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PRODUCTION OF TORTILLA CHIPS FROM CORN AND/OR SORGHUM:
II.EVALUATION OF STARCH GELATINIZATION AND CRYSTALLINITY

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ABSTRACT

Tortilla chips are made from corn and sorghum by nixtamalization process. Nixtamalization caused some changes in physical apperaence of the protein and starch of the kernel. Gelatinization and crystallinity are of cardinal importance in starch technology. So, this study was performed to follow-up the degree of starch gelatinization (loss of birefringence and structure disruption) and the changes in starch crystallinity during tortilla chips processing. Starch granules in raw grains appeared as distorted spherocrystals, with a typical dark cross. The majority of starch granules in nixtamalized samples were swollen, adhered to other granules and exhibited partial birefringence. Masa had irregular in shape and often the external part of the granules exhibted birefringence. Tortilla chips lost about 95% of birefringence. The structural changes as evaluated by scanning electronic microscope revealed that raw samples had a mixture of round granules from the floury endosperm and angular granules from the horny endosperm. Starches granules are disrupted and swollen by cooking. Masa had irregular and swollen starch granules and a cohesive, glue like structure was appeared. Misshape starch granules along with more uniform, smoothes and more amorphous appearance of starch granules were appeared in tortilla and tortilla chips samples. The X– ray diffractograms of raw samples show pattern closely to A-type cereal straches. A diffraction peak of a feature of V-type starch was appeared in the nixtamal samples. Masa samples had the main peaks of A-type starch along with developing a diffraction peaks feature of V-patterns of starch. Starch was changed from a crystalline to an amorphous state as a result of baking and frying.

INTRODUCTION

Corn is considered as one of the principal crops in Egypt and its production is increasing annually. Corn production through 1994-1996 ranged between 4.1-4.5 million tons while from 1996-2000 it ranged between 4.5-5.5 million tons (Anon., 2000). The produced corn is mainly directed for animal and poultry feeding, although there is a shortage of cereal-based foodstuffs. Therefor, it would be beneficial to manufacture corn as foodstuffs and trying to introduce a new corn products to Egyptian food market. Snaks and covenience foods seemed to be regularly consumed in developing countries as well as have been long consumed in the Egyptian diets as popular food. Therefore, corn should be used in such products, i.e. tortilla chips.

On the other hand, sorghum is the fifth most important cereal in total world production. However, sorghum is predominantly used in Egypt as animal feed. Therefore, sorghum is utilized in this study to prepare cereal-based human foodstuffs, i.e. tortilla chips.

Tortilla chips are made from corn and sorghum by the nixtamalization process. Nixtamalization caused some changes in the physical appearance of the protein bodies and starch of the kernel. X- ray diffraction shows the differences in the crystalline forms of the starch and yields useful evidence regarding changes in the structure of starch during processing. Differences in size and shape of starch granules during processing can also be followed with microscopic techniques (Paredes-Lopez and Saharopulos, 1982; Pflugfelder *et al.*, 1988a; Gomez *et al.*, 1989; Gomez *et al.*, 1992 and McDonough *et al.*, 1993).

This study was conducted to evaluate the grains for their suitability for tortilla chips production, thoughout follow-up the physical changes occurred in starch granules during tortilla chips processing including starch gelatinization as deternined by loss of birefrengence, starch structure changes as documened with the scanning electrone microscop (SEM), and the changes in starch crystallinity as demonstrated by X-ray technique .

MATERIALS AND METHODS

Materials:

Four varieties of Egyptian corn (*Zea mays*) and sorghum (*Sorghum bicolor L.*) were used in this study. Two varieties of white corn, i.e. single cross 10 and triple cross 321; two varieties of yellow corn, i.e. single cross 152 and 156; and local variety (Dorado) of sorghum were purchased from Corn Breeding Section, Field Crops Department, Agric. Res. Centre, Giza, Egypt.

Methods:

Preparation of tortilla chips:

Tortilla chips were prepared in Food Technology and Dairy Department, National Research Centre, Dokki, Cairo, Egypt. Corn varieties and sorghum were lime-cooked (PH 10.5 to 11) at boiling temperature for 60 and 20 min., respectively (Serna-Saldivar *et al.,* 1988). The cooked corn were steeped for 8-12 hrs., whereas the cooked sorghum was steeped for 4-6 hrs. The resulted nixtamals (lime cooked and steeped grains) were washed and ground three times using a home meat mincer (Braun) with a 3, 4.5 and 8 mm plates, respectively. The resulted masas (PH 6.5 to 7) were further ground using a home blender (Braun). The different kinds of masas were sheeted, cut into circular shapes and baked for 10 sec. in microwave oven into tortillas. The tortillas were then deep fried for 1min. at 180°C in sunflower oil. Tortilla chips were drained, cooled for 5min. and examined.

Analytical methods

There are numerous methods for determining starch gelatinization, so more than procedure were used as a means of verification. Therefore, loss of birefringence as examined by polarized-light microscopec and structure disruption as observed by SEM were performed.

Loss of birefringence:

Loss of birefringence was evaluated in water-glycerol (50:50) suspensions of flour from grain, nixtamal, masa, tortilla and tortilla chips. Samples were sieved to remove the larger endosperm pieces , leaving mostly starch granules as particles smaller than 150 µm. Bright-field and polarized-light microscopic examinations were performed using a Zeiss Universal microscope equipped with a 100-W tungsten light source (Snyder, 1984).

Scanning Electron Microscopy:

Raw corn, nixtamal, masa tortilla and tortilla chips for observation by scanning electron microscopy (SEM) were dried in a vacuum oven at 4°C overnight and viewed on a JEOLJS M-25 scanning electron microscope at an accelerating voltage of 15kv. Samples were mounted with conductive adhesive and coated with 200A of gold-palladium. Comparable magnifications and positions of samples were photographed. The scanning electron microscope (SEM) JSM-T20 JEDL, available at Physical Chemistry Department, National Res. Centre, Dokki, Cairo, Egypt was used for viewing the specimens with different magnifications (Gomez *et al.*, 1992).

Starch crystallinity:

The crystallinity of starch was evaluated by X-ray diffraction patterns of samples using monochromatic Cuk radiation on a philips X-ray diffractometer at 35 kv and 15mA. Lyophilized samples were placed on the I cm^2 surface of a glass slide and equilibrate overnight at 91% rh and run at 2-32° (diffraction angle 2θ). The spacing was computed according to Bragg's law (Gomez *et al.*, 1992).

RESULTS AND DISCUSSION

To obtain a good quality tortilla chips, it is very important to follow-up the changes occurred during processing and control them.Otherwise, great variations in quality parameters of the product will occur according to grain variety and processing conditions. One of the most critical aspects of the tortilla chips process is masa texture. Masa texture depends on many factors include grain variety, dry matter losses, water uptake, degree of starch gelatinization and the differences of starch crystalline form in size and shape. Therefore, starch gelatinization and starch crystallinity were measured for all grains during tortilla chips preparation.

The term "gelatinization" generally is used to describe the swelling and hydration of starch granules. This process is of cardinal importance in starch technology. So, degree of starch gelatinization were determined and followed during tortilla chips processing. Two different microscopic techniques were used in this study to measure the starch gelatinization, e.g. loss of birefringence as evaluated by light microscope and structural changes as examined by electronic microscope.

Loss of birefrengence:

Starch gelatinization is often measured by loss of granule birefringence because of its simplicity in equipment and application. Therefore, loss of starch birefringence as a mean of starch gelatinization evaluation was followed during tortilla chips production using polarized light microscope. The obtained photographs are given in Figs (1, 2 and 3) for white corn (WC), yellow corn (YC) and Dorado sorghum (DS), respectively. It could be observed that starch granules in raw grains appeared as distorted spherocrystals, with a typical dark cross. The sign of birefringence is positive with respect to the spherocrystal radius. The apparent intensity of birefringence is depending on the granule thickness as well as on the degree of crystallinity and orientation of the crystallinity (French, 1984). The dark arms of the polarization cross occur along the local average axes of the optic refractive index ellipsoids and intersect at the hilum (French, 1984).

The majority of starch granules in nixtamalized samples (Fig.1, 2 and 3) were swollen, adhered to other granules and exhibited partial or total birefringence. However the maltese crosses of starch granules were less distinct than those of raw kernels. Therefore, the initial stage of starch gelatinized could be indicated by a darkened and enlarged hilum as well as the interference cross begins to fade and eventually disappears as starch gelatinized proceeds (Gomez *et al.,* 1992). Snyder (1984) indicated that surface gelatinization causes individual starch granules to stick together and form aggregates. After cooling starch molecules can reassociate into crystalline regments (retrograde). Amylose crystallization occurs rapidly after cooling of gelatinized starch, while amylopectin crystallization is a slower process (Aguilera and Stanley, 1999). Both are located surrounding swollen granules, thus they may be the main factor in the loss of birefringence of the gelatinized starch.

In masa samples, it is easy to observe that the kernel was physically torn apart by the mechanical cutting and shearing actions of the grinding during the conversion of nixtamal to masa. Gomez *et al.* (1992) indicated that starch of masa was no longer adherent and approximately 4-7% of the starch granules completely lost birefringence. Starch granules in raw grains appeared as distorted spherocrystals, with a typical dark cross. The majority of starch granules in nixtamalized samples (Fig. 1, 2 and 3) were swollen, adhered to other granules and exhibited partial or total birefringence. Consequently, many of starch granules in masa (Fig. $1C'_1$, $2C'_2$, $3C'_3$) appeared irregular in shape when compared to raw (Fig. $1A'_1$, $2A'_2$, $3A'_3$) and nixtamalized (Fig. $1B'_1$, $2B'_2$, $3B'_3$) samples, and often only the external part (60-70% total area) of the granules exhibted birefringence.

Baking masa into tortillas caused several microstructural changes in starch birefringence and the physical appearance of corn components. These changes were due primarily to the intense heat which further gelatinized the starch. The masa particles in the tortillas were cooked more in the centre of the tortilla than on the edges. This was reflected by the differences in birefringence observed in these areas.

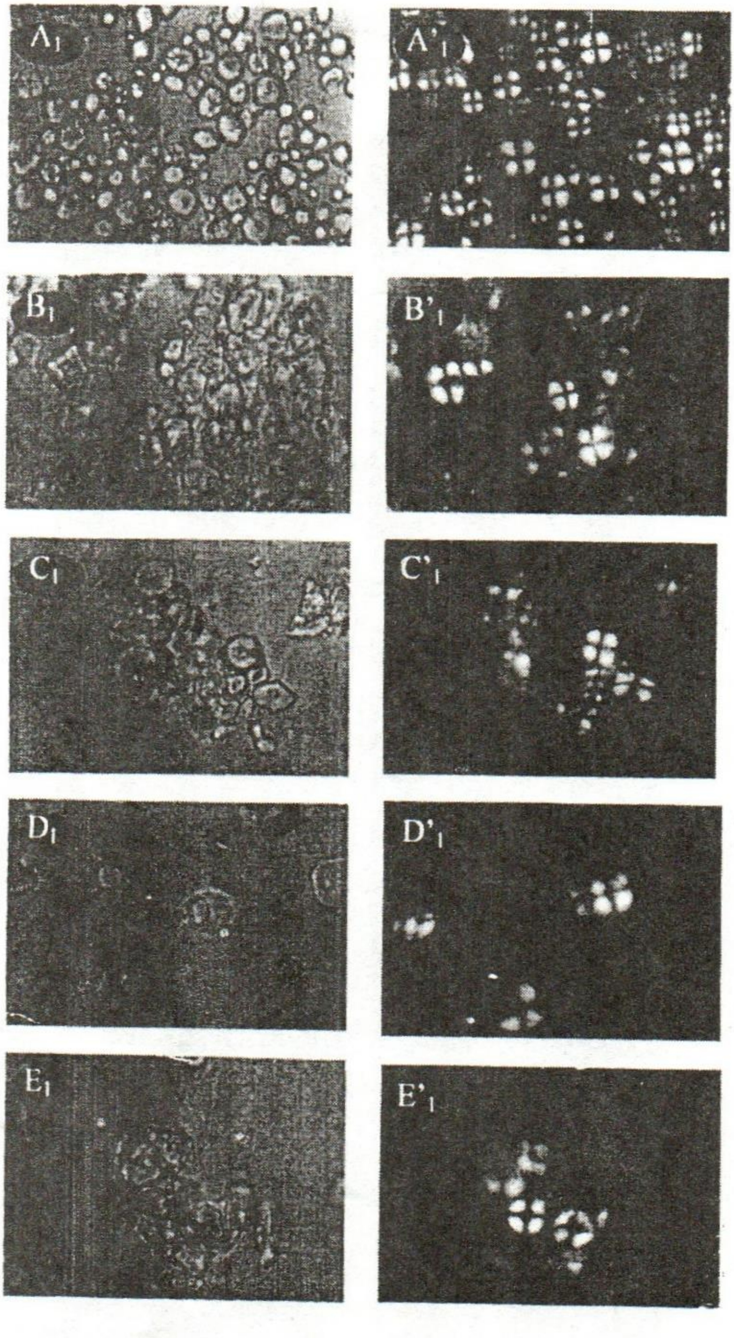


Fig. (1): Loss of birefringence in white corn flour samples. (A_1 and A'_1) raw corn; (B_1 and B'_1) nixtamal; (C_1 and C'_1) masa (D_1 and D'_1) tortilla and (E_1 and E'_1) tortilla chips. (A_1 - E_1), bright-field, (A'_1 – E'_1), polarized.

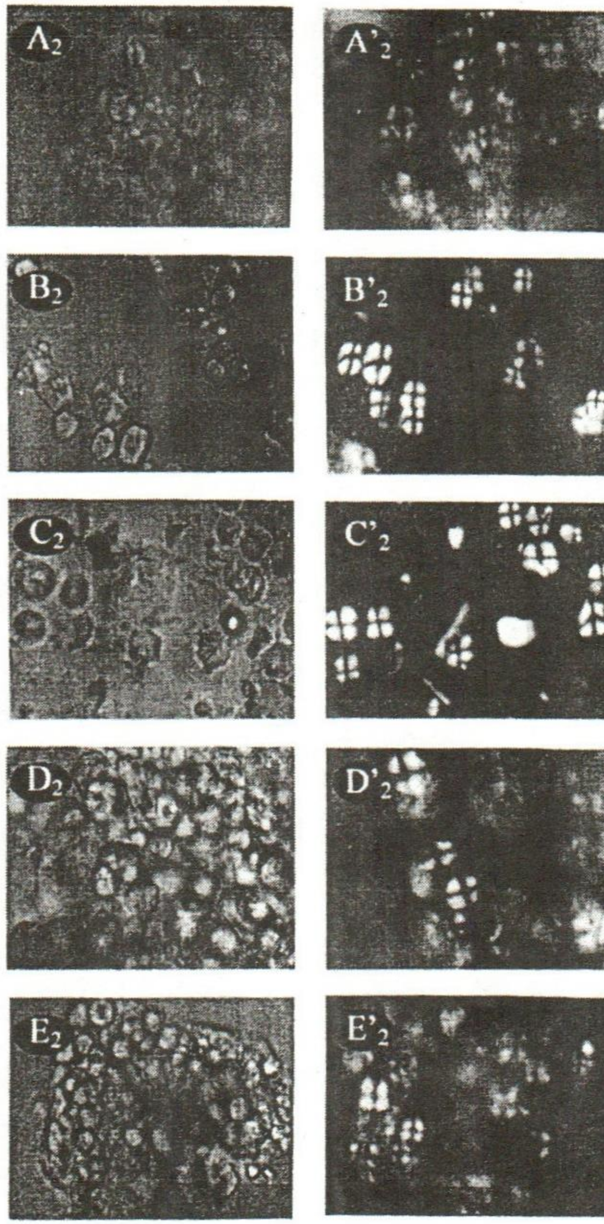


Fig. (2): Loss of birefringence in yellow corn flour samples. (A_2 and A'_2) raw corn; (B_2 and B'_2) nixtamal; ($C_2$and C'_2) masa (D_2 and D'_2) tortilla and (E_2 and E'_2) tortilla chips. (A_2 – E_2), bright-field, (A'_2 – E'_2), polarized.

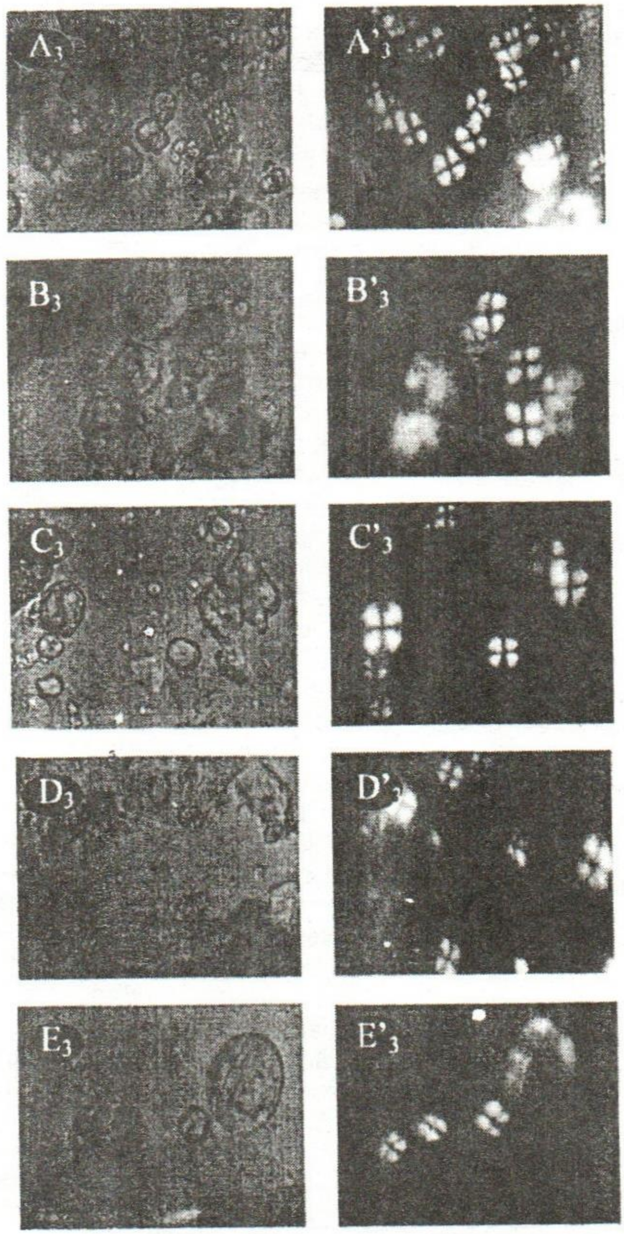


Fig. (3): Loss of birefringence in sorghum flour samples. (A_3 and A'_3) raw corn; (B_3 and B'_3) nixtamal; (C_3 and C'_3) masa (D_3 and D'_3) tortilla and (E_3 and E'_3) tortilla chips. (A_3 - E_3), bright-field, (A'_3 – E'_3), polarized.

Up to 95% of birefringence in the starch granules was lost after the tortillas were fried into tortilla chips. However, some starch granules located in the outside surfaces of the chips displayed strong birefringence, presumably because they were the first part dehydrated during baking and insufficient water was present for gelatinization during frying in the three varieties under investigation .

Young *et al.* (1990) stated that the processing of sorghum grain had significantly reduced levels of birefringent starch (12-15%) in the flour. Many of starch granules that exhibited birefringence had only partial maltese crosses with enlarged dark centre (hillum area), indicating partial gelatinization.

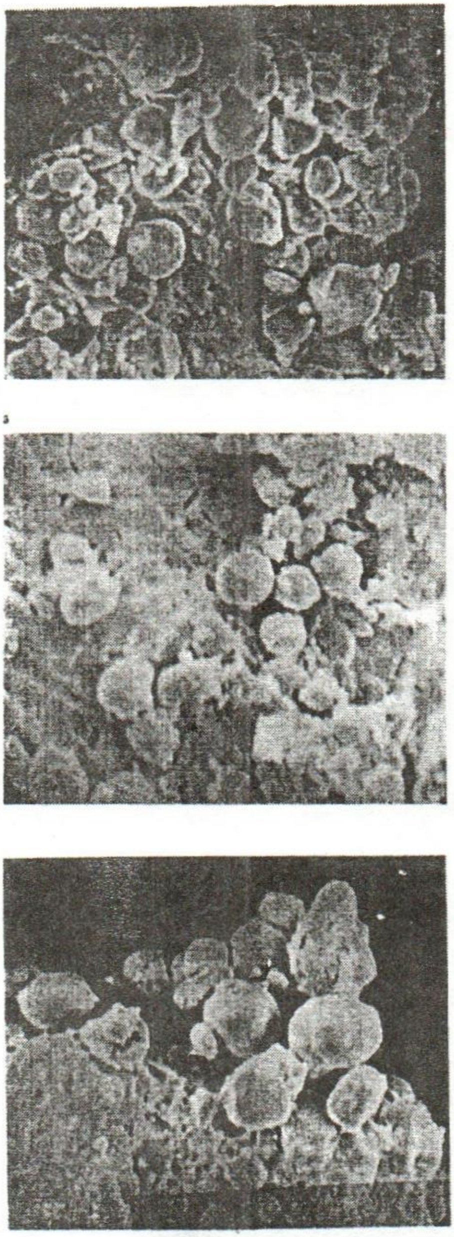
Starch structure:

The structural changes of the grains during processing affect the functional properties of the final nixtamalized product. These changes could be documented with the scanning electronic microscope (SEM) which has some advantages over use of the light microscope (Larry and Snyder, 1984).

Figs (4-8) are electronic photomicrographs of WC 10, YC 152 and DS which represent the different steps of tortilla chips production. It could be noticed that raw samples for every one of the three samples (Fig. 4) showed a mixture of round granules from the floury endosperm and angular granules from the horny endosperm. The more spherical granules usually have smooth or more regular surfaces compared to those of the angular granules, which are grooved or dimpled (Larry and Snyder, 1984). Similar description was previously given to native corn starches (Gerard *et al.,* 2001).

Starches are gelatinized by cooking. Depending on the gelatinization conditions, the degree of granular disruption and swelling can very widely Fig. (5) illustrate starch granules of different samples that were cooked in $Ca(OH)_2$ to produce nixtamal. The SEM graphs showed that nixtamalized kernels of corn grains exhibited swelling and hydration of starch granules. The nixtamalized sorghum grains had a variety of starch granules, nearly all of which have collapsed during the cooking process. The dimples and pits often present on raw starch granules (fig. 4) are no longer visible in the nixtamalized product (Fig. 5). When the nixtamalized dried corn kernels were milled for microscopic examination the starch granules were released from the protein matrix cells while some remained packed in the original form of the endosperm cells. On the contrary, gelatinized starch granules of dried nixtamal sorghum which were released from the corn cell were aggregated together forming dough-like structure .

Photographs given in Fig. (6) represent masas structure. Irregular and swollen starch granules were observed in masa samples. Grinding of masa resulted in the disruption the grain structure, releasing starch granules from the endosperm cells and dispersing cellular components and starch polymers (Gomez *et al.,* 1990). Swollen and partially gelatinized starch granules act as deformable particles in a net work of dispersed starch polymers allowing tortilla shaping during kneading gas retention and puffing during baking. A cohesive, glue like structure appeared to hold the masa pieces together.

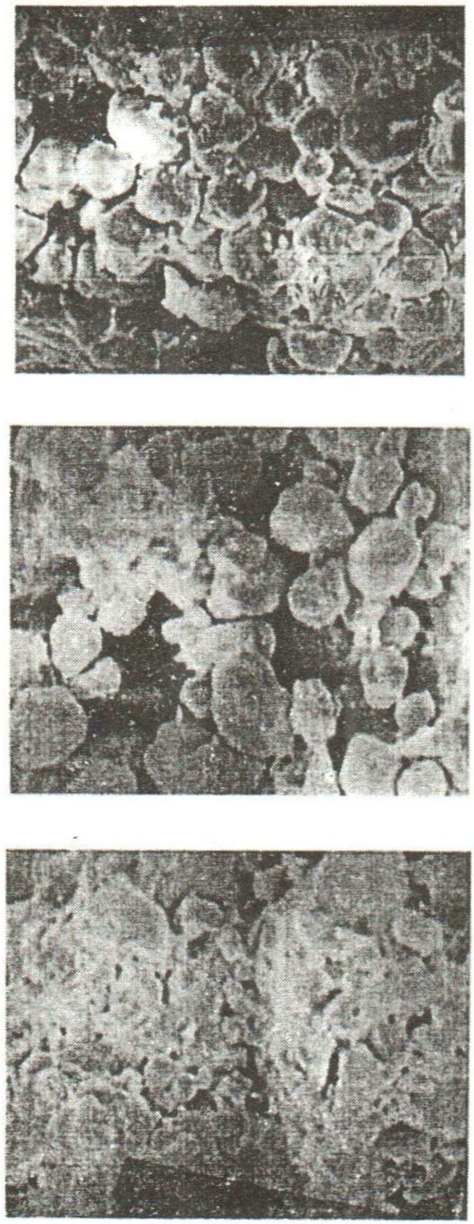


White

Yellow

Sorghum

Fig. (4): Scanning electron micrographs of raw flour samples.

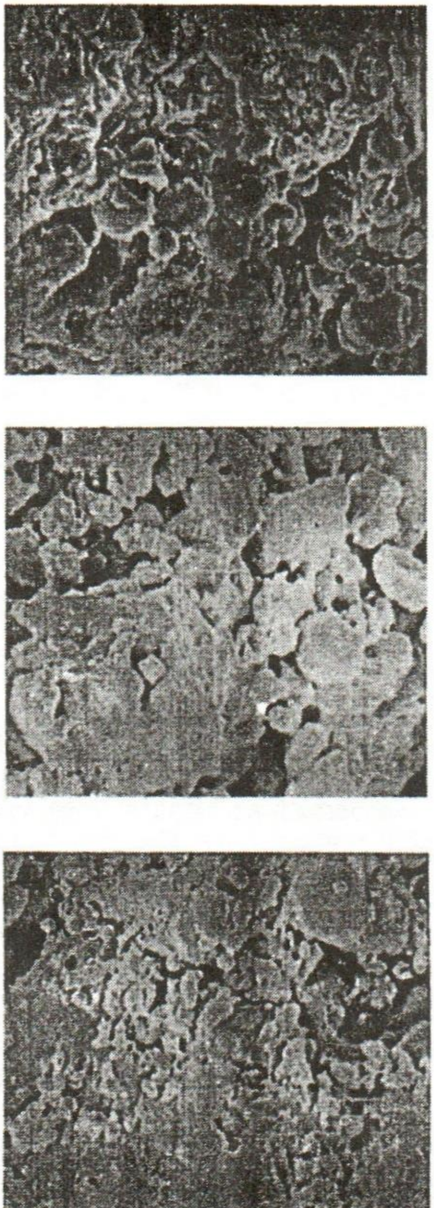


White

Yellow

Sorghum

Fig. (5): Scanning electron micrographs of nixtamals flour samples.



White corn

Yellow corn

Sorghum

Fig. (6): Scanning electron micrographs of masas flour samples.

This glue probably is composed of a mixture of gelatinized and dispersed starch hydrated and denatured protein matrix and free and emulsified lipids (Gomez *et al*, 1992).

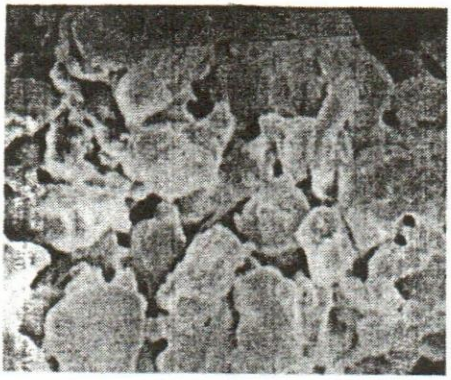
SEM evaluations of different tortilla (Fig. 7) had showed misshape starch granules along with more uniform, smoothes and more amorphous appearance of starch granules. These microstructure changes were due primarily to intense heat of baking, which caused fast water evaporation from the tortilla surface leading to a sever starch gelatinization. The sorghum tortillas seemed to be more severely affected by heat than the corn as indicated by the smoother and apparently more gelatinized starch. The starch granules in both sorghum and corn tortillas were shaped very irregularly and were swollen .

The SEM picture of tortilla chips (Fig. 8) indicates that most of the starch granules were completely gelatinized losing their smoothness. The most of changes mentioned for tortilla could be reported that for tortilla chips where starch granules were misshapen, uniform and had amorphous appearance. However, the granules were larger than those of tortilla . Tortillas are exposed to very high temperatures (180°C) in a nonaqueous medium during frying. Avery low moisture content in the tortilla chips results after frying. Ultimately, the tortilla structure is oily, dehydrated and set. Additional starch gelatinization occurs during the first 10-15 Sec of frying before most of the water is removed. Interactions between amylose and lipid during frying reduce the potential for dispersion of starch (Gomez *et al.*, 1992).

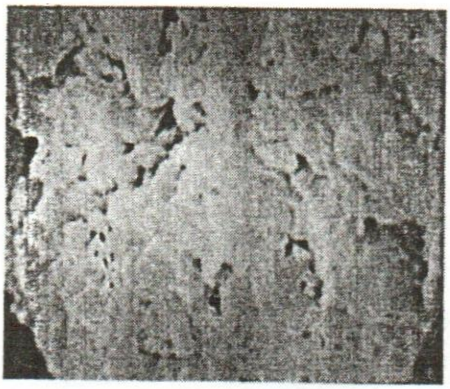
Starch crystallinity:

Changes of organized crystalline of raw starch granules during tortilla chips production could be demonstrated by x-ray technique. Starch granules are known to vary in their proportion of amylose and amylopectin, crystallite type (Zobel, 1988) and extent of amylopectin branching (De Boer, 1991), so X-ray diffraction patterns of such granules are subsequently varied. According to X-ray diffraction data, the structure of starch can be grouped into four types; A, B, C and V (Zobel *et al.*, 1988).

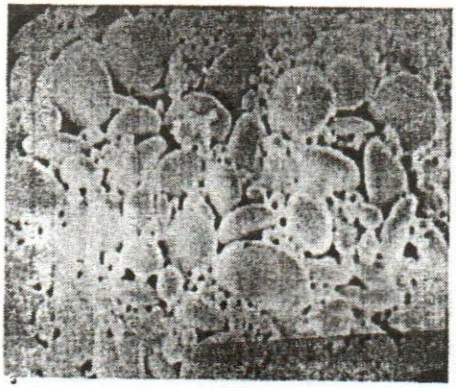
Figs (9, 10 and 11) show the diffractograms of analyzed samples and their respective crystallinity value. The X–ray diffraction trace of the raw WC (Fig. 9) shows sharp diffraction peaks around 2θ value of 17.5, 20 and 26.5 A, corresponding to d-spacings of about 5.8, 5.1 and 3.9 A, respectively. This pattern closely matches reported values of A-type cereal straches (Zobel, 1988). However, additional peak was observed at 2θ value of 11°A (9.4 ° d-spacing value). The diffractogram of raw YC (Fig. 10) shows similar peaks with some shifting where these peaks appeared at 2θ value of 18, 21 and 27 ° A, indicating a d-spacing value of 5.7, 4.9 and 3.8 °A , respectively. Also, another peaks were observed at 2 θ value of about 11.50, 16.0, 25 and 35.5 ° A. Concerning raw sorghum (Fig. 11), the main peaks of A-type starches were observed with some shifts as well as additional peaks were recorded at d-spacing value of 14.4, 9.9, 8.0 and 4.4°A. The shifts in the main peaks in the scattering profils of different starches indicating some change in lamellar spacing without changes in the type of starch (Serna-Saldivar *et al.*, 1990).



White

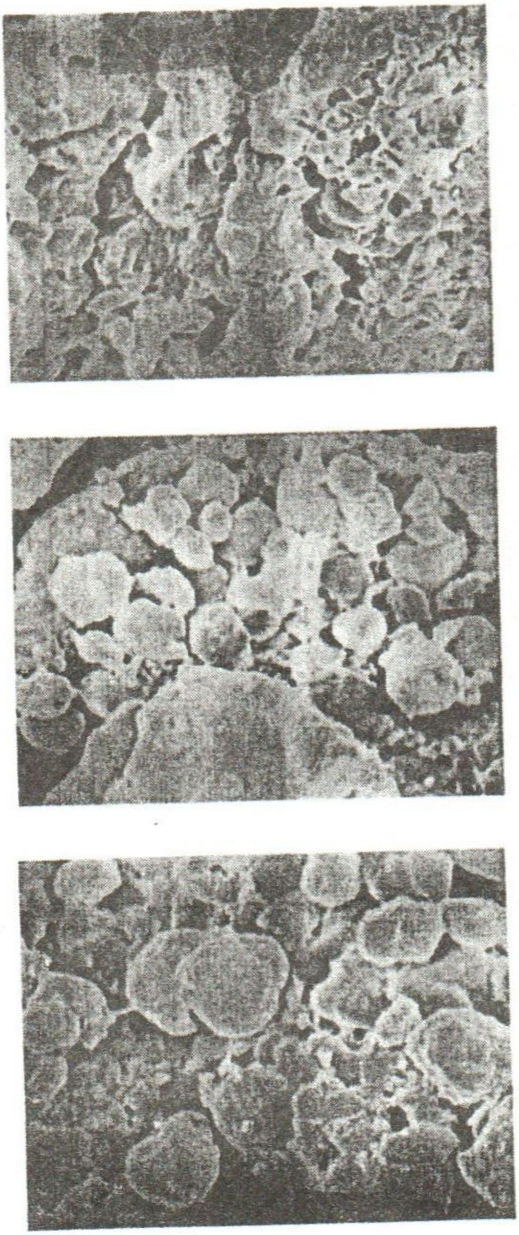


Yellow



Sorghum

Fig. (7): Scanning electron micrographs of tortillas flour samples.



White corn

Yellow corn

Sorghum

Fig. (8): Scanning electron micrographs of tortilla chips flour samples.

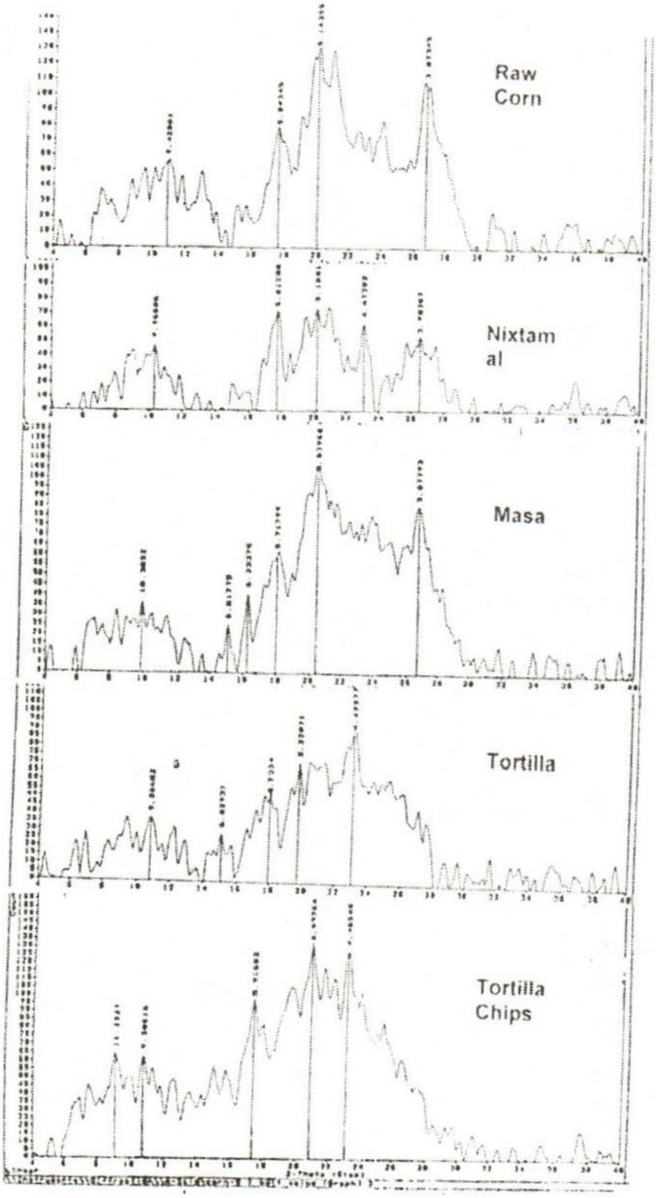


Fig. (9): X-ray diffractograms of raw white corn, mixtamal, masa, tortilla and tortilla chips. Maximum intensity=2000 count per second.

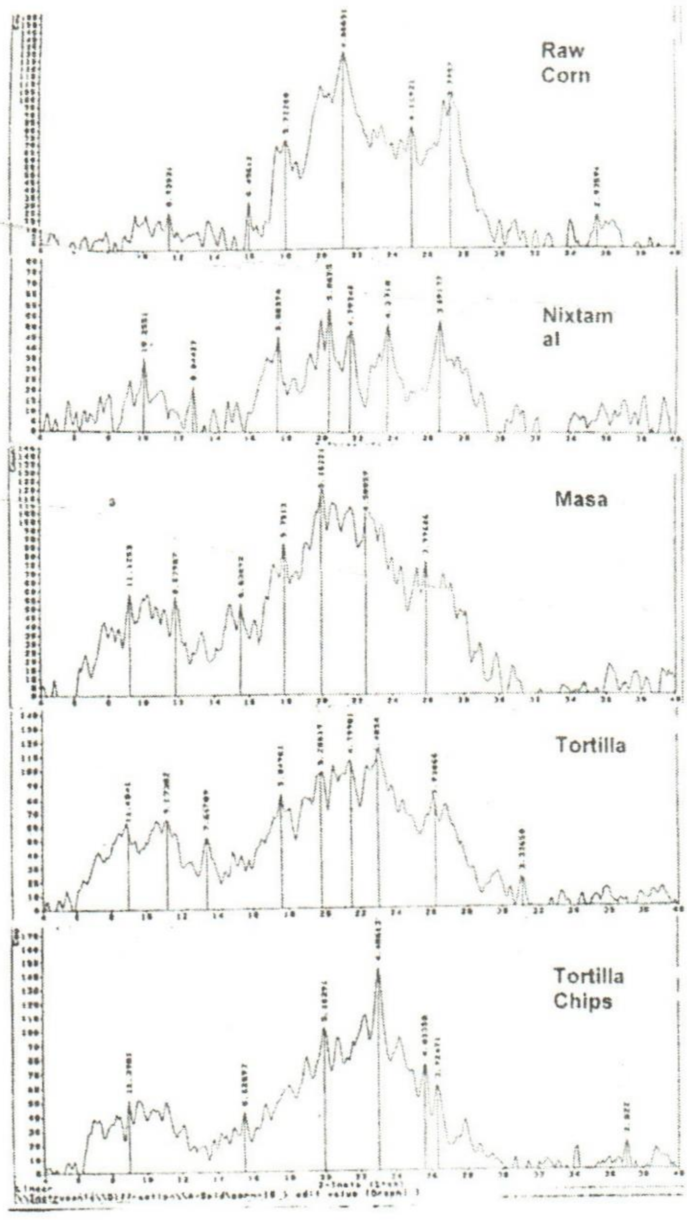


Fig. (10): X-ray diffractograms of raw yellow corn, mixtamal, masa, tortilla and tortilla chips. Maximum intensity=2000 count per second.

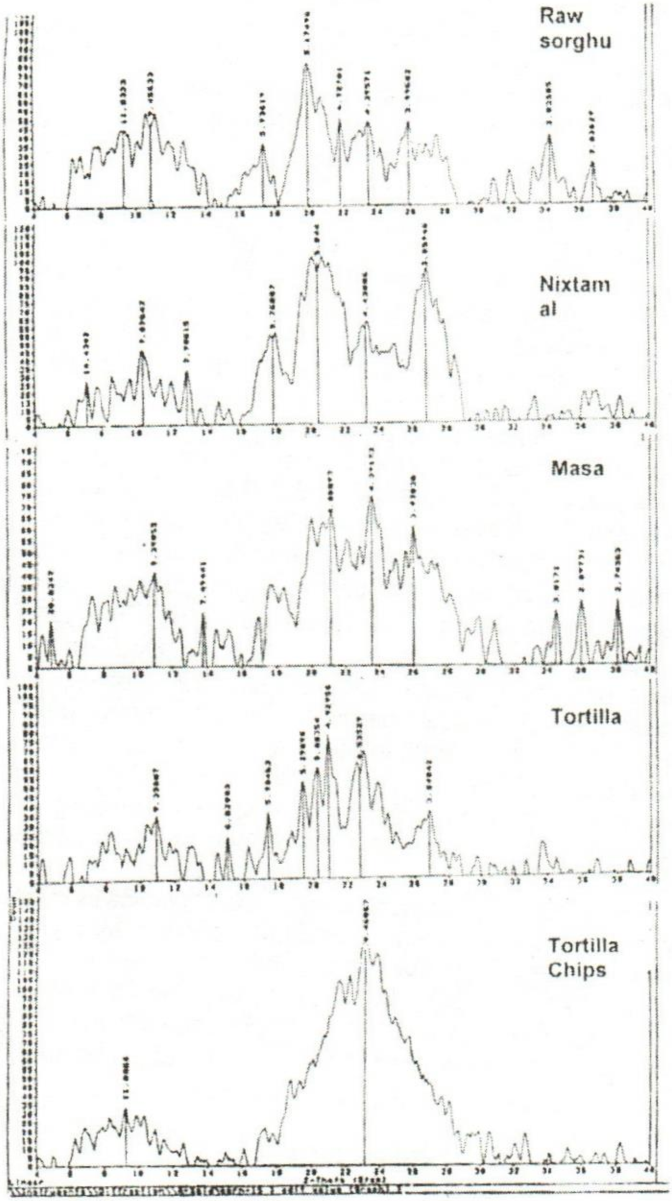


Fig. (11): X-ray diffractograms of raw Dorado sorghum, mixtamal, masa, tortilla and tortilla chips. Maximum intensity=2000 count per second

The X-ray diffraction patterns of the nixtamal samples (Figs 9, 10 and 11) revealed that structure of the native starch granule is partially disrupted, where a less organized x-ray pattern is observed along with a development of a diffraction peak at about 4.4 ˙A in the corn samples (Fig. 9, 10), whereas this peak was observed in the raw sorghum (Fig. 11). This peak is the distinguishing feature of v-type starch (Arambula *el al.*, 1998). From the same figs, it is clear that the intensity of the sharp peaks observed for raw samples is decreased in the nixtamal samples, indicating that the degree of crystallinity decreased with respect to that in the amorphous phase as mentioned by Arambula *et al.* (1998). This indicates that A-type cereal starches retain their initial "A" X-ray diffraction pattern, but the intensity falls as granules gradually loss their crystallinity during the treatment (Galliard,1987). In this concern, Gomez (1988) reported that during cooking the structure of the native starch granule is partially disrupted, whereas steeping results in a more like that of the native starch . Also, Serna-Saldivar *et al.* (1990) stated that alterations in starch crystallinity caused by cooking are partially restored by recrystallization during steeping.

Grinding the nixtamal to produce masa did not cause significant changes in corn starch crystallinity (Figs 9, 10) where the main peaks of A-type starch were presented at d-spacing of 5.7, 5.0 and 3.9 for WC sample and at 5.8, 5-2 and 3.9 d-spacing for YC sample. How ever, developed diffraction peaks were observed at about 15 and 16 ˙A for WC and at 12 and 16Ȧ for YC. It is of important to mention that the line at 12 Ȧ is another feature of v-patterns of starch (Arambula *et al.*, 1998). The 12Ȧ line has been observed before on starch processed under selected treatments that include combinations of moisture and temperature (Zobel, 1988). For sorghum samples (Fig. 11), it could be observed that the main peaks of raw sample (5.8 and 5.0Ȧ), disappeared in masa sample indicating that marked changes in starch granule were occurred.

Masas were exposed to high temperatures during baking into tortilla and frying into tortilla chips. Most of the starch crystallinity of raw corn was lost during baking and frying where a sharp, defined peaks partially disappeared and abroad (halo) appeared (figs 10&11) indicating a change of starch from a crystalline to an amorphous state (Zobel, 1988). Accordingly, the material is no longer a rigid but rather exhibits the properties of a liquid (Chakraverty & Kaleemulla, 1991). Sorghum tortilla chips displayed another amorphous X-ray pattern with a peak around 4.5Ȧ. The location of this peak was slightly displaced from the strong 4.4Ȧ peak characteristic of the v-type amylose-lipuid complex pattern (Gomez *et al.*, 1992).

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إنتاج شيبس الطرطية من الذرة/السورجم ٢. تقييم جلتنة وبلورة النشا

على شلبى*– نفيسة حجازى*– سمية عبد المنعم – فوزى سالم** – أحمد حسين***

* قسم الصناعات الغذائية – المركز القومى للبحوث

** قسم علوم الأغذية – كلية الزراعة – جامعة الزقازيق – الزقازيق – مصر

يصنع شبسى الطرطية من حبوب الذرة الشامية والذرة الرفيعة وذلك بطبخها فى محلول قلوى وفرمها وتشكيلها ثم خبزها وقليها . أثناء المراحل التصنيعية السابقة يحدث تغيرات فى درجة جلتنة حبيبات النشا وشكلها البللورى يتوقف عليها جودة المنتج النهائى . أجريت هذه الدراسة لتتبع جلتنة حبيبات النشا والتغير فى شكلها البللورى أثناء تصنيع شبسى الطرطية . وقد أمكن – باستخدام ميكروسكوب الضوء المستقطب – التعرف على ظاهرة الـ birfringence (الشكل الكروى المضئ المحتوى على شرائط صليبية غامقة) فى حبيبات النشا الخام . أثناء مراحل التصنيع تتشرب حبيبات النشا الماء وتنتفخ وتلتصق ببعضها وتفقد تدريجيا ظاهرة الـ birfringence . أظهر الفحص بالميكروسكوب الإلكترونى أن حبيبات النشا الخام عبارة عن خليط من أشكال بللورية كرويه ومنشورية ، تتحول نتيجة الطبخ والفرم إلى الشكل غير المنتظم ذات المظهر الصمغى اللزج نتيجة لإنتفاخها وتكسيرها . أما الخبيز والقلى فقد أدى إلى تكوين بللورات ملساء مشوهة غير متماثلة فى الحجم . أظهر الفحص بالأشعة السينية أن النشا الخام يحتوى على بللورات من النوع A ، تتحول الى النوع V نتيجة الطبخ ، أما نشا العجينة فقد احتوى على النوعين المذكورين . وقد فقدت حبيبات النشا شكلها البللورى تماما نتيجة عمليتى الخبيز والقلى .