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Efecto del calentamiento sobre la ultra estructura del almidón de batata (*Ipomoea batatas* L.)

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RESUMEN

En este estudio se caracterizó la morfometría del almidón de batata (*Ipomoea batatas* L.) empleando los microscopios óptico y electrónico (SEM). También, se evaluó el efecto del calentamiento sobre la estructura de los almidones mediante el uso de técnicas con SEM. Los resultados indican que la población granular varió en una amplio rango de tamaños (4,84-25,81 μm) con un valor promedio de 12,53 μm. Los gránulos presentaron formas que variaron de redondas y poliédricas pequeñas, a grandes en forma de campana. Los gránulos mostraron una textura lisa con algunas excoriaciones en la superficie. Las transformaciones principales ocurridas durante el calentamiento se podrían resumir como sigue: primero, hinchamiento granular, seguido por la fusión de la mayor parte de los gránulos debido a la solubilidad y a la lixiviación completa de la amilosa; segundo, la gelatinización completa de los gránulos y tercero, la transformación de la organización granular en una estructura reticular planar. En esta estructura reticular está embebido el resto de los gránulos hinchados libres de amilosa, que no han perdido su estructura externa. Se observaron estructuras o micro fibrillas que fueron consideradas como moléculas de amilosa, las cuales pudieron ser lixiviadas a través de los microporos de la superficie granular. Por otra parte, se observó la presencia de los gránulos llamados "fantasma". El conocimiento de las características y la estructura del almidón de batata facilitarían grandemente su uso.

Palabras clave: *Ipomoea batatas*, microscopia, calentamiento, estructura granular.

Effect of heating on the ultra structure of sweet potato (*Ipomoea batatas* L.) starch

ABSTRACT

The size and morphology of sweet potato (*Ipomoea. batatas* L.) starch were characterized by using the optic and scanning electronic microscopes (SEM). Also, the effect of heating on the same structure was evaluated with SEM. The results indicated that the granular population varied with an ample diameter range (4.84-25.81μm) and with an average value of 12.53 μm. The granules had small round, polyhedrical and large bell-like forms. The granules showed a smooth texture with some excoriations in their surface. The main transformations involved during the heating could be summarized as follow: first, the maximum granular swelling was reached, followed by the fusion of most of the granules due to the solubilization and complete leaching of the amylose; second, the complete gelatinization of granules, and third, the transformation of the granular organization into planar reticular structure. In this structure, the rest of the amylose-free swollen granules that did not lose the external structure was imbibed. Structures or microfibrills that were considered as

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amylose molecules were observed. This amylose could be leached out the microspores. Moreover, the presence of "ghost" granules was observed. The knowledge of the properties and the structure of the sweet potato starch would greatly increase its use.

Key words: *Ipomoea batatas*, microscopy, heating, granular structure.

INTRODUCTION

Ipomoea batatas tubers are important providers of energy rich foods. Their crops have good yields and adaptability, making them and important, sustainable, an independent source of energy and nutrients. Their tubers have a higher nutritional value than the common potato, because they are a good source of vitamins A, B and C, iron, calcium, and phosphorus. They are also high in complex carbohydrates and dietary fiber, but low in protein (INN, 1999). Multidisciplinary, integrated research and development activities aimed at improving production, storage, postharvest and processing technologies, and quality of the sweet potato and its potential value-added products are critical issues, which should be addressed globally (Bovell-Benjamin, 2007).

The intensive starch production from sweet potato is an alternative source to decrease the imports of conventional starch in the tropical areas, since starch is a main by-product due to its edible portion, especially in the eastern hemisphere (Ostertag *et al.*, 1988; Valetudie *et al.*, 1995; García and Walter, 1998; Ishiguro *et al.*, 2000). However, in the west it has not received the proper and deserved economical consideration. Indeed this starch has to be extensively studied in order to respond the questions about its structure and its relationship with its functional properties.

The native starch has four levels of structure: chemistry, conformational, microscopic and crystalline (Mestre, 1996). Each one favors the previous level, and consequently each level plays an important role in the functional properties of the starch. Since the invention of the microscope, the starch granule has been put under numerous structural investigations. Indeed, it is a potential tool for archaeological investigation (Torrence *et al.*, 2004). Moreover, the microscopic study of starch granules can be used to judge the quality (damaged granule presence) and gelatinization degree. However there are numerous questions that still need to be answered in this regard. The gelatinization of the starch is required to convert raw starch to several processed products. The gelatinization is generally achieved by heating the starch slurry up to the gelatinization temperature. The research on sweet potato starch has been focused on the extraction, purification, gelatinization, functional properties and its use (Radley, 1976; Shen and Sterling, 1981; Takeda *et al.*, 1986; Madhusudhan *et al.*, 1992; Woolfe, 1992; Batistuti *et al.*, 1993/94; Lee and Shin, 1994; Noda *et al.*, 1998; Collado *et al.*, 1999; Ishiguro *et al.*, 2000; Hoover, 2001; Noda *et al.*, 2001; Ramesh Yadav *et al.*, 2006; Zaidu *et al.*, 2008). Beside the studies of Valetudie *et al.* (1995), no other authors has widely studied, by using pictures in detail, the gelatinization process of the sweet potato starch, as it has been done for other starch sources (maize, cassava, potato). For this reason, this investigation could be a significant contribution to know the structure, as well as the properties of swelling of the starch of sweet potato during the heating process. The knowledge of the properties and the structure will determine the possibilities for its use.

The objective of this study was to evaluate the effect of the temperature on the granular organization of the granule, throughout the use of different microscopic techniques and light diffraction of the batata (*I. batatas* L.) starch.

MATERIALS AND METHODS

Materials

Three batches of clean *I. batatas* tubers were obtained from a Venezuelan local market. It was ensured there were not varietal mixes. The starch of *I. batatas* was obtained from three different batches of the tubers, following the method described by Pérez *et al.* (1993), with some modification. The cleaned tubers were peeled, weighed, sliced and ground for 2 min at high speed in a waring blender, with small volumes of distilled water. The homogenate was passed through a cheese cloth sieve. This grinding and screening operation was repeated four more times. The resulting slurry was centrifuged at 1500 rpm for 20 min. After removing the mucilaginous layer, the sediment was washed several times by suspending it in distilled water and centrifuging it until it appeared to be free of non-starch material. The sediment was then dried out in an oven at 45 °C. The dried starch was milled, passed through a 60-mesh sieve, and stored at room temperature in sealed plastic bags.

Optic microscopy

To perform the assay, a small portion of the starch was scattered and 2–3 drops of distilled water were added and the sample was held for 5 min. A coverslip was placed on top (Blaszczak *et al.*, 2003; Koroteeva *et al.*, 2007).

After the waiting time, the preparations were observed and photographed using different magnifications with the Nikon Optiphot-2 microscope with a connected camera Nikon FX-35DX.

Scanning electron microscopy

Granular shape and size and granular distribution were studied by using scanning electron microscopy (SEM). The starch was suspended in a solution of 40% ethanol and dispersed totally by using ultrasound. Several drops of the starch suspension were transferred with a Pasteur pipette onto double-sided adhesive tapes, attached to circular specimen stub. The prepared stub was coated with pt/palladium (8 min) using a Hitachi E 102 Ion Sputter. Then, examined at 20,0 kV, and photographed with a Hitachi S 2400 scanning electron microscope. The diameter range of starch granules was estimated by measuring 20–30 randomly selected granules from triplicates microphotographs (Sívoli *et al.*, 2005).

Study of the gelatinization changes

The effect of the heating on the morphology of granules was evaluated using the technique of SEM. In order to perform the assay the amylograph method was used. Aliquots of 3 mL were taken directly from the suspension of the native starch of sweet potato (8% solids) from the bowl of the Amylograph Brabender. During the heating cycle from 30-90°C (at rate of 1,5°C/min) the samples were taken every 6,6 min equivalent to 10°C each. The samples were cooled down and dried out at 45°C during 24 h. The dried sample corresponding to each temperature of heating was placed onto double-sided adhesive tapes and they were processes following the same methods described above.

Statistical analysis

Each granular size analysis was performed in triplicate and the mean calculated.

RESULTS AND DISCUSSION

Figures 1 and 2 show the native morphometric characteristic of the *I. batatas* starch viewed by optic and electron microscopy, respectively. The native granules showed an ample diameter range (4,84 – 25,81μm) and an average value of 12,53 μm (Figure 2). The results obtained in relation to the form and the size of granules of the sweet potato starch agrees with the reported by other authors (Gallant *et al.*, 1982; Noda *et al.*, 1995; Valetudie *et al.*, 1999). The granules also have small round, polyhedrical, and large bell-like forms. The most predominant form was the bell-like. sizes of granules, small and large, have a smooth texture

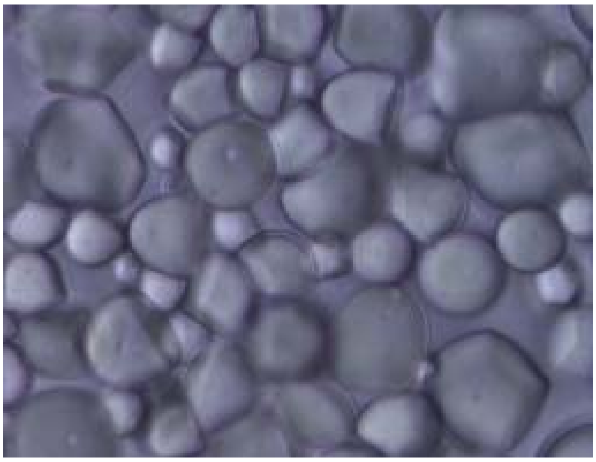

Figure 1. Microphotography of native starch (Optic microscopy at 100 X).

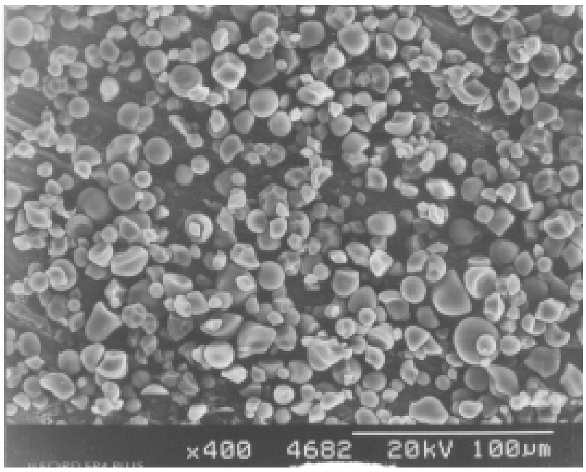

Figure 2. Microphotography of native starch with SEM.

with some exoerosion in the surface clearly visible and the bell-like granules have a depression in the center (Figure 3).

In the studies about the stages of gelatinization by microscopy (Figure 4), some observation must be discussed; the visual starch granule structure did not disintegrate and still keeping the granular form, even at 80 °C, but showed the structures that has been named "ghost" (Figure 4f). All the gelatinization morphologic changes were observed from Figure 4a to 4f. However, as can be seen in Figure 4, remarkable morphologic changes still could happen in pastes cooked at medium temperatures. At 30°C granules keep the native form and size. At 50°C (Figure 4b), the granules became agglomerates-granules compound, with numerous granules still intact. On the other hand, an increase in the granular size produced by initial swelling of the granules could be observed. This is an indication of the beginning of the water absorption by effect of the heating. As the temperature increased, the changes were more noticeable.

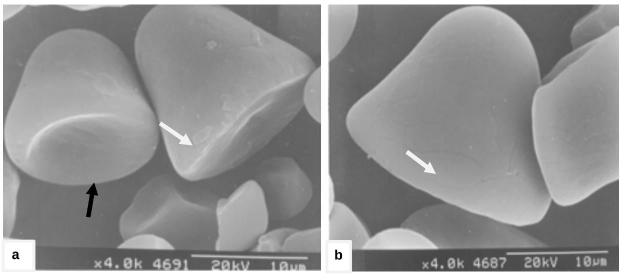

Figure 3. Microphotography (SEM) of the native starch. **a**) Exoerosions (black arrow) and depression (white arrow) and **b**) Exoerosion.

Figure 4c show that at 60°C, the granules were swollen and larges as compared to those measured at low temperatures, but still keept its natural granular shape. During the heating at 70ºC up to 80ºC, more drastic structural changes took place (Figures 4d and 4e).

In Figure 4f is shown the microphotography corresponding to the heating process at 90ºC. Species of planar reticular structure with a crystalline aspect can be seen in Figure 4e (McPherson *et al.*, 2000). This result suggests the coexistence of amylose and amylopectin in a dispersed phase, which could have given origin to the crystalline structure in the form of a fine film.

The main transformations involved during the heating could be summarized as follows: the maximum granular swelling was reached, followed by the fusion of most of the granules due to the solubilization and complete leaching of the amylose, the complete gelatinization of granules, and finally, the transformation of the granular organization into a planar reticular structure. In this structure the rest of amylose-free swollen granules that has not lost the external structure are imbibed (Figure 4e). This last process produces the so called "ghost" granules (Nuessli *et al.*, 2000). These "ghost" granules have been described as skeletons or shells of the granule, which have been absorbed in a mass of amylose. They could be structurally formed by amylopectin (Atkin *et al.*, 1999).

As discussed above, when the temperature of the paste increased, remarkable changes in the appearance of the granules were observed. Figure 5a shows the granules at 60ºC increasing their capacity of swelling and maintaining their granular integrity. At the same temperature, the leaching of intra-granular material begins. This can be seen through the appearance of small structures between some granules. These structures have been described as microfibrills (Chabot *et al.*, 1976) and it has been suggested that theses microfibrills are amylose molecules that could be leached through the microspores (Valetudie *et al.*, 1995). This amylose leaves the granule mainly through the truncated

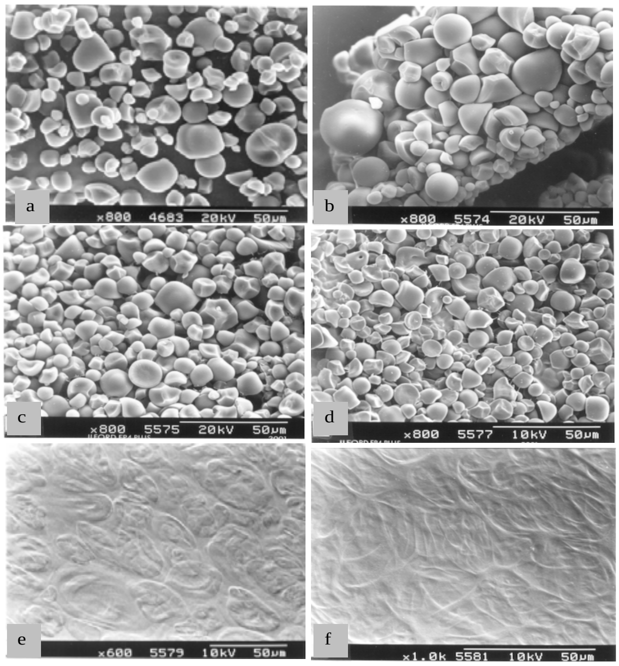

Figure 4. Microphotography (SEM) of the native starch suspension at 30ºC (**a**), and the cooked pastes at 50ºC (**b**), 60ºC (**c**), 70ºC (**d**), 80ºC (**e**) and 90ºC (**f**).

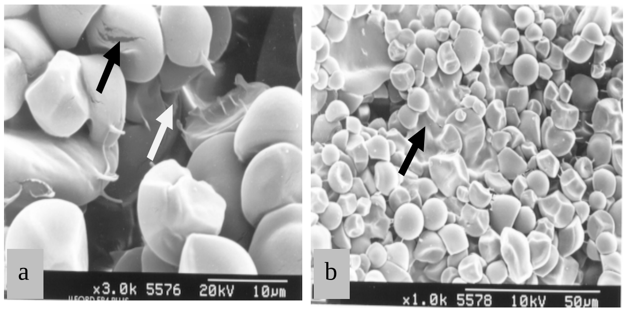

Figure 5. Microphotography of starch pastes at 60ºC (**a**) and at 70ºC (**b**) (SEM).

end of the granule (Koch and Jane, 2000). A granular crack can also be observed. Also, extra granular material can be seen accumulating in the surface, which could be soluble amylose (Figure 5a). At 70ºC, the different structural changes stages of the granules are observed. In Figure 5b, whole granules and very swollen granules can be seen, which begin to be fused with other granules constituting an amorphous mass (see black arrow). It can also be observed incipient points of union between granules. The most common are very swollen granule, which maintains its whole granular structure (Figure 5b).

CONCLUSIONS

The changes that happened on the granular microstructure by effect of the heating temperature at excess of water can perfectly be observed by SEM. At temperatures superior to 70ºC, the granules maintained their granular integrity, corroborating therefore the resistance of granules of the starch of sweet potato to the heating and the prolonged agitation. The amylose lixiviation by the truncated end of the granules should indicate that the internal bounded forces of the zone are weaker than the other extreme of the granule. The structural resistance of granules of the sweet potato starch can be a rheological important property in order to select it, before the application of technological processes, when incorporating this starch, in nutritional systems.

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