

# Microstructure of a Third Generation Snack Manufactured by Extrusion from Potato Starch and Orange Vesicle Flour

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## Abstract

The objective of this work was to evaluate the effect of extrusion on microstructural properties of a third generation snack food expanded by microwaves manufactured from orange vesicle flour, commercial nixtamalized corn flour, and potato starch. A Brabender 20DN laboratory extruder was used to get the pellets (unexpanded extruded products). Viscosity profiles (RVA), scanning electron micrographs (SEM), X-ray diffraction patterns, and thermogram data (DSC) were obtained. Analyses were made on raw materials, unprocessed mixture, extruded pellets, and microwave expanded products to study the native starch structure changes. Analyses suggest that the snacks obtained by the extrusion process were modified to a desirable microstructure for achieving physicochemical properties necessary for acceptance by the consumer.

**Keywords:** Microstructure; Extrusion; Snack; Biopolymers

## Introduction

Extrusion is a continuous thermo-mechanical process where materials containing biopolymers are plasticized and cooked by the combined action of pressure, heat, and shear stress [1]. This thermo-mechanical process is very useful in producing low-fat snacks and has the advantage of increasing protein and starch digestibility, solubilizing fiber, inactivating toxins, anti-nutritional factors, and undesirable enzymes, such as lipo-oxygenases and peroxidases [2]. In addition, microwaves have been widely used for expanding this type of snacks and some researches on 3G snacks have focused on the effect of processing on different physical and physicochemical properties [3,4]. During extrusion, raw materials experience chemical and structural transformations such as starch gelatinization, protein denaturation, complex formation between amylose, lipids and/or proteins, and degradation of pigments and vitamins [5], but starch is the most important component due to the changes it undergoes by the thermal process as it affects expansion and final texture of the extrudate. Starch gelatinization can occur at levels from 12 to 22% moisture content; however, it has been indicated that at low moisture contents, gelatinization is accentuated because of the high shear stress, the heat generation, and the mechanical disruption of the granules [6]. Lee, et al. [4] mentioned that a partial gelatinization ( $\approx 50\%$ ) is necessary for obtaining third generation snack foods (pellets), and a further degradation will reduce the size of sugar chains and thus the product stability after expansion will be lost. However, a lesser degradation will not be enough for opening the starch granules reducing the ability to adsorb water, which serves later as a means for expansion. For these reasons, it is important to know the microstructural changes after the extrusion process and microwave expansion of the pellets. Likewise, in Mexico, the citrus industry generates large amounts of residues that are normally discarded in landfills and left to decompose. Including these residues in technological processes can add value for industry and reduce contamination generated by residue decomposition; thus reducing their impact on ecosystems. Added to this, the use of by-products derived from the citric industry and mainly staple vitamins and fibers contained in the juice vesicle of oranges can be increased improving their functional properties. Dietary fiber acts as a bulking agent, normalizing intestinal motility and preventing

diverticular disease. Some types may also be important in lowering serum cholesterol levels, in reducing colonic cancer and in preventing hyperglycemia in diabetic patients [7-9]. The objective of this work was to evaluate the effect of extrusion on microstructural properties of a third generation snack food expanded by microwaves manufactured from orange vesicle flour, commercial nixtamalized corn flour, and potato starch.

## Materials and Methods

### Raw material preparation

Orange vesicles were dried and milled in a hammer mill (Laboratory Mill 3100 Perten, Ireland). Commercial nixtamalized corn flour 10% (NCF), potato starch 79.9% (PS), and orange vesicle flour 10% (OVF) were homogeneously and mixed with 0.1% monoglycerides. This formulation was obtained in preliminary studies.

### Extrusion process

Extrusion was carried out in a laboratory extruder (Brabender 20DN, 8-235-00, Brabender OHG, Duisburg, Germany). A rectangular matrix with internal dimensions of 20 mm wide  $\times$  1.0 mm high  $\times$  100 mm long was used. Screw speed was 1.08 Hz (65 rpm), using a 2:1 L:D screw ratio and a feed rate of 33 g/min (dry matter), [0.42 Hz (25 rpm)]. Temperatures at the feed and out zones were 60 and 75°C. The transition zone temperature was 130°C and moisture content was 23%; these conditions were obtained from an optimization process

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by response surface methodology considering expansion index, bulk density, penetration force, and total carotenoid content [10]. The extruded material was cut manually into approximately 1.5 cm long pellets, and dried at room temperature for approximately 24 h until reaching 9-13% moisture content.

### Pellet expansion by microwave heating

Extruded products (pellets of 1.5 cm long) were expanded in a microwave oven (LG, R-501CW, 900 W, 2450 Hz) for 28 s. This condition was determined through expansion kinetics at different heating times [10].

### Rapid visco-analyzer

Viscosity properties were evaluated using a Rapid Visco-Analyzer (RVA, 3C, Newport Scientific PTY Ltd., Sydney, Australia) following the instruction manual and suggestions from Aguilar-Palazuelos, et al. [11]. Two grams of sample were diluted with distilled water to get 28 g in the sample aluminum cup. Sample was continuously agitated and heated at 50°C for 1 min. Temperature was raised to 92°C at 5.5°C min<sup>-1</sup>, held at 92°C for 5 min, lowered from 92 to 50°C at a rate of -5.6°C min<sup>-1</sup>, and held at 50°C for 2 min (Newport-Scientific 1992). From RVA amylograph profiles, the next viscosities were measured: initial viscosity ( $V_{ini}$ ), viscosity at 92°C ( $V_{92}$ ) (maximum temperature), minimum viscosity ( $V_{min}$ ) at 92°C, and final viscosity ( $V_{fin}$ ) (higher viscosity for the cooling period). From these values, total retrogradation viscosity ( $V_r$ ) (final viscosity minus minimum viscosity) was calculated [12].

### Scanning electron microscopy

Analyses were made in raw materials, unprocessed mixture, extruded pellets, and microwave expanded products, according to methodology described by Zazueta-Morales, et al. [13] and Aguilar-Palazuelos, et al. [14]. A scanning electron microscope model JSM-6300 was used; a secondary electron detector and electron bombardment at 15 kV were used; samples were placed at high vacuum. The milled samples (60-mesh) were mounted on an aluminum 12 mm diameter holder, PIN type, previously prepared with carbon conductive double coated tapes and colloidal silver adhesive. Morphologies and particle size were observed.

### X-ray diffraction

Analysis was performed on milled samples which pass through a 60-mesh sieve (250 μm), at moisture content between 9 and 13%. Samples were packed in a glass slide (0.5 mm deep) and mounted on a Rigaku X-ray diffractometer (Ultima D/Max-2100, Rigaku Denki Co. Ltd, Japan) according to procedures described by Zazueta-Morales, et al. [13]. Analyses were made in raw materials, unprocessed mixture, extruded pellets, and microwave expanded products, in order to determine the effect of processing on the crystallinity of starches.

### Differential scanning calorimetry

Analyses were performed in raw materials, extruded pellets, and microwave expanded products. A calorimeter (Mettler-Toledo 821 e, Columbus, OH) was used following recommendations from Toro-Vazquez, et al. [15]. Samples of about 4 mg were added with 16 μl of distilled water and hermetically sealed in aluminum pans. Subsequently, they were vigorously shaken and allowed to stand for 18 h. Pans were heated from 40 to 100°C at a heating rate of 5°C/min; the heating chamber was vented with nitrogen at a flow rate of 20 ml/min. The enthalpy change, initial temperature (onset), (maximum)

peak temperature, and the final temperature were determined, using the software STARe Thermal Analyzer.

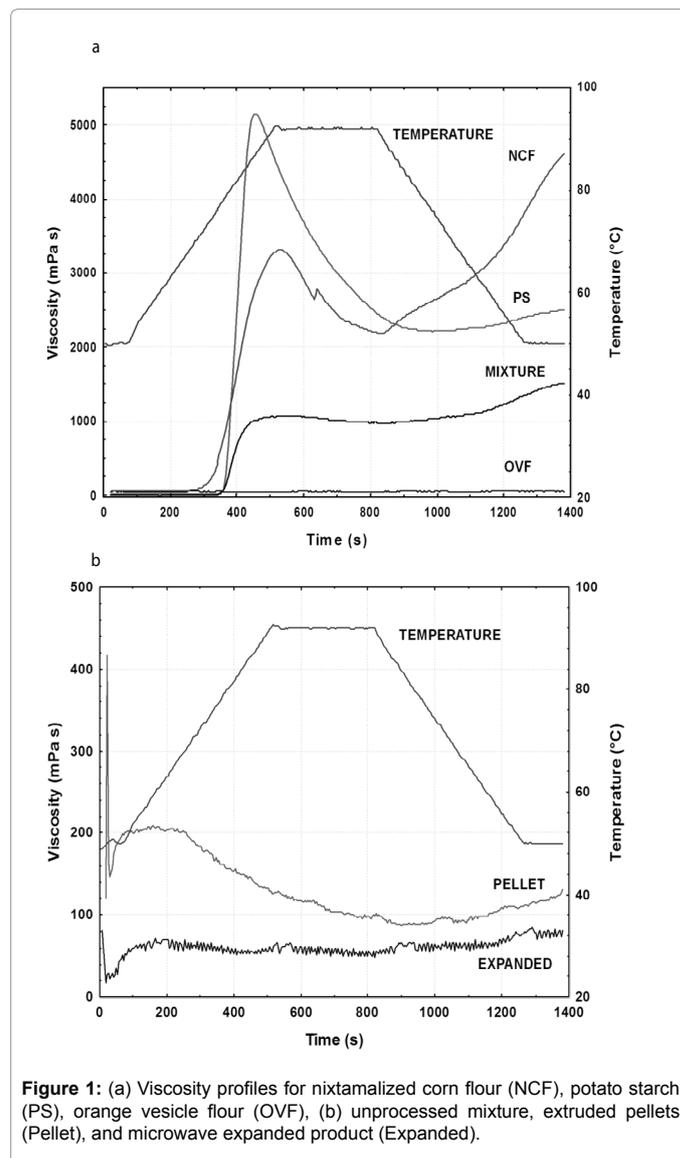
## Results and Discussion

### Rapid visco-analyzer

Viscosity properties (Table 1) and viscosity profiles (Figure 1) are

Material	Viscosity (mPa s)				
	$V_{ini}$	$V_{92}$	$V_{min}$	$V_{fin}$	$V_r$
NCF	47	3114	2191	4613	2422
PS	7	4809	2395	2404	9
OVF	37	43	43	49	6
Mixture	7	1059	981	1516	535
Pellet	120	133	97	127	30
Expanded	17	58	61	92.15	31.15

**Table 1:** Initial viscosity ( $V_{ini}$ ), viscosity at 92°C ( $V_{92}$ ), minimum viscosity ( $V_{min}$ ) at 92°C, final viscosity ( $V_{fin}$ ), and total retrogradation viscosity ( $V_r$ ) for nixtamalized corn flour (NCF), potato starch (PS), orange vesicle flour (OVF), unprocessed mixture, extruded pellets, and microwave expanded product.



**Figure 1:** (a) Viscosity profiles for nixtamalized corn flour (NCF), potato starch (PS), orange vesicle flour (OVF), (b) unprocessed mixture, extruded pellets (Pellet), and microwave expanded product (Expanded).

shown for commercial nixtamalized corn flour (NCF), potato starch (PS), orange vesicle flour (OVF), unprocessed mixture, extruded pellets, and microwave expanded products. Viscosity of nixtamalized corn flour and potato starch increased from 47 ( $V_{ini}$ ) to 3114 ( $V_{92}$ ) mPa s and from 7 to 4809 mPa s, respectively. The difference between NCF and PS viscosity may be due to chemical composition and the previous thermal treatment NCF has undergone; NCF contains gums and during the nixtamalization process, the more susceptible starch granules are partially gelatinized, which are generally the larger ones, remaining the most resistant granules practically unchanged [16]. This may explain why NCF showed a lower viscosity peak ( $V_{92}$ ) of 3114 mPa s than PS of 4809 mPa s. Maximum viscosity ( $V_{92}$ ) of PS is similar to that reported by Alvis, et al. [17]. Orange vesicle flour did not develop a maximum peak viscosity ( $V_{92}$ ) during heating.

The unprocessed mixture showed a peak viscosity ( $V_{92}$ ) of 1059 mPa s; this lower viscosity, compared to NCF and PS viscosity, may be due to the presence of fiber, affecting the development of viscosity and the type of link that hinder viscosity development and gel formation. Another reason may be water competition as pericarp can be chemically modified turning fiber from insoluble to soluble and competing for water; also gums in the mixture can be interacting with potato starch. The mixture showed minimum viscosity at 92°C ( $V_{min}$ ) of 981 mPa s, which is lower than that for raw materials; this could be due to fiber affects starch gelatinization as starch molecules become less soluble and tend to aggregate [14].

Nixtamalized corn flour had a  $V_{min}$  of 2191 mPa s at min 14 and retrogradation started from this point. In like manner, potato starch had a  $V_{min}$  of 2395 mPa s at min 14 and retrogradation started at minute 18. This time difference for NCF and PS to start retrogradation could be due to amylose retrogradates faster, since as a result of its linear nature and polarity tends to form hydrogen bonds between hydroxyl groups of adjacent molecules (amilose and amilopectin); therefore, they start losing their hydration capability, which yields a partial shrinkage of starch [16,18].

In viscosity profiles for extruded pellets and microwave expanded products (Figure 1b), a slight increase in viscosity during retrogradation was observed, reaching their maximum viscosity at 50°C, after cooling. This result is similar to that reported by Zazueta-Morale, et al. [13] and Aguilar-Palazuelos, et al. [14]. These authors mentioned that the differences having an extruded starch, relative to a native starch, are the absence of a gelatinization peak, a high initial viscosity and a continuous fall in viscosity from 50 to 92°C. In addition, during cooling, a slight or no increase in viscosity is obtained, achieving its maximum value at 50°C. Similarly, the temperature used during extrusion (130°C) may have caused the dough to become less viscous, allowing the molecules become more susceptible to shearing action. In this way, a higher thermal and mechanical action is achieved, resulting in further degradation of starch and consequently lowering viscosity. This result is similar to that reported by Carvalho, et al. [19], who stated that viscosity is affected by the extrusion temperature (above 80°C) and moisture content. Lee, et al. [4] and Delgado-Nieblas, et al. [20] reported that the best physicochemical properties of third generation products, processed by extrusion, occur when there is a partial degradation of starch, which is confirmed by the above. The physicochemical (functional) properties of extruded starch products depend on the rheology of both the material during processing and the final product, since rheology is highly related to the microstructural changes the product underwent and hence to the ability the material

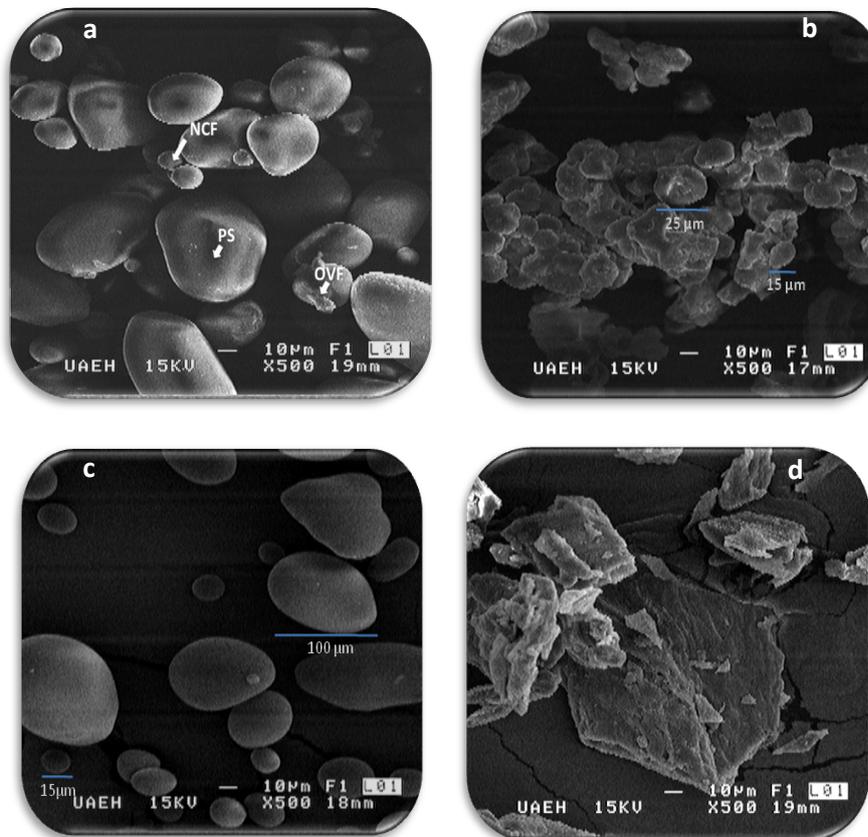
has to interact between the polymeric matrix and the other components of the extruded product.

### Scanning electron microscopy

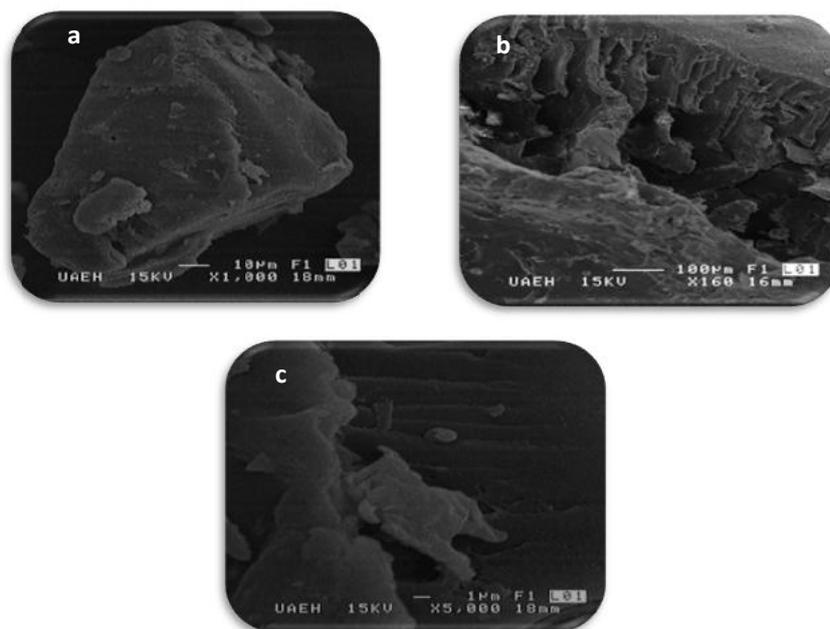
Figure 2 displays micrographs of raw materials (nixtamalized corn flour, potato starch, and orange vesicle flour) and unprocessed mixture used in the extrusion process. In the unprocessed mixture, different sizes of starch granules are observed, with circular and oval shapes due to the diversity of used materials. In addition, the presence of fiber attached to the starch granules can be seen. The corn flour starch granules are smaller than those of potato, with diameters from 15 to 25  $\mu$ m and from 15 to 100  $\mu$ m, respectively (Figure 2b and 2c). Similar data were reported by Medina and Salas [21], who reported that corn starch granules have a smaller size (2-30  $\mu$ m) than those of PS (5-100  $\mu$ m). Figure 2d shows the fiber fragments of OVF. Micrographs of extruded pellets obtained using the optimal processing conditions (130°C and 23% moisture content) are shown in Figure 3. It can be distinguished the starch granules were melted and plasticized; furthermore, certain channels and holes are observed in the product surface, which may be due to some leakage of water from the material, when leaving the extruder, as reported by Zazueta-Morales, et al. [13]. This type of structure is similar to that reported by Aguilar-Palazuelos, et al. [11] for third generation snacks. Also, it can be seen that pellet fragments are extended (Figure 3a and 3b); this may be due to an alignment effect of the short chain starches. The extrusion process is able to break the covalent bonds in biopolymers and both the intense structural disruption and mixing facilitate reactions that in other processes are limited [22]. Similarly, it appears that these products are covered by certain particles (Figure 3c), which can be very small size particles of the same material as suggested by Aguilar-Palazuelos, et al. [14]. Figure 4 shows the micrographs of the microwave expanded product. A porous (air cells with thin walls) and extended structure can be viewed (Figure 4a and 4b). This structure is very similar to that reported by Lee, et al. [4], with air cells of uniform size, for third generation products expanded by microwaves. This pattern can be caused by a sudden release of pressure generated by steam inside the pellet. Pellets were expanded by microwave, which caused the trapped water inside the extruded began to evaporate, creating an increase in pressure inside the product. When the internal pressure was high enough, the internal structure of the pellet could no longer endure, so that exploded and expanded, causing the air cells of the expanded product were elongated and with smooth surface walls with an approximate size of 1 mm (Figure 4a and 4c). The holes on the surface could be produced during the escape of steam; this is similar to that reported by Moraru and Kokini [23].

### X-ray diffraction

X-ray patterns of nixtamalized corn flour, potato starch, orange vesicle flour, unprocessed mixture, extruded pellet, and microwave expanded product are shown in Figure 5. Nixtamalized corn flour showed an A-type diffractogram, which is typical for cereal starches, having two main peaks at approximately  $2\theta$  of 16 and 21 Å. These peaks have been reported by Zazueta-Morales, et al. [13] and Aguilar-Palazuelos, et al. [11]. For potato starch and the unprocessed mixture B-type patterns were obtained, which are typical for tubercle starches; the latter behavior was expected as the main component in the mixture was potato starch [20]. Extruded pellets and expanded products showed an amorphous structure. The presence of amorphous structure in extruded materials, at temperatures near 70°C and moisture contents above 20%, have been reported by Aguilar-Palazuelos, et al. [11]. These amorphous structures apparently occur because these



**Figure 2:** Scanning electron micrographs of (a) unprocessed mixture, (b) nixtamalized corn flour (NCF), (c) potato starch (PS), and (d) orange vesicle flour (OVF) used for the extrusion process.



**Figure 3:** Scanning electron micrographs of milled extruded pellets obtained using the optimal processing conditions, 130 °C and 23% moisture content; a, b, and c correspond to different zones of the pellet.

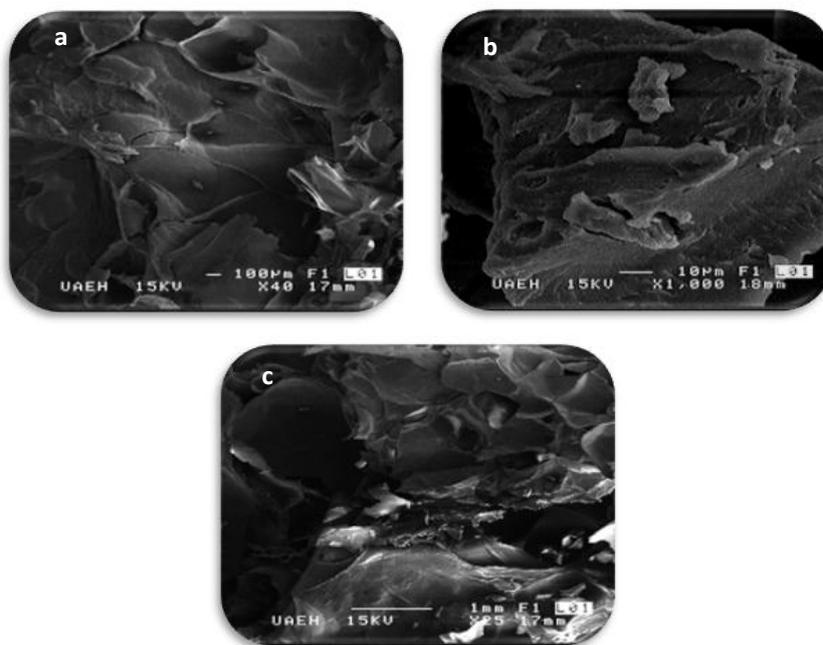


Figure 4: Scanning electron micrographs of the microwave expanded product; a, b, and c correspond to different zones of the expanded product.

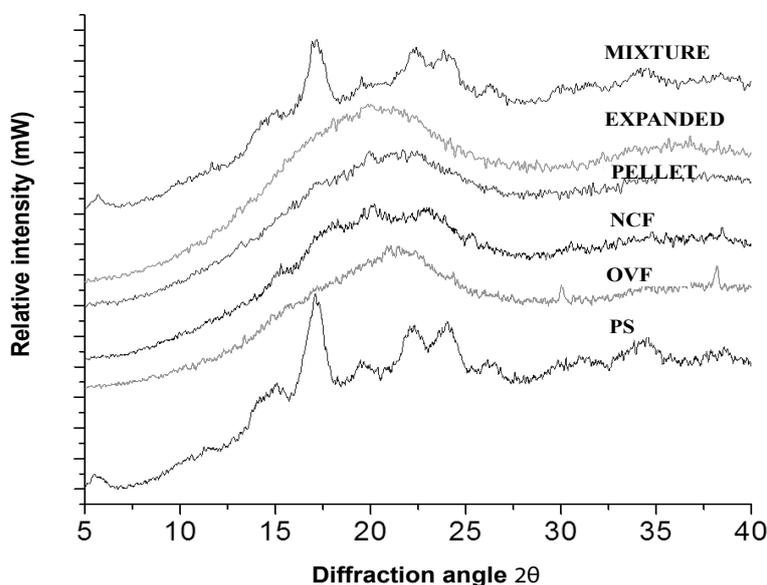


Figure 5: X-ray diffraction patterns of nixtamalized corn flour (NCF), potato starch (PS), orange vesicle flour (OVF), unprocessed mixture, extruded pellet, and microwave expanded product.

conditions are enough for modifying the native crystalline structure, but are not sufficient for the formation of new crystalline structures. Extrusion processing can partially or totally break the crystalline structure of starch, depending on the extrusion conditions such as moisture content, shear stress, and temperature. High temperatures can completely destroy starch structure, leading to the formation of an X-ray diffractogram typical of an amorphous state or can induce the formation of a new structure [24]. During extrusion, extruded materials show large amounts of plasticized and slightly damaged granules [25].

#### Differential scanning calorimetry

The thermogram data of raw materials [nixtamalized corn flour (NCF), potato starch (PS), orange vesicle flour (OVF)], unprocessed mixture, extruded pellet, and microwave expanded product are shown in Table 2. Nixtamalized corn flour had temperatures of 65.2°C for onset ( $T_i$ ), 68.5°C for peak ( $T_g$ ), and 69.7°C for final temperature ( $T_f$ ). Potato starch and unprocessed mixture showed 59.5, 63.7, 69.5°C and 61.3, 64.9, 69.7°C for  $T_i$ ,  $T_g$ , and  $T_f$  respectively. Temperatures for

	$T_i$ (°C)	$T_g$ (°C)	$T_f$ (°C)	$\Delta H$ (mJ)
NCF	65.2	68.5	69.7	$-15.8 \times 10^{-3}$
PS	59.5	63.7	69.5	-107.08
OVF	--	--	--	--
Mixture	61.3	64.9	69.7	-55.48
Pellet	--	--	--	--
Expanded	--	--	--	--

(-- No gelatinization peak),  $T_i$  is initial temperature [onset],  $T_g$  is gelatinization temperature,  $T_f$  is final gelatinization temperature [endset], and  $\Delta H$  is the transition enthalpy.

**Table 2:** Thermogram data of raw materials [nixtamalized corn flour (NCF), potato starch (PS), orange vesicle flour (OVF)], unprocessed mixture, extruded pellet, and microwave expanded product.

mixture are similar to those found for potato starch, which is attributed to potato starch is the main component in the mixture. The transition enthalpy was 26.8 and 13.9 J g<sup>-1</sup> for potato starch and unprocessed mixture. The orange vesicle flour, extruded pellet, and microwave expanded product did not show a significant gelatinization peak, which can occur because starch is gelatinized. These results agree with those reported by Maninder, et al. [26] and Aguilar-Palazuelos, et al. [11]. Lee et al. [27] reported that the degree of gelatinization of starch, extruded under critical conditions, was higher as the extrusion temperature was lowered. They found that the extrudates at 80°C showed a small peak of gelatinization, meanwhile, at 90 and 100°C no endothermic peak was found. In our study, extrusion was accomplished at 130°C and 23% moisture content and no gelatinization peak was observed in the extrudates.

## Conclusions

The analyzed optimum processing conditions were adequate for producing a third-generation snack from a mixture of corn starch and orange vesicle flour. These raw materials are a promising material for production of third-generation snacks and the analyses of the X-ray diffractograms, viscosity profiles, scanning electron microscopy and differential scanning calorimetry suggested that the extruded products presented changes mainly due to fragmentation, gelatinization and plasticization of the starch granule, causing the loss of the granular starch (modification in its native structure). The snacks obtained by the extrusion process and expanded by microwave were modified to a desirable microstructure for achieving adequate physicochemical properties for acceptance by the consumer.

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