to Figure 2(a), (G') showed a linear increase of up to 10 rad/s. Then, the frequency rate increased and the structure of the polymers collapsed. (G"). The greatest thing observed in fresh dough is probably the concentration of lime used during traditional nixtamalization. In contrast, wet milling includes the treatment of the starch granule with a higher concentration of lime, which promotes stronger interactions with calcium ions and leads to gradual gelatinization. In addition, the soaking period in the cooking liquor reduces the glass transition temperature and imparts mobility to the amorphous region of the starch granules, making them softer, deformable, and possibly more elastic. However, the thermomechanical method of extrusion and dry grinding destroys the native structure of the starch granule, they analyzed that with low water content gelatinization is accelerated, this leads to the decrease of the value (G'). (Cabral, 2024) (Aguilar, 2022)(Morales J. , 2023)(Hernández, 2023) (Olaechea, 2020)

Figure 2

(a) Elastic modulus (G') and (b) viscous modulus (G") of maize mass as a function of the frequency of extruded nixtamalization (ENP) and traditional nixtamalization (TNP) processes.



(a)



(b)

They describe the behavior of G" (Figure 2(b)), the lower values in mass are the result of a higher amount of pregelatinized starch. When the extruded mass is rehydrated, it presents a compact and solid state that originates due to the small difference between the viscoelastic modulus, which is a characteristic of a solid hook. Dry grinding and thermomechanical processes produce a mass with lower strength and lower viscoelastic properties.(Gastelum, 2023)(Martinez, 2023)(Huchin, 2021)

Another reason why this behavior of dry milling is explained is because it induces a deeper alteration of the crystal structure and degradation of starch molecules.(Ramírez, 2021)

According to him, the higher water content of nixtamalized corn flour increases the elastic behavior of the dough. Wet grinding involves the formation of an elastic network due to a crystal domain derived from the complexation reaction between amylose and lipids. Therefore, he mentions that the viscoelastic modules show a big difference and the elasticity increases.(Ungureanu, 2022)(Intriago, 2020)

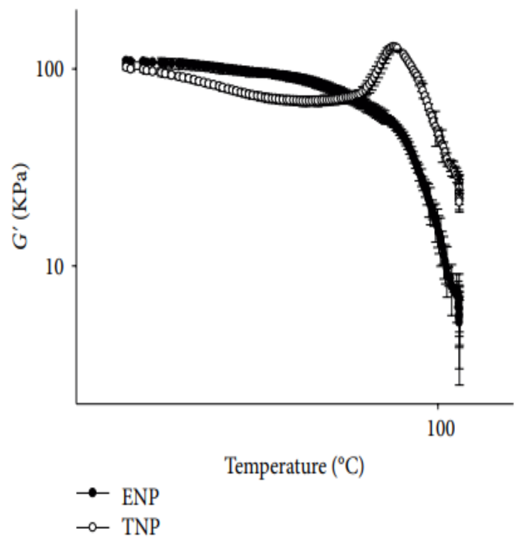
3.3.2 Temperature Sweep Test

Figure 3 shows the viscoelastic behavior of the mass from ENP and TNP as a function of temperature. indicates that fresh dough (G') has a higher viscoelasticity than extruded dough, indicating the formation of two different starch granule structures (Figure 3(a)). At the maximum peak detected (129 KPa) in the fresh dough plot (G'), they found that starch gelatinization of native and fragmented starch granules occurred between 76°C and 80°C as explained by . (Amores, 2022)(Pérez, 2023)

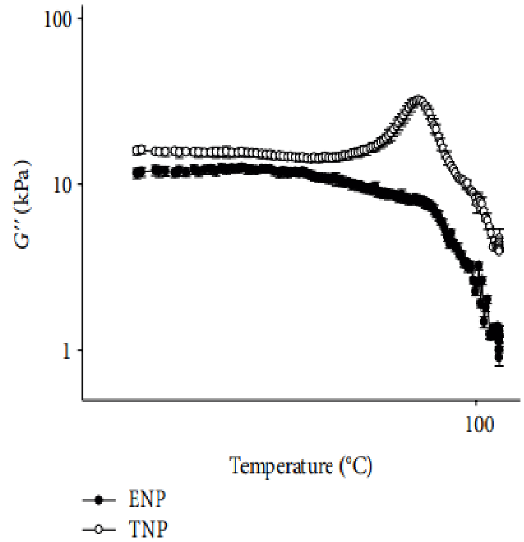
They determined that this is due to the jamming of small particles that they get in the fine grinding of cornmeal. This also explains how several granules remain intact during this step of the process.(Mejía, 2022)

Figure 3

(a) Elastic modulus (G') and (b) viscous modulus (G") of corn dough as a function of the temperature of the extrusion (ENP) and traditional (TNP) nixtamalization processes.



(a)



(b)

(Hernández, 2023) indicates that the extruded masses did not show peaks and, as the temperature increased, the viscoelasticity curve (G') showed a tendency to decrease, as shown in the (Figure 3(a)). A drop in (G") indicates further weakening of the network structure. The straight line observed in the extruded mass graph likely indicates the presence of pregelatinized starch and the decrease in the highly branched structure of the amylopectin chains due to shear and heat degradation.(Roldán, 2020)(Gastelum, 2023)

(Quinteros, 2021) He mentioned that the results for elastic modules were similar to those obtained for viscous modules (G") for both processes (Figure 3(b)). The fresh mass (G") had a gelatinization temperature range between 76°C and 80°C, a maximum peak value of 32 kPa and an abrupt decay when gelatinization was completed. On the contrary, they decreased the extruded mass with increasing temperature and did not show any peak.(Carrillo, 2023)

This pronounced reduction in the viscosity modulus of the extruded mass is related to the rapid gelatinization and previous modification of the structure of the starch granules. G' was higher than for both processes (G"), indicating the predominance of elastic over viscous .(Váquez, 2020)(Olaechea, 2020)

The author has found the temperature sweep test to be a very useful tool because the rheological changes, mainly the peaks observed for elastic and viscous modulus, are clearly attributable to changes in the starch granules due to gelatinization, and allows to graphically observe the differences in the degree to which both processes produce changes in starch. Manipulating WAC and WAI is a way to control extrusion conditions so that the physicochemical changes of starch are similar in both processes. They observed similar behaviors in the sweep test for both dough produced from nixtamalized extruded corn flour and for fresh dough, produced by the traditional nixtamalization process. Extrusion conditions are identified where starch changes are similar, which will result in tortillas with better texture and organoleptic properties obtained through the extrusion process. (Rojas, 2020) (Madrigal, 2022)(Quinteros, 2021)(Dominguez, 2020)(Escobedo, 2023)

3.4 Physicochemical properties of the tortilla

3.4.1 Moisture content

According to the study, the moisture content of the tortillas in the ENP was significantly higher (P<0.05) than those in the PNT (Table 3). This difference is likely due to the amount of water added by the operator. When they prepared masses beforehand in the laboratory, they empirically established a moisture content. From this point on, measuring the subjective texture was the responsibility of the operator. They prepared the tortillas from both processes in the same commercial plant with the same conditions of the baking process. The proper temperature, water content, cooking time and drying allowed to improve the textural properties of the dough and tortilla. (Intriago, 2020) (Huchin, 2021)(Jiménez, 2021)(Tejeda, 2021)

Table 3

1.4 ± 0.11b

Características	**PEV**	**PNT**
Contenido de humedad (%)	45,0 ± 0,18a [1]	40,7 ± 1,63b
Almidón resistente (g/100 g de muestra)	1,64 ± 0,06a	0,90 ± 0,04b
Características físicas		
Peso (gramos)	26,81 ± 1,0a [2]	20,3 ± 1,57b
Diámetro (cm)	14,0 ± 0,14a	13,9 ± 0,09b
Espesor (mm)	2,5 ± 0,1a	1,4 ± 0,11b

Note: [1]Mean standard deviation.[2]Means with the same letter are not significantly different (p>0.05).The moisture content of the corn tortilla is related to the water absorption capacity (WAC) and the water absorption index (WAI), which depends on the damage to the starch granules.(Rizo, 2024)

3.4.2 Resistant starch

Table 3 shows the RS content in tortillas made with extruded nixtamalized corn flour and fresh dough. They analyzed this value, which was significantly higher (p<0.05) in tortillas made with ENCF (1.65 g/100 g sample) than with fresh masa (0.90 g/100 g sample). This behavior is likely due to the processing steps in the extrusion nixtamalization process, such as dry milling, baking, and baking, where the starch granules are exposed to a more severe mechanical and heat treatment than they used to obtain an extruded cornmeal. The high RS content in tortillas produced with ENCF is also linked to the higher dietary fiber content in the pericarp. (Cabral, 2024) (Magaña, 2020)(Zagaceta, 2020)(García R. , 2022)

The production of RS in the traditional nixtamalization process occurs during the stages of cooking, maceration, dough forming, and tortilla baking. The RS content may decrease or increase with the degree of starch gelatinization and the type of resistant starch. Similar SR values for maize flours reported above are consistent with this study.(López, 2020)(Acosta, 2023)(Carrillo, 2023)

Retrograde is directly related to the content of RS. The rearrangement reactions between amylose and amylopectin in retrograde starch depend on the storage time and the ability of these polymers to form a new crystal lattice. The author made mention of amylose in short-term storage and is responsible for the hardening and loss of flexibility of tortillas. Amylopectin has long-term effects and is related to the recrystallization process.(Amores, 2022)(Intriago, 2020)(Tejeda, 2021)

3.4.3 Physical Properties

Table 3 shows the weight, diameter and thickness of tortillas made with both technologies. They made the tortillas with ENCF, where weight, diameter and thickness values were obtained significantly higher ($p<0.05$) than those observed for the fresh dough. This is likely due to the whole corn milled to produce ENCF, which resulted in an increase in the diameter and thickness of the dough discs. According to the size of the corn sand particles and the thickness of the dough disk are the most important attributes in the subjective measurement of tortilla quality.(Cabral, 2024)(Dominguez, 2020)(Cuevas, 2023)

3.4.4 Firmness

Figure 4(a) shows the firmness of the tortilla evaluated at 2, 24 and 48 h of storage at room temperature (25°C). The tortillas were preheated (60°C) and then cooled to 30°C before firmness was measured. They suggested this heating process by the corn flour industry (personal communication) allowing firmness to be measured in the usual way a tortilla is consumed. ANOVA indicated that tortillas made with ENCF were significantly harder ($p<0.05$) than those made with fresh dough. Firmness increased to 18% and 50% in extruded tortillas and to 0.75% and 1.25% in nixtamalized tortillas after 24 and 48 h of storage, respectively. They determined that this is likely due to the processing conditions used during extrusion and changes in gelatinization, melting, and dextrinization that directly affect the texture of starch gels. Another explanation they mentioned is the effect of the fiber content of ground whole corn used in the ENP. (Camaño, 2021) (Escobedo, 2023)(García A. , Actividad antihipertensiva de péptidos de zeína extraídos de maíz (Zea mays L.) criollo (azul y rojo) del Estado de México, 2020)(Galindo, 2021)

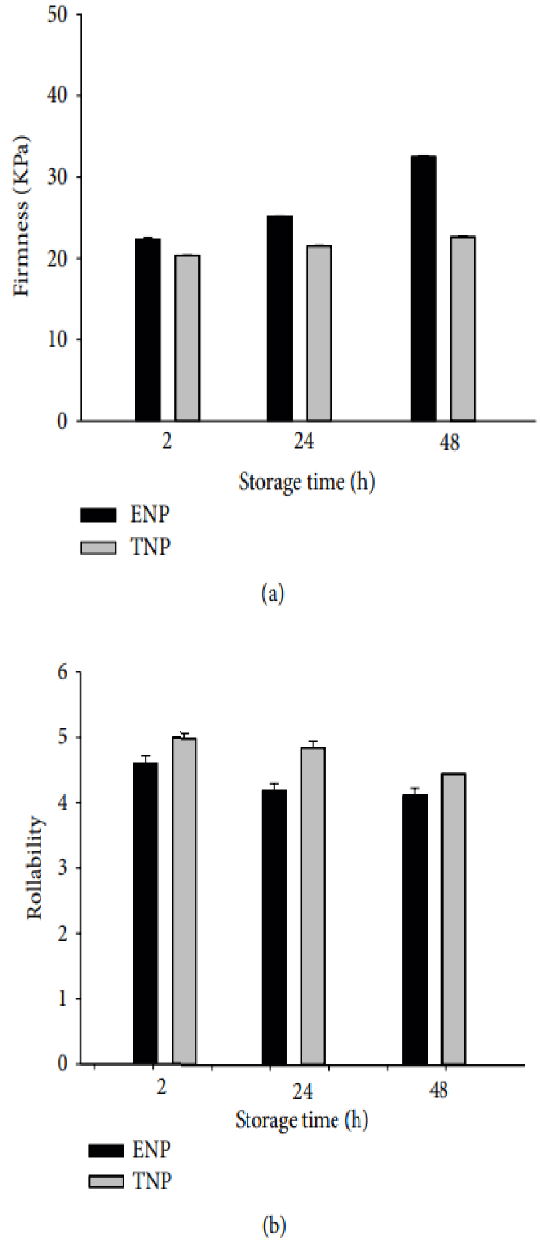


Figure 4

(a) Firmness and (b) rollability of corn tortillas from the extruded nixtamalization (ENP) and traditional (TNP) processes during storage. The bars indicate the standard deviation.

Morales (2023), mentioned that flours with a smaller particle size result in a starch fiber matrix with a rigid structure and therefore a harder product. Hardness is probably the sensory attribute most affected by extrusion and associated with the texture of tortillas. The firmness values obtained for ENCF, as reported by and , showed a similar behavior in the analysis.(Rojas, 2020)(Hernández, 2023)

3.4.5 Rollability

Rollability is a common subjective test related to the gelatinization of starch achieved during nixtamalization, and they observed the affectation by lime concentration and cooking time. Rollability values are measured by assigning a score that reflects tortilla breakage and somehow measure starch damage.(Campos, 2023)(Huchin, 2021)

Figure 4(b) The rollability of the tortilla at 2, 24 and 48 h of storage at room temperature (25°C). They determined significant differences ($p<0.05$) at 2 h and very significant differences ($p<0.01$) at 24 h between both types of tortillas. The measurement was performed at 48 h and showed no significant differences ($p>0.05$). This is probably due to the retrogradation process that all types of tortillas undergo. For both processes, the tortillas became more brittle as the storage time progressed. The ANOVA showed that the type of process and the storage time very significantly affected the rollability ($p<0.01$). The interaction between both variables (time per process) was not significant ($p>0.05$). (Montoya, 2020) (Orellana, 2023)(Cabral, 2024)(Martinez, 2023)

The process of reheating the tortilla was similar to that reported in the tortilla firmness section. They allowed this procedure to obtain high values of rollability, which led to a score above 4, and in the opinion both types of tortilla determined that it is suitable for the consumer. Other studies showed that the rollability of tortillas made with ENCF without a preheating process decreased dramatically as the storage time increased.(Váquez, 2020)(Intriago, 2020)(Orwa, 2023)

3.5 Morphology

Figure 5 shows the scanning electron micrograph (SEM) images of corn grain, flour, dough, and tortilla from the ENP (Figures 5(a), 5(b), 5(c), and 5(d), respectively) and TNP (Figures 5(e), 5(f), 5(g), and 5(h), respectively). The author realized the presence of native and fragmented starch granules in each step of both processes. Showing Figures 5(a) and 5(e) SEM images of corn, starch granules (S) are round and polyhedral in shape with smooth surfaces and minimal depressions and wide distribution prior to processing. They also observed starch granules with a smooth surface or with some depressions. (Caves, 2023) (Medina, 2023)(Quinteros, 2021)(Váquez, 2020)

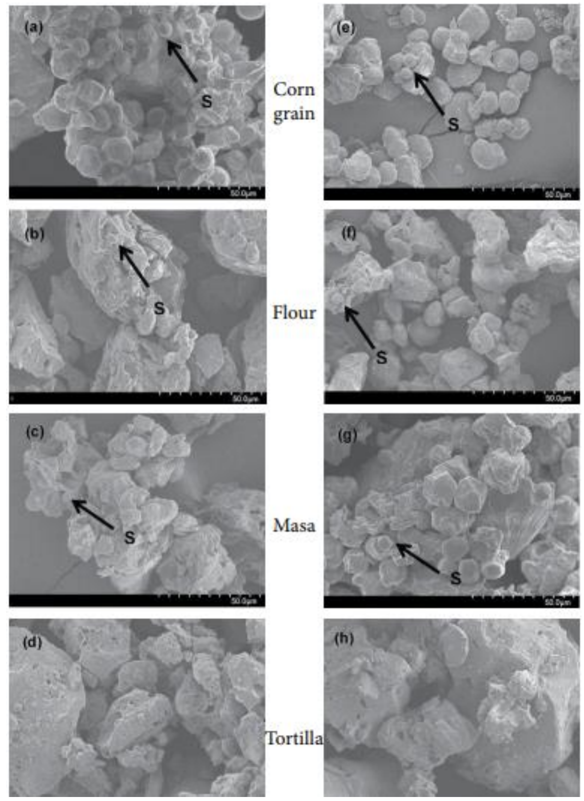


Figure 5

(a, e) SEM images of corn, (b, f) flour, (c, g) masa and (d, h) tortilla from ENP and TNP. The magnification is 1000X.

Figures 5(b) and 5(f) SEM images of ENP extruded nixtamalized corn flour and TNP freeze-dried dough, respectively. With respect to the ENCF, they indicated that Figure 5(b) is the reduction in the number of granules and the increase in irregular shape and surface area of the pores for both treatments, determined that it was greater in extruded corn flour, probably due to increased enzymatic hydrolysis and starch digestibility. They analyzed that the dry milling of the corn kernels they produce in extrusion-cooking is carried out with low moisture content and causes a large number of starch granules to fragment and imbibe in the endosperm matrix, as well as several granules to disperse outside it. They indicated that this agglomeration of SD granules were amorphous structures and, at this point, dextrinization was possible. (Perez, 2023) (Hernández, 2023)(Campos, 2023)(Amores, 2022)(Rojas, 2020)

On the other hand, the author indicates in Figure 5(f), the SEM images of TNP lyophilized flour, as well as ENP, show a reduction in the number of granules and irregularly shaped surfaces. However, they determined its overall effect on flour is lower in TNP than in ENP. They analyzed the reduction in particle size of both flours by increasing porosity and reducing cellular connectivity, which directly affected the dough and textural parameters of the tortilla. , observed an extensive area of damaged starch granules in cornmeal during the extrusion process. (Rojas, 2020) (Escobedo, 2023)(Palacios, 2023)(López, 2020)

Figures 5(c) and 5(g) showed mass SEM images of ENP and TNP, respectively. They obtained the mass with ENCF in Figure 5(c) that show an almost zero presence of native starch granules with defined structure. In the extrusion-firing process they originate more with SD and an amorphous structure due to the high degree of hydrolysis of gelatinized starch. They examined in this step, where they controlled the mixing time and the addition of water where they avoided extruded adhesive mass. In contrast, Figure 5(g) shows nixtamalized mass with fragmented starch granules and several without fragmentation in the native state. They determined the prevenient enzymatic attack of wet grinding. They obtained the structures where they are more defined due to the partial gelatinization that the granule undergoes during annealing and retrogradation. They increased the formation of resistant starch through the nixtamalization process and allowed several granules to remain with their native crystal structure and others to collapse completely. (Dominguez, 2020) (Váquez, 2020)(Ramírez, 2021)(Carrillo, 2023)(Rojas, 2020)(Morales J. , 2023)

Figures 5(d) and 5(h) show SEM images of tortillas from the ENP and TNP, respectively. They analyzed for both extrusion and traditional nixtamalization, tortilla production present continuous amorphous structures in the form of agglomerates. This is a consequence of the melting of starch granules during thermal processing due to high temperatures (270-290°C). They indicated that at this stage, complete gelatinization of starch granules occurs, the volume of starch granules decrease considerably, and they are only present in a small amount of starch granules. Finally, they observed that the greatest damage to starch granules by ENP occurs during extrusion to produce ENCF and at the dough-tortilla baking stage. They mentioned that in TNP, the greatest starch damage occurs in the dough and tortilla baking step. (Intriago, 2020) (Aguilar, 2022)(Huchin, 2021)(Song, 2023)(Orellana, 2023)

4 CONCLUSIONS

In general, mention is made about the physicochemical, rheological and morphological characteristics of the products coming from the traditional nixtamalization and extrusion processes that are different from the nixtamalization extrusion process. This may be why there is more damage to corn starch in EPN than in TNP. The corn flour from the SOP was indicated to have lower PSI and resistant starch but higher water absorption capacity than the ENP flour.

The viscoelastic parameters G' and G" as a function of frequency mentioned that they are higher in the mass of the SOP than in the mass of the ENP. According to the results they obtained from the temperature sweep test, the fresh mass of TNP showed a peak in G' (364 KPa) and in G" (138 KPa), while the mass of ENP obtained lower values of these peaks (G' = 113 KPa, G" = 33.5 KPa).

In the study, they determined that tortillas produced using the traditional nixtamalization process have lower hardness and higher rollability after 24 hours of storage. However, after 48 hours of storage, similar values were observed in these parameters for both processes. These results highlight the efficiency of extrusion as a valuable alternative in the production of corn flour and tortillas. Importantly, however, dry grind (ENP) resulted in greater starch damage compared to wet grind (TNP). These results demonstrate the importance of considering processing methods in corn-based food production to ensure the quality and acceptability of the final product.

Finally, they diagnosed the resistant starch content in corn flour, masa and tortillas; gradually increased in both processes, but obtained a higher content in ENP than in TNP. They represented the nutritional benefits on the health of tortilla consumers. They indicated that the greatest damage to starch in the ENP occurs during the extrusion stage for flour production and in the dough-tortilla baking stage. In TNP, the greatest starch damage occurs in the dough and tortilla baking step.

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