Journal of Food Engineering xxx (2017) 1–10

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Study of the morphological, structural, thermal, and pasting corn transformation during the traditional nixtamalization process: From corn to tortilla

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ARTICLE INFO

Article history:
Received 6 December 2016
Received in revised form
10 May 2017
Accepted 30 May 2017
Available online xxx

Keywords:
Nixtamalization
Gelatinization
Critical points
Starch
Infrared images
Milling
Masa
Nejayote

ABSTRACT

The changes in the structural, thermal, morphological, and pasting properties of corn were studied along all stages of the nixtamalization process with especial emphasis on the critical points such being the cooking and steeping, wet milling, and baking. Differential scanning calorimetry found an endothermic peak located between 65 and 70 °C that is directly related to the starch gelatinization, and the second exothermic peak about 100 °C that is related mainly to the presence of resistance starch. It was found that corn starch undergoes at least three different gelatinization stage phenomena along the process that provides as a result the changes in its physicochemical properties. One of the most significant findings in this work was the fact that the amount of resistant starch increases with consecutive stages of the process. The quantification of the structural changes that starch undergoes was done by using an X-ray diffraction and pasting profiles. The thermal images of the nixtamal during the wet milling and of the tortilla baking show that the temperature in both cases increases up to the characteristic gelatinization temperature of corn starch and produces a consecutive gelatinization in the starch.

1. Introduction

The traditional nixtamalization process is used in Central America, Mexico and southern United States to transform corn mainly into tortillas and fried snacks. The nixtamalization to produce tortilla is a multi-step process (Fernández-Muñoz et al., 2004; Gutiérrez et al., 2007). The first step is related to the cooking of the grains in an over saturated solution using lime from 0.5 to 2% w/w (calcium hydroxide). After the cooking, the grains are steeped from 0 to 24 h, this step is under the thermo-alkaline condition and the high temperature produce gelatinization within the starch located in the outermost layers of the endosperm (Rojas-Molina et al., 2007), after that, the nixtamal is drained off for the removal of the lime excess and fractions of the pericarp, germ, and endosperm. Then, the grains are rinsed once or twice to reduce the calcium content, giving thus: whitened nixtamal. The second step is one of the most significant unitary operations for the tortilla production, such as the wet milling that in most of the cases is made by using a stone milling process.

Another important aspect is to define if during the cooking and steeping there are pregelatinized grains or partially gelatinized grains (first gelatinization phenomenon). To the best of our knowledge pregelatinized means that some starch grains were previously gelatinized, while partially gelatinized starch granules

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http://dx.doi.org/10.1016/j.jfoodeng.2017.05.034
0260-8774/

are grains in which some regions of the amylose/amylopectin were modified. Due to different thermal and thermo mechanical process the corn grains go through different gelatinization stages that are critical for tortilla preparation.

In the case of wet milling, no information about the physicochemical changes that take place in the nixtamalized corn had been reported in detail. According to Serna-Saldivar, 2012, stone grinding plays an important role because it disrupts the swollen pregelatinized starch granules and distributes the hydrated starch and protein around the ungelatinized portions of the endosperm that forms the *masa*. Fine grinding produces soft *masa* for table tortillas while course masa is destined for the elaboration of fried snacks. However, the evaluation of the physicochemical changes that took place during this process were not reported and the germ contribution was not included.

The wet milling is an inefficient energy process because part of the electric power is transformed into noise, vibrational energy and heat inside the stones (Geankoplis, 2005). The wet milling of nixtamal can be defined as a thermo-mechanical process to integrate the corn modified components for the thermo-alkaline process: pericarp, germ, and endosperm. Two cylindrical stones are used to mill the corn grains, one of the stones is fixed, and another one rotates at different frequencies between 900 and 1230 RPM. The stones used in the milling are striped to allow the dough to flow from the inside thereof to the outside.

The milling is an operation that reduces the average volume of the solid particles; that implies not only the size reduction as well as the physico-chemical transformation of the raw granular material (Leewatchararongjaroen and Anuntagool, 2016) into solid granules due to the combination of different forces that act during the milling. The forces applied during the milling may be compression, impact, or shear (Weber and Langlois, 2010), at this stage, it is important to note that the magnitude of the force as well as the time of application affect the extent of grinding achieved as well as the properties of the final products such as tortillas, tostadas, tamales, etc.

The applied forces during the milling allows for the particles to absorb the energy producing a deformation in their structures and when the force is up to the breaking force, the raw material is broken, creating a new set of particles and surfaces and new integrated particles formed by the raw materials.

There is a combination of forces that produce a particle size reduction between the stones (Zeki-BerK, 2009). At the beginning of the milling process, the distance between the stones is adjusted (compression force) by the operator to obtain a different wet particle size distribution; at high pressure, the masa is soft, and it is used to produce tortillas, while if the pressure decreases, the resulting masa turns into coarse particles, and it is used in different products such as fried and baking snacks, gorditas, and cookies, among others. The shear force depends on the time and the rotation frequency, so far there is not information about the role that this force plays in the *masa* characteristics. Finally, for the analysis of the impact forces it is necessary to assume a completed alignment between the stones, in this case this force is produced by intercomponent interaction (germ, pericarp, and endosperm), and this is the cause for the particle integration. The temperature in the sample between the stones may increase until it reaches the corn starch gelatinization temperature that can be between 55 and 85 °C (Belitz et al., 2004; Pineda-Gómez et al., 2012). The particle temperature increases due to different forces. Such event means that a second gelatinization process takes place due to the high-water content in the nixtamal and the increase of the temperature between the stones.

Fig. 1 shows the stones and their striping used for the milling process; these disks are located in front of each other and the separation between them (compression force) depends on the utilization of the final wet granular product. During the milling process, the operator can also increase the moisture content of the product by adding water.

The third stage is the baking process: it is well known that the molded tortilla (crude) is heated at different temperatures 50-310 °C. Because during baking, the tortilla is heated in three stages. During the first stage of the baking, one of the surfaces is heated, and after a few seconds, it is turned over in order to heat the other surface, after that, the tortilla is turned over again and heated until it is flattened. During this process the moisture content decreases due to the heating, but still there is a high moisture content that allows the third stage gelatinization phenomenon.

During the nixtamalization, the corn grains go through different processes: thermo-alkaline (cooking), thermo-mechanical (wet milling), and again a thermal process during the baking. In each process, the corn components go through physicochemical changes that modify the final properties of the nixtamal, masa, and the tortilla. In the case of starch, in the presence of water as solvent, the masa suffers at least three different gelatinization changes.

In the case of a pasting profile of whole corn that includes pericarp, germ, and endosperm with and without nixtamalization, the influence of each one of these parts on the pasting profile is still an open problem. The pasting profile is governed mainly by the starch transformation due to the gelatinization of the starch granule in which the internal order between amylose and amylopectin is disrupted, changing the system from diluted starch particles in water to a hydrogel (Rincón-Londoño et al., 2016a,b).

Within the germ, there are approximately 85% total fatty acids of the corn and these constitute about 30% of the dry weight of this structure; approximately, 95.6% of fatty acids are triglycerides, and 1.7% are free fatty acids (Watson, 2003). The nixtamalization process modifies the corn oil quality characteristics through the formation of oxidized compounds derived from lipids (Martínez-Flores et al., 2006). The effect of the germ on the pasting profile has been recently studied by Vega-Rojas et al. (2017), they found that the inclusion of the germ (lipids, proteins) on the *masa* increases the peak viscosity and this fact so far is attributed to the lipid-amylose complex.

The pericarp is also a well-studied structure. Using x-ray diffraction Caballero-Briones et al., 2000, studied the effect of the nixtamalization time on the structural, morphological, and thermal properties of the corn pericarp and its physicochemical changes. They proposed that nixtamalization can be viewed as a series of physicochemical phenomena that involve hemicellulose dissolution, swelling, alkaline cellulose formation, among others.

In the same direction, Gutiérrez et al. (2007) studied the morphological and physicochemical transformation in the pericarp, they found that this structure is degraded during the cooking and steeping. This structure governs the water and calcium diffusion (Gutiérrez-Cortez et al., 2010), and the thermo-alkaline process produce a pericarp percolation that allows the entrance of calcium into the endosperm and germ (Valderrama-Bravo et al., 2010; Pineda-Gómez et al., 2012), On the other hand, the structural analysis of the corn, *masa*, and tortilla has been reported. The endosperm is formed mainly by starch which is formed by two macromolecules called "amylose" and "amylopectin" with different physicochemical properties and structures; the amylose has a linear structure of d-glucan units bonded together by α (1,4) bonds, in the shape of a helix which consists of six fragments of glucose per turn, while amylopectin is a branched polymer that is composed of linear chains with α (1–4) bonds and α (1–6) bonds (Imberty et al., 1988). Generally, the corn used for nixtamalization is rich in amylopectin while amylose is present but in an amorphous manner (Rincón-Londoño et al., 2016a,b). The structural