Elaboration of functional snack foods using raw materials rich in carotenoids and dietary fiber: effects of extrusion processing


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Published online: 30 Jun 2014.


To link to this article: http://dx.doi.org/10.1080/19476337.2014.915892

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Elaboration of functional snack foods using raw materials rich in carotenoids and dietary fiber: effects of extrusion processing

Elaboración de alimentos botana funcionales utilizando materias primas ricas en carotenoides y fibra dietaria: efectos del proceso de extrusión


Abstract

This research studied the effect of extrusion temperature (ET, 93.45–140.55°C), moisture content (MC, 21.27–34.73%), and the winter squash flour content (WSF, 0.43–15.57%) on physicochemical characteristics and content of bioactive compounds of third-generation (3G) snack foods expanded by microwave heating. The ingredients used for their elaboration were corn starch, whole-grain yellow corn and winter squash flour. A single-screw extruder was employed, and the response surface methodology was applied. The lowest bulk density and the highest water solubility index (WSI) and water absorption index, occurred at high ET with low MC. The highest values of total carotenoids and dietary fiber (total and soluble) were obtained at high WSF and ET. Furthermore, when the WSF was increased, the color L* value diminished, whereas b* value and WSI increased. These results suggest that it is possible to elaborate 3G snack foods with acceptable physicochemical characteristics and excellent bioactive compounds content, improving their potential health benefits.

Keywords: functional snacks; extrusion; third-generation snacks; carotenoids compounds; dietary fiber

Introduction

Extrusion cooking technology is a versatile and efficient process for converting raw materials into finished food products. Food extruders provide thermo-mechanical energy (shear) needed to cause physico-chemical changes of foods, implying mixing and homogenization (Anton & Luciano, 2007). Extrusion technology plays a very important role in modern industrial production of snacks, especially those produced from corn, wheat, and rice. Snack foods are mainly made from cereals, and are widely available, especially those produced from corn, wheat, and rice. Extrusion cooking technology is a versatile and efficient process for converting raw materials into finished food products. It is relatively cheap and easy to prepare at home and, once expanded, the final products have low oil content (Bastos-Cardoso, Zazueta-Morales, Martinez-Bustos, & Kil-Chang, 2007). In the microwave heating, the microwave energy heats the pellet by vibrational energy directed to the moisture contained within. This heating generates the superheated steam that causes the pellets to expand and form a porous structure. Maximum expansion of 3G snacks takes place at 10–12% of moisture content (MC) of the pellets (Boischot, Moraru, & Kokini, 2003). There have been investigations where microwave has been used to expand this type of snacks and some reports about 3G snacks have focused on the effect of processing on different physical and physicochemical characteristics (Gimeno, Moraru, & Kokini, 2004; Lee, Lim, Lim, & Lim, 2000). In extruded snack foods, physical parameters as bulk density (BD), expansion index, and texture have shown to be appropriate quality parameters (Chessari & Sellahewa, 2000; O’Shea, Arendt, & Gallagher, 2013). On the other hand, other studies have focused on the improvement of nutritional or nutraceutical properties of 3G snacks, by incorporating raw materials

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that provide human health benefits (Aguilar-Palazuelos, Zazueta-Morales, & Martínez-Bustos, 2006; Limón-Valenzuela, Martínez-Bustos, Aguilar-Palazuelos, Caro-Corrales, & Zazueta-Morales, 2010). According to Brennan, Derbyshire, Tiwari, and Brennan (2013), nowadays a large production of ready-to-eat (RTE) snacks exist, which are relatively high in sugar and salt, thus being regarded as energy dense but nutritionally poor. However, there is a potential to manipulate the nutritional status of extruded RTE snacks by incorporating raw materials rich in bioactive components (such as carotenoids, dietary fiber, etc.). Among the raw materials which could contribute to improve the nutritional content of snacks are some pigmented maize varieties, including yellow corn. Yellow corn is a cereal that is an important source of carotenoids. Different authors have reported values between 14 and 77 µg of TC/g of sample (db) in different varieties of normal yellow corn (Safawo et al., 2010; Weber, 1987). However, in improved varieties of this type of cereal, values have been reported between 139.2 and 278 µg of TC/g (Burt, Caston, Leeson, Shelp, & Lee, 2013; Maziyva-Dixon, Kling, Menkir, & Dixon, 2000). The main carotenoids present in yellow corn are xanthophylls (lutein, zeaxanthin and β-cryptoxanthin) (De Oliveira & Rodríguez-Amaya, 2007). Carotenoids protect against age-related macular degeneration (AMD) which is the main cause of blindness in elderly people in the industrialized world (Krinsky, Landrum, & Bone, 2003). Another foods which could improve the nutritional content of the snacks are the vegetables, for example, winter squash. The winter squash (Cucurbita moschata D.) belongs to the family Cucurbitaceae, and is a good source of carotenoids, dietary fiber, and minerals (Jaco-bo-Valenzuela, Maróstica-Junior, Zazueta-Morales, & Gallegos-Infante, 2011). However, in Mexico it is mostly used for the preparation of regional sweets and for animal feed. It has been reported that the winter squash may have a total carotenoid (TC) content in fresh basis, between 24 and 84 µg/g (Rodríguez-Amaya, 1999). Jacobo-Valenzuela et al. (2011) reported TCs values between 160 and 1399.4 µg/g on a dry basis for pulp of winter squash, cv Cehuala, being β-carotene the main carotenoid. Ortiz-Grisales, Sánchez-Ledesma, Valdés-Restrepo, Baena-García, and Vallejo-Cabrera (2008), working with winter squash (C. moschata D.) dried by hot air, reported TCs values between 122 and 509 µg/g (db). The main carotenoids present in this winter squash, reported by the same authors, were β-carotene, α-carotene, and lutein. The β-carotene, α-carotene, and β-cryptoxanthin are the major provitamin A carotenoids found in most foods (Inocent, Aba-Ejoh, Some Issa, Schweigert, & Tchouanguep, 2007). Vitamin A Deficiency (VAD) is the most important preventable cause of morbidity, mortality, and childhood blindness, and has been declared a public health problem (Boucheron-Houston et al., 2013; WHO, 2009). Squashes can be included in the group of fruits and vegetables with health-promoting properties. Furthermore, the sweetness of their flesh can make them acceptable by children, who are the most exposed to VAD (Mawamba, Gouado, Leng, Touri dston, & Mbiapo, 2009). The aim of this investigation was to study the effect of the extrusion temperature (ET), the MC, and the winter squash flour content (WSF) on physicochemical and functional properties of 3G snack foods expanded by microwave.

Materials and methods

**Raw materials**

Blends of flours for extrusion were formulated using commercial corn starch (IMSA, S.A. de C.V., Guadalajara, Mexico), whole-grain yellow corn (Zea mays L.) flour, and winter squash (C. moschata D.) cv Cehuala flour. The yellow corn and the winter squash were obtained from the local market, in the Sinaloa State, Mexico. The winter squash flour was prepared from the pulp (peel and seeds were discarded). The pulp was cut into 2-mm slices, blanched at 95 ± 2°C for 2 min, followed by a drying process, using a forced hot-air tray dryer (air speed 1.45 m/s, at 72°C for 110 min), with parallel airflow. Then, the dehydrated slices were milled (Hammer mill, Pulvex model 200, Mexico, D.F., Mexico) up to a particle size ≤250 µm to obtain the winter squash flour. The yellow corn grains were milled to obtain the whole-grain yellow corn flour with a particle size ≤420 µm. The whole-grain yellow corn flours and corn starch were mixed (1:1 ratio), and the WSF was added to the previous mix at different ratios according to the experimental design.

**Extrusion process**

The samples to be extruded were mixed using a laboratory mixer (Kitchen Aid, Model K5SS, St. Joseph, Michigan, USA), adjusted to different MCs (Table 1), placed into plastic bags, and maintained overnight (12–14 h) before processing. In each treatment, approximately 1.5 kg of sample was used. The pre-conditioned samples were fed (forced feeder) at 40–45 g/min, into a single-screw laboratory extruder of three heating zones (Brabender 20DN, model 8-235-00, O HG Brabender, Duisburg, Germany). The temperatures of feeding and die zones were maintained constant at 75°C, whereas the mixing/cooking zone temperature varied from 93.45°C to 140.55°C. An extrusion screw (compression ratio: 2:1) at a speed of 75 rpm and a rectangular die (aperture: 20 mm wide, 1.0 mm high, 100 mm long) were used. The extruded products (pellets) were cut into pieces of 30 mm long, dried at room temperature (23–26°C) until a MC of 9–13%, wrapped in sealed plastic bags, and stored under refrigeration (3–5°C).

**Expansion of pellets**

The expansion of the pellets was performed using a heating time of 23 s in a conventional microwave oven (MS-0746T, LG, Monterrey, Mexico) at 950 W and 2450 Hz.

**Bulk density**

It was performed on the products expanded in microwave according to Penfield and Campbell (1990), with some modifications, using the volume displacement method with millet seed. The BD was determined by dividing the weight of the product for its volume (30 measurements). The results were reported in kg/m³.

**Water absorption index (WAI) and water solubility index (WSI)**

These were performed according to Anderson, Conway, Pfeifer, and Griffin (1969), with some modifications. A sample of 0.25 g of flour was suspended in 12 mL of water at 25°C in a centrifuge tube of 15 mL. The tube was stirred at moderate speed (Vari-Mix Aliquot Mixer, Modelo M48725, Dubuque, Iowa, EUA) for 30 min, and the suspension was centrifuged (Eppendorf 5804R, Hamburg, Germany) at 4500 × g for 20 min, at a temperature of 25°C. The supernatant was carefully decanted and used for determination of the WSI; the sediment was weighed, and the
A calibration curve of HEAT (hexane, ethanol, acetone, and toluene, 10:6:7:7 v/v/v/v). TCs were extracted from pulverized extruded products (particle size ≤ 250 μm) using solution HEAT (hexane, ethanol, acetone, and toluene, 10:6:7:7 v/v/v/v). A calibration curve of β-carotene was used. Four repetitions per treatment were done and the results were expressed as μg of TC/g of sample (db).

**Color parameters**

A tristimulus colorimeter (Minolta, CR-210, Tokyo, Japan) was used to measure the color parameters L* and b*. Samples of expanded products for each treatment were milled to a particle size less than 250 μm. The milled samples were placed in 5 cm Petri dishes, and 3 equidistant readings were done.

**Total carotenoids**

The analysis was performed according to the method reported by Association of Official Analytical Chemists (AOAC, 1999), using a spectrophotometer Model 10, UV GENESYS, Series 2H7G229001, Madison, USA. TCs were extracted from pulverized extruded products (particle size ≤250 μm) using solution HEAT (hexane, ethanol, acetone, and toluene, 10:6:7:7 v/v/v/v). A calibration curve of β-carotene was used. Four repetitions per treatment were done and the results were expressed as μg of TC/g of sample (db).

**Dietary fiber**

This determination was performed by AOAC (1999) method 985.29. The method was based on the enzymatic removal of protein and starch of samples, and the separation of the soluble and insoluble fractions by filtration (Martín-Cabrejas, Esteban, Lopez-Andreu, Waldron, & Selvendran, 1995).

**Experimental design and data analysis**

A central composite rotatable experimental design with a value α = 1.682 was used (Table 1). Factors were the ET (°C), the MC (%), and the WSF (%), with five levels for each factor (Box & Behnken, 1960). The quadratic model applied was:

\[
y_i = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3
\]

where, \(y_i\) is a generic response, \(X_1\) is the ET, \(X_2\) is the MC, \(X_3\) is the WSF, and \(b_0, b_1, ..., b_{33}\) are the regression coefficients. Data were analyzed using the Design Expert statistical software Version 7.1.6 (StatEase, 2008; Minneapolis, USA), whereas Pearson correlations were performed with the Statistica 7.0 software (Statsoft, 2004; Tulsa, USA).

**Results**

The results of the statistical analysis of the data obtained in laboratory for BD, WAI, WSI, color (L* and b* values), and TCs are shown in Table 2. It can be seen that \(R^2_{adj}\) values were greater than 0.75 for the response variables, except for TC. Mathematical models were significant (\(p < 0.025\)) and showed no lack of fit. Although some of the terms of the model, particularly in the interactions, had no significant effect on the responses.

**Bulk density**

This response was analyzed using a quadratic model showing a \(R^2_{adj} = 0.85\) without lack of fit (\(p = 0.289\)). It was found that the extrusion temperature (ET, \(b_1, p < 0.001\) and \(ET^2, b_{11}, p = 0.002\)) and the moisture content (MC, \(b_2, p = 0.045\) and \(MC^2, b_{22}, p = 0.009\)) showed significant effect in the pure-linear and quadratic terms, and also in the interaction term (ET * MC, \(b_{12}, p = 0.006\)). Figure 1 shows the effect of ET and MC on BD at a constant WSF of 8%. It can be seen that, at low MC, BD decreased as ET was

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<th>Winter squash flour content (%)</th>
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In the present study BD showed a moderate negative correlation with the ET factor ($r = -0.60, p = 0.005$). This behavior could be due to an augmented starch gelatinization caused by high ET which enables an increased water entrapment inside the pellets and as a consequence an increased expansion and a reduced BD of the pellets expanded in microwave. In 3G snack foods the MC of the pellets have an important influence on the matrix properties during the expansion process and acts as the driving force for air-bubbles formation. As ET was increased, the pellet viscoelastic properties could have been improved, generating a sufficient vapor pressure to expand the pellets properly. This could have caused the formation of large amount of small air bubbles by which the pressure was increased and the cell walls were broken, allowing water vapor to escape, pellets to expand, and BD to decrease. A similar expansion mechanism was reported by Kocer, Hicsasmaz, Bayindirli, and Katnas (2007) during the expansion of the cake batter. Aguilar-Palazuelos et al. (2006), working with 3G snacks made from a mixture of potato starch, quality protein maize (QPM), and soybean meal, and Singh, Sekhon, and Singh (2007), working with products obtained from a mixture of pea and rice grits by means of direct-expansion processing, mentioned that the BD is inversely related to the degree of expansion. The results found in this study agree with those reported by Dehghan-Shoar, Hardacre, and Brennan (2010) for the directly expanded extruded snacks.
made from rice flour, wheat bran, and corn grits, and enriched with tomato lycopene. It was found that when the ET is increased above 140°C, BD decreased. Altan, McCarthy, and Maskan (2008) found in extruded mixtures of barley and grape that when ET was increased, BD decreased, whereas the volumetric expansion increased. These authors believed that as ET was increased, a higher amount of moisture was lost from the product at the outlet of the extruder die, becoming a less dense product. In the present work, the lowest values of BD (152 kg/m³) occurred in those treatments combining high ET (>117°C) and low MC (<24%). This BD value is close to that found in commercial products (130 ± 4.2 kg/m³) marketed in Culiacán, Mexico. These commercial 3G snacks are regarded as nutritionally poor products because they are elaborated using a high starch content (about 98% db). For this reason, the 3G snacks obtained in the present work advantageously possess an improved nutrimental status due to the carotenoids and dietary fiber content found in the raw materials used for their elaboration, such as winter squash flour and whole-grain yellow corn flour.

On the other hand, it can be seen that at high ET (>117°C), when MC was increased, BD showed a tendency to increase. This could be explained as follows: when MC was increased, the viscoelastic properties were affected and air bubbles could have collapsed inside the pellets causing a decrement in expansion and a BD increment. Similar results were reported by Escalante-Aburto et al. (2013) in direct expanded snack foods obtained from nixtamalized blue corn flour. Also, this is consistent with the results reported by Ding, Ainsworth, Plunkett, Tucker, and Marson (2006) in directly expanded snacks made from wheat. In addition, Stojceska, Ainsworth, Plunkett, and İbanoğlu (2009), in RTE snack foods made from by-products, found that as MC was increased, BD also increased, due to the plasticizing effect of MC, which caused a reduced elasticity, starch gelatinization, expansion, and an increase in BD.

Furthermore, the WSF factor had no significant effect in the simple (WSF, b₁, p = 0.09) and quadratic (WSF², b₃, p = 0.46) terms. This can be seen in Figure 2, where ET and MC showed a major effect on BD, diminishing BD when ET was augmented and MC was diminished, whereas varying WSF, BD was not affected.

Water absorption index

This response was analyzed using a quadratic model showing a $R^2_{adj} = 0.80$ and a CV = 6.62%, with no lack of fit ($p = 0.073$). Regression coefficients of the model and the significance levels are shown in Table 2. It can be seen that the ET and the MC had significant effect in their pure linear ($b₁$ and $b₂$) and interaction terms ($b₁₂$), in addition to the quadratic term of MC ($b₂²$). On the other hand, WSF showed only significant effect on its quadratic term ($b₃²$). Figure 3 shows the effect of ET and MC on WAI values, at a constant level of WSF of 8%. The experimental values ranged between 3.0 and 7.0 g of absorbed water/gram of dry solids, and it can be seen that by increasing both ET and MC, WAI tends to increase. WAI was positively

<table>
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<th>ET (°C)</th>
<th>BD (kg/m³)</th>
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<td>93.45</td>
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<td>21.27</td>
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Figure 2. Photographs showing the visual effect of extrusion temperature in °C, at MC = 28%, and WSF = 8% (A); moisture content in %, at ET = 117°C and WSF = 8% (B); and winter squash flour content in %, at ET = 117°C and MC = 28% (C), on bulk density of expanded 3G snacks.

Figura 2. Fotografías mostrando el efecto visual de la temperatura de extrusión en °C, CH = 28%, y HCAL = 8% (A); contenido de humedad en %, TE = 117°C, y HCAL = 8% (B), y contenido de harina de calabaza en %, TE = 117°C, y CH = 28% (C), sobre la densidad aparente de botanas 3G expandidas por microondas.
correlated with the ET study factor with a value of $r = 0.50$ ($p = 0.023$) and with MC ($r = 0.55$, $p = 0.012$). This behavior may be due to a greater exposure of hydrophilic groups of the extruded products, mainly starch and soluble dietary fiber, permitting a better water penetration inward the pellet structure. During the extrusion process, starch gelatinization occurs and starch molecules are converted into a digestible material due to the interaction of water and heat with the material. The absorbed water is bound to the starch molecules, resulting in a change of the starch granule structure (Ding, Ainsworth, Tucker, & Marson, 2005). According to Yağcı and Göğüş (2008) this process of starch dispersion in excess of water is favored at high ET. The same authors reported that at high MC the starch viscosity could have decreased, allowing for internal mixing and uniform heating which would account for the enhanced water absorption associated to starch gelatinization. Also, Stojceska et al. (2009) in RTE snacks, made of by-products of food, reported that increasing MC from 14% to 17%, the WAI values increased for all formulations studied. Lee, Ryu, and Lim (1999) in directly expanded extruded products made of corn starch reported that WAI was affected by ET, MC, and screw speed in all their terms (linear, quadratic, and interactions). These authors found that WAI increased rapidly with increasing ET to 90°C, but decreased at higher ET. Furthermore, these authors note that this parameter increased with increasing MC.

**Water solubility index**

This response was analyzed using a cubic model, presenting a value of $R^2_{adj} = 0.92$ and a CV = 4.39%, with no lack of fit ($p = 0.78$) (Table 2). Statistical analysis showed that the ET had a highly significant effect on this response in its linear term (ET, $b_1$, $p < 0.001$), but not in the quadratic term (ET$^2$, $b_{11}$, $p = 0.56$). The MC of the mixtures had a significant effect in the linear (MC, $b_2$, $p = 0.001$) and quadratic (MC$^2$, $b_{22}$, $p = 0.026$) terms. Figure 4 shows the effect of ET and MC on WSI at a constant WSF of 8%. It can be seen that when ET was increased, the WSI values increased in all the range of MC. The highest WSI values (>18%) occurred at high ET in combination with low MC. WSI presented a moderate positive Pearson correlation with the ET study factor, with a value of $r = 0.49$ ($p = 0.025$). This behavior could be explained as follows: when ET was increased, an increased degradation of molecules of starch and pectin of winter squash flour could have occurred, releasing in this way low molecular weight compounds and thus increasing their solubility in water. Also, at low MC, more friction inside the extruder could have been occurred, causing high mechanical damage and an increased WSI. WSI which is often used as an indicator of molecular degradation measures the degree of starch fragmentation during extrusion (Kirby, Ollett, Parker, & Smith, 1988). These results agree with those reported by different authors (Anderson et al., 1969; Dogan & Karwe, 2003; Gomez & Aguilera, 1983). They agree to mention that high ET and low MC increase the water solubility of the materials, and decrease the viscosity with respect to the raw materials without process, or with respect to the extruded products at low ET and high MC. Singh-Gujral, Singh, and Singh (2001) studied the effect of the extrusion process of sweet corn grits, and found that the WSI increased when ET was increased and decreased when MC was increased, being higher the effect of MC. This behavior was attributed mainly to the increase in MC, which could have caused a decrease in the friction inside the extruder and thus a decrement in the specific mechanical energy (SME), and consequently a reduction in the degradation of the starch. The same behavior was reported by Yağcı and Göğüş (2008) in directly expanded snack foods by extrusion technology derived from by-products of food and by Agustíno-Osorno et al. (2005) in the production of resistant starch from mango by extrusion. These authors mentioned that the extrusion produces a complete gelatinization of the starch at low water content, when the temperature is between 110°C and 135°C. A combination of heat treatment and mechanical shear would explain the disappearance of the granular structure and crystallinity of extruded starch.

Figure 3. Effect of extrusion temperature (°C) and moisture content (%) on water absorption index (grams of absorbed water/gram of dry solids) of 3G snacks expanded by microwave, at WSF = 8%.

Figure 4. Effect of extrusion temperature (°C) and moisture content (%) on water solubility index (%) of 3G snacks expanded by microwave, at WSF = 8%.
Moreover, WSF factor had significant effect in the quadratic term \((p < 0.001)\) for WSI, but not in its linear term \((b3)\). Also, the interaction MC\(^2\)\(\times\)WSF \((b22\times3, p < 0.001)\) was the only one that presented significant effect. The effect of WSF and MC on WSI at ET = 117°C was analyzed (Figure not shown). It was found that the highest values of WSI were exhibited at high WSF (>11%) in both low and high MC. Before processing, the raw materials showed WSI values of 1.8%, 8.4%, and 36.03% for corn starch, whole-grain yellow corn flour, and winter squash flour, respectively. The WSI value found in winter squash is close to that reported by Que, Mao, Fang, and Wu (2008) in pumpkin \((C. moschata\) D.) flours obtained by hot-air drying which showed a water solubility of 34.90 ± 0.80%. Therefore, in the present work, when the WSF was increased in the extruded blend, the WSI values were augmented too. Also, due to the effect of the extrusion process, when high WSF was used, some low molecular weight compounds could have been formed by degrading primarily the dietary fiber contained in the winter squash flours, leading to an increase in the values of WSI.

**Color parameters**

Table 2 shows that both responses \(L^*\) and \(b^*\) were analyzed using a cubic model with values of \(R^2_{adj}\) equal to 0.76 and 0.89, respectively. Also, the model presented low coefficients of variation (≤2.84%) and showed no lack of fit. Statistical analysis showed in both color parameters that, WSF was the main factor exhibiting a significant effect \((p < 0.05)\) on these responses in the linear \((b_3)\) and quadratic \((b_{33})\) terms, as well as in its interaction with MC \((MC^2\times\text{WSF}, b22\times3)\). Additionally, the ET and MC factors showed significant effect on the color parameter \(b^*\) in their pure linear terms \((b_1, b_2)\). Figure 5 shows the effects of WSF and MC at ET = 117°C on color parameters \(L^*\) and \(b^*\) for expanded pellets. It can be seen that, in all the studied MC interval, when the WSF level was increased, the values of \(L^*\) tend to decrease (Figure 5A). The lowest experimental value of the \(L^*\) parameter \((76)\) was exhibited at levels of WSF higher than 12% and lower than 24% of MC. This behavior may occur because dietary fiber content is increased by increasing WSF in blends, pectins and gums being important components of winter squash flour (Pitchkina, Novokreschnerova, Piskunova, & Morris, 1998). Holguín-Acuña et al. (2008) reported that \(L^*\) color values decreased by the addition of fiber in extruded products made from corn and oat. On the other hand, increasing the WSF could have increased the darkening of the product, given the coloration yellow–orange present in winter squash flour as can be seen in its increased \(b^*\) values. Furthermore, the lowest values of \(L^*\) at low MC levels could be due to a greater friction of the material within the extruder, causing depolymerization of the gum and starch molecules and formation of sugars which could have suffered caramelization and/or Maillard reactions and thus darkening the product. Similarly, Figure 5B shows the effect of WSF and MC at ET = 117°C, on the parameter \(b^*\). It can be seen that by increasing WSF, the \(b^*\) values increased. The \(b^*\) parameter showed a high positive correlation with WSF \((r = 0.76, p = 0.001)\). The highest values of the \(b^*\) parameter were exhibited at high WSF (>12%) and low MC (<24%). Before processing, the raw materials showed \(b^*\) values of 1.5, 31, and 43 for corn starch, whole-grain yellow corn flour, and winter squash flour, respectively. Therefore, when WSF was increased in the extruded blend, the \(b^*\) value augmented too. On the other hand, the \(b^*\) parameter showed a significant negative Pearson correlation with the \(L^*\) parameter, with a value of \(r = -0.78\) \((p = 0.001)\). Yang, Hattori, Kawaguchi, and Takahashi (1998) working on samples of corn starch and potato, conjugated with amino acids, reported that the yellow color largely contributes to the increase of the parameter \(b^*\), and concluded that these values may be increased by Maillard browning reactions. The above mentioned may have resulted in lower values of \(L^*\) in the present study. Other authors have also reported significant negative correlations between the parameters \(b^*\) and \(L^*\) in extruded products for mixtures of oats with grape (Altan et al., 2008b) and oat with tomato (Altan, McCarthy, & Maskan, 2008a).

**Total carotenoids**

This response was analyzed using a cubic model, with a \(R^2_{adj}\) of 0.62, and CV of 8.85%, and showed no lack of fit \((p = 0.368)\) (Table 2). From the statistical analysis, it can be noted that the main factor showing a significant effect on TC was WSF in its pure linear term, with a level of significance of \(p = 0.003\) and a relatively high regression coefficient \((b_3 = 37.418)\). This demonstrates the contribution of WSF as a source of carotenoids in the

![Figure 5](image-url)
expanded product. The statistical analysis also showed that the other terms of the model that had significant effects on TC were MC in its quadratic term \(b_{22}\) and the interaction ET*WSF\(b_{13}\). Figure 6 shows the effect of ET and WSF on TC, at an intermediate level of MC of 28%. It can be seen that, at low WSF (<8%), when the ET levels were increased, the TC content was decreased, suggesting thermo-degradation of carotenoids. Several authors (Guzman-Tello & Cheftel, 2007; Marty & Berzet, 1986) have mentioned that ET affects the TC content in extruded products. On the other hand, the highest content of TC (74.78 µg/g db) was found at high WSF (>12%) and ET higher than 130°C. The Pearson correlation analysis showed that the TC content presented a moderate correlation with WSF \((r = 0.55, p = 0.01)\). This behavior could be due to several reasons: first, to the direct increment in TC content by increasing the WSF in the mixtures (winter squash flour as a raw material showed a value of 337.39 µg of TC/g db); and second, by the plasticizing effect (increased fluidity) of the winter squash components, such as naturally occurring sugars and saccharides derived from the degradation of dietary fiber during the extrusion process at high ET. The plasticizing effect increases the fluidity of the mixtures inside the extruder which could have caused a decrease of their residence times and also a decrease of the SME. Kumar, Ganjyal, Jones, and Hanna (2006) reported that when ET was increased, the residence time inside the extruder decreased for starch containing a MC of 20% (db). Finally, a lesser retention time of the mixtures inside the extruder could have resulted in a lower degradation of TC, at high ET. It is possible that the combinations of levels among MC and WSF, could have caused for the blends, a value of glass transition temperature \((T_g)\) lower than the applied levels of ET, being the \(T_g\) lower, at the highest levels of MC and WSF. Villada, Acosta, and Velasco (2008) reported that some non-aqueous plasticizers, such as glucose, may interact with the starch, causing the decrease of water absorption of this compound and reducing the sample viscosity. It has also been reported that various polymers exhibit rheological changes when the \(T_g\) is achieved. This may cause the conversion of a viscous material in a more fluid and elastic one (Mosicicki et al., 2012). Apruzzese, Pato, Balke, and Diosady (2003) studied the effect of ET, MC, and feed flow on the residence time of yellow corn flours, in a twin-screw extruder, finding that as ET was increased, the residence time decreased. In the present work, the Pearson correlation analysis showed that the TC content presented a moderate correlation with the color parameters \(b^* (r = 0.54, p = 0.013)\) and \(L^* (r = -0.54, p = 0.014)\).

**Total, insoluble, and soluble dietary fiber**

Table 3 shows the effect of ET, MC, and WSF on the dietary fiber (total, insoluble, and soluble) content of the 3G snack foods, expanded by heating in microwave. It can be seen that increasing the value of any of the three study factors, values of Total Dietary Fiber (TDF) and Soluble Dietary Fiber (SDF) show a tendency to increase. However, the first two levels of variation of the factors did not show significant difference \((p = 0.05)\). Similarly, it can be seen that the values of Insoluble Dietary Fiber (IDF) showed a tendency to decrease with increased ET and MC, whereas IDF increased when WSF was augmented. It can be assumed that the increase of TDF when ET was increased may be due mainly to the increase of SDF, which was higher than the decrease of the IDF. A similar behavior has been reported by different researchers, for example, Vasanthan, Gaosong, Yeung, and Li (2002) studied the effect of extrusion process on dietary fiber content of two varieties of oats, finding that the highest values of TDF and SDF were found at high ET (140°C) and high MC (50%). These authors mention that the increase in TDF in this conditions depended largely on the conversion of IDF to SDF, during the extrusion process; although, according to these authors, the increase of SDF was considerably higher than the decrease in the IDF, suggesting a SDF formation from components not present in dietary fiber, such as starch of the raw materials. Theander and Westerlund (1987) conducted a study on the effect of heat processing of starch and wheat flour, and reported that in wheat flour samples, the dietary fiber values increased when the severity of the thermal process was increased, using over-heating and extrusion cooking. According to these authors, this increase may be due to three types of reactions: (1) formation by external transglycosidation of glycans resistant to enzymatic action; (2) polymers formed by the Maillard reactions quantified as lignin; and (3) formation of resistant starchy by retrogradation. Consequently, newly formed indigestible glycans may have contributed to the increased SDF. Furthermore, Méndez-Garcia, Martínez-Flores, and Morales-Sánchez (2011) studied the effect of the extrusion process on the dietary fiber content in lemon waste (peel and endocarp or carpel) extrudates. These authors reported that when ET was increased, the TDF and IDF content increased, while the IDF content decreased. Similar results are reported in the present work.

In the same order of ideas, the effect of MC on dietary fiber content is shown in Table 3, and it can be assumed that when MC was increased, a high degradation of the compounds was presented, resulting in greater solubilization of the samples and increased values of TDF and SDF, as well as decreased values of IDF. In this way, Larrea, Chang, and Martínez Bustos (2005) reported that when MC was increased in the range of 22% to 38%, the contents of TDF, IDF, and SDF increased, when orange bagasse was processed by extrusion. However, the content of TDF and IDF was smaller in the extruded product than in the non-extruded, while SDF was bigger. According to these authors, this behavior was attributed to a conversion of IDF to SDF during the extrusion process, resulting in a decrease of the IDF, due to the breaking of covalent and non-covalent linkages.

**Figure 6.** Effect of extrusion temperature (°C) and winter squash flour content (%) on total carotenoids content (µg/g db) of expanded 3G snacks, at MC = 28%.

**Figura 6.** Efecto de la temperatura de extrusión (°C) y contenido de harina de calabaza (%) sobre el contenido de carotenoides totales (µg/g b. s.) de botanas 3G expandidas por microondas (CH = 28%).
between carbohydrates and proteins associated with the fiber, resulting in small molecular fragments able to be solubilized.

Furthermore, in the present work, regarding to the effect of WSF, the increase of dietary fiber (total, insoluble, and soluble) is mainly due to the fact that winter squash flour provides large amounts of this component, in comparison with the other raw materials used, as shown in Table 4.

On the other hand, as can be seen in Table 4, between the non-extruded blend and the extruded one at optimized conditions (ET = 113°C, MC = 24%, WSF = 12.5%, optimization procedure not shown), TDF and SDF contents significantly decreased in 0.37% and 1.05%, respectively, whereas IDF content decreased not shown), TDF and SDF contents significantly decreased in 0.68%.

Based on Quirós-Sauceda, Palafos, Robles-Sánchez, and González-Aguilar (2012), a complex group of polysaccharides which form dietary fiber can act trapping phenolic compounds and forming chemical interactions with them. Such interactions may occur by hydrogen bonds (between the hydroxyl group of the phenolic compounds and the oxygen atoms of the glycosidic linkages of the polysaccharides), by hydrophobic interactions, and by covalent bonds (ester linkages between phenolic acids and polysaccharides). Therefore, it is possible to hypothesize that both increments in TDF and SDF could be due to the releasing of gums and mucilages (linked to polyphenols) from the insoluble cellulosic matrix as reported by Goñi, Díaz-Rubio, Pérez-Jiménez, and Saura-Calixto (2009). Such release of fragments due to the extrusion process (temperature, MC, screw speed, cutting, etc.) could have increased the quantification of SDF compounds. On the other hand, as previously mentioned, the SDF increment could also be due to the conversion of IDF to SDF, and due to the observed decrease of IDF in this work.

Table 4. Dietary fiber content of raw materials and optimized 3G snacks.

Table 4. Comportamiento de la fibra dietaria de las materias primas y de las botanas 3G optimizadas.

<table>
<thead>
<tr>
<th>Material</th>
<th>TDF (%)</th>
<th>IDF (%)</th>
<th>SDF (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Winter squash flour</td>
<td>30.23 ± 0.28</td>
<td>21.03 ± 0.07</td>
<td>9.20 ± 0.21</td>
</tr>
<tr>
<td>Whole-grain yellow corn flour</td>
<td>14.10 ± 0.15</td>
<td>13.17 ± 0.26</td>
<td>0.93 ± 0.11</td>
</tr>
<tr>
<td>Corn starch</td>
<td>0.46 ± 0.09</td>
<td>0.45 ± 0.04</td>
<td>0.01 ± 0.004</td>
</tr>
<tr>
<td>Optimized mix and snack</td>
<td>10.60 ± 0.18²</td>
<td>9.27 ± 0.22²</td>
<td>1.32 ± 0.41³</td>
</tr>
<tr>
<td>Optimized products²</td>
<td>10.97 ± 0.05³</td>
<td>8.59 ± 0.02³</td>
<td>2.37 ± 0.03³</td>
</tr>
</tbody>
</table>

Note: TDF = Total dietary fiber; IDF = Insoluble dietary fiber; SDF = Soluble dietary fiber.

Values are means of three determinations ± standard deviation. Means of the Optimized mix and snack, with different lower-case letters within columns showed significant difference (p < 0.05).

Conclusions

The mathematical models derived from the data analysis showed variables of response with values of $R^2_{adj}$ > 0.75 (except for TC), $p$ of $F_{(model)} < 0.025$, and none of the variables of response showed lack of fit. The factors ET and MC exhibited the higher effect on the responses of BD, WAI, and WSI. Furthermore, the WSF factor showed the highest impact on the content of TC, dietary fiber, and the color parameters L* and b*. The relatively low values of BD observed in the present work indicate that our snacks possess expansion characteristics similar to commercial 3G snack foods. From our results, it is possible to develop 3G snack foods with similar or better physicochemical characteristics than commercial products, by adding ingredients such as winter squash and whole-grain yellow corn flours. These snack foods may have excellent nutraceutical characteristics, due to the high amounts of carotenoids and dietary fiber that are provided by the raw materials employed. Further research is needed looking for the assessment of the nutraceutical/functional value of 3G snack foods based on the winter squash flour, by means of animal and cellular models.

Acknowledgments

This research was funded by the Universidad Autónoma de Sinaloa through ‘Programa de Fomento y Apoyo a Proyectos
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de Investigación’ (PROFAPI-2009) and ‘Programa de Mejoramiento del Profesorado’ (PROMEP-2008). The authors thank Consejo Nacional de Ciencia y Tecnología (CONACYT) and University of São Paulo, Brazil, for supporting the doctoral research stay of C.I. Delgado-Nieblas.
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