Effect of extrusion on physicochemical, nutritional and antioxidant properties of breakfast cereals produced from bran and dehydrated naranjita pomace

Carlos Delgado-Nieblas, Karen Ruiz-Beltrán, Jessica Sánchez-Lizárraga, José de Jesús Zazueta-Morales, Ernesto Aguilar-Palazuelos, Armando Carrillo-López, Irma Leticia Camacho-Hernández and Armando Quintero-Ramos

ABSTRACT
Extrusion technology is widely used for the production of breakfast cereals (BC). In order to improve their nutritional/nutraceutical quality, the addition of citrus fruit pomace rich in bioactive compounds has been suggested. The objective of this work was to study the effect of extrusion temperature (ET, 88.79–131.21°C) and dehydrated naranjita pomace content (DNP, 0.64–13.36%) on physicochemical, nutritional and antioxidant properties of BC. For statistical analysis, the response surface methodology was used. When ET was elevated, the bulk density, breaking stress, color b* and soluble dietary fiber increased, whereas when DNP was higher, the water solubility index, color b*, soluble dietary fiber, total phenolic compounds, and antioxidant activity increased. The extrusion process decreased the β-glucans content compared to a non-extruded control. These findings indicated that it is possible to produce BC with acceptable physicochemical, nutritional and antioxidant characteristics by adding DNP, whose consumption presents potential benefits in human health.

Introduction
The consumption of extruded breakfast cereals has been increased in recent years due to the convenience of consumption and easy preparation, being this type of products part of Ready-to-Eat (RTE) foods (Brennan, Derbyshire, Tiwari, & Brennan, 2013). The extrusion process is a technique that involves the interaction between the characteristics of the samples to be processed, such as moisture content and chemical composition, and some processing parameters including extrusion temperature, and screw speed. These parameters can have a significant impact on the characteristics presented by the extrudates (Gulati, Weier, Santra, Subbiah, & Rose, 2016), due to the thermo-mechanical energy (shear) provided by the food extruders (Anton & Luciano, 2007). In extruded breakfast cereals the bulk density, texture and water solubility index are quality physical properties. Also, it is important that these products have important content of dietary fiber, phenolic compounds, and antioxidant activity, which are considered functional/nutraceutical characteristics (Charunuch, Limsangouan, Prasert, & Wongkrajang, 2014).

For the preparation of these foods, the use of whole cereal grains has been recommended, since they conserve the bran fraction, which refers to the external parts of the grain (pericarp, testa and aleurone layer). The bran presents a high content of dietary fiber, vitamins, minerals, as well as phenolic compounds. The consumption of these compounds has been linked to important benefits in human health since they could help reduce the risk of suffering different diseases (Andersson, Andersson, Jonsälv, Andersson, & Fredriksson, 2017). Among...
the types of bran that can be used for the production of breakfast cereals are the oat-bran and wheat-bran. The oat (Avena sativa L.) bran, is an important source of soluble dietary fiber, mainly β-glucans and arabinoxylans, as well as cellulose (Drzikova, Dongowski, Gebhardt, & Habel, 2005). Soluble dietary fiber has been reported to reduce elevated blood cholesterol, triglycerides, and glucose levels, being also known that can reduce the incidence of different diseases such as atherosclerosis, diabetes and colon cancer (Holguín-Acuña et al., 2008). Also, the high presence of phenolic compounds and antioxidant capacity in foods may be associated with the prevention of some diseases. Handelman et al. (1999) reported that oat fractions rich in phenolic compounds possess an important level of antioxidant activity, presenting the ability to inhibit the oxidation of low-density lipoproteins (LDL), thus preventing cardiovascular diseases. These authors suggested that this activity could be attributed to caffeic and ferulic acids found in cereals. On the other hand, the wheat bran could improve the nutritional properties in breakfast cereals, since this by-product of wheat milling contains important levels of dietary fiber, mainly the water-insoluble type, in addition to being a rich source of proteins, minerals, vitamins, phenolic compounds and carotenoids (Rashid, Rakha, Anjum, Ahmed, & Sohail, 2015).

Currently, there is a high interest in the production of breakfast cereals through the combination of cereals with fruits and vegetables, since cereals are an important source of carbohydrates and proteins, while fruits and vegetables could provide some phytochemical compounds with important antioxidant potential (Gandhi & Singh, 2015). Among the materials that can be used to produce breakfast cereals are citrus fruits, which are an important source of antioxidant compounds, such as phenolic compounds and flavonoids (Rathod & Annapure, 2013). In Mexico, an important part of the production of naranjita is used by family businesses or small companies to obtain juices and vegetable, since cereals are an important source of carbohydrates, minerals, vitamins, proteins, minerals, vitamins, phenolic compounds and carotenoids (Rashid, Rakha, Anjum, Ahmed, & Sohail, 2015).

The seeds were discarded, and the remaining parts of the fruit (peel, segment walls, and the pressed juice vesicles) constituted the pomace. The dehydrated naranjita pomace (DNP) was obtained through the optimized hot air drying process reported by Delgado-Nieblas et al. (2017). The raw materials were ground individually (hammer-mill, Pulvex, model 200, Mexico City, Mexico), yellow corn grits (Industria de Alimentos S.A. de C.V. Mexico City, Mexico), and powdered malt (Complementos Alimenticios S.A. de C.V. Mexico City, Mexico). The naranjita pomace was obtained as the by-product of the fruit juice extraction process. The seeds were discarded, and the remaining parts of the fruit (peel, segment walls, and the pressed juice vesicles) constituted the pomace. The dehydrated naranjita pomace (DNP) was obtained through the optimized hot air drying process reported by Delgado-Nieblas et al. (2017). The raw materials were ground individually (hammer-mill, Pulvex, model 200, Mexico City, Mexico), with a 1.0 mm round-holes mesh and sieved to obtain products ≤420 µm (passed mesh 40). The ground raw materials were mixed to obtain a “base-mixture”, using a constant ratio of wheat-bran, oat-bran, yellow-corn grits and powdered malt of 54: 35: 8: 3, to which different concentrations of dehydrated naranjita pomace were added, according to the experimental design (Table 1).

### Extrusion process

The breakfast cereals (BC) were elaborated by mixing the raw material flours (approximately 1.0 kg) and adjusting the moisture content to a fixed level of 30.0% ± 1.0, using a laboratory-blender (KitchenAid, Model K5SSWH, Michigan, USA), shaking at minimum speed. Later, the mixtures were stored in polyethylene bags at 5–7°C for approximately 12 h until processing with a laboratory extruder (Brabender 20DN, model 8–235-00, O HG Brabender, Duisburg, Germany), with three heating zones. The feeding and mixing zone temperatures were kept constant at 75°C and 130°C, respectively, whereas the temperature of the outlet die varied from 88.79 to 131.21°C, in accordance with the experimental design (Table 1). In the present study, the extrusion temperature was selected as a study factor because in a preliminary study it had a greater effect on bulk density and texture of breakfast cereals. Furthermore, the feed moisture (FM) level of 30% used in the present study was derived from a preliminary assay at which products with acceptable properties were obtained. A simple extrusion screw (compression ratio 2:1 at 110 rpm), a feed rate of 51 g/min, and a circular die with an aperture of 2 mm were used. The obtained BC were dried (52°C ± 1) during 24 h or up to reaching a moisture content of 4–6%. The BC were cut into pieces of 5 cm long, wrapped in sealed plastic bags, and stored under refrigeration conditions (5–7°C) until analysis.

### Proximate composition

This determination was performed in the raw materials and breakfast cereals obtained at extrusion temperature (ET) = 125°C and dehydrated naranjita pomace (DNP) content = 9.21% (ET and DNP conditions obtained in a previous study not published yet). The AOAC (2005) methodology was used as follows: ash (923.03), fat (920.39), moisture (925.10), protein (960.52), and the carbohydrate content calculated by difference. The determinations were performed in triplicate.

### Bulk density (BD)

This determination was done in breakfast cereals obtained in the different treatments (Table 1) by using 15 pieces of the product (50 mm length). The weight (ws), the diameter (d) (an average value of 3 equidistant measurements), and the length (l) were measured. The BD values

---

**Materials and methods**

**Raw materials**

The raw materials used to elaborate the breakfast cereals were wheat-bran (obtained from a local market in Culiacan, Mexico), oat-bran (acquired from the Mother Nature company, Zapopan, Mexico), yellow corn grits (Industria de Alimentos S.A. de C.V. Mexico City, Mexico), and powdered malt (Complementos Alimenticios S.A. de C.V. Mexico City, Mexico). The naranjita pomace was obtained as the by-product of the fruit juice extraction process. The seeds were discarded, and the remaining parts of the fruit (peel, segment walls, and the pressed juice vesicles) constituted the pomace. The dehydrated naranjita pomace (DNP) was obtained through the optimized hot air drying process reported by Delgado-Nieblas et al. (2017). The raw materials were ground individually (hammer-mill, Pulvex, model 200, Mexico City, Mexico), yellow corn grits (Industria de Alimentos S.A. de C.V. Mexico City, Mexico), and powdered malt (Complementos Alimenticios S.A. de C.V. Mexico City, Mexico). The naranjita pomace was obtained as the by-product of the fruit juice extraction process. The seeds were discarded, and the remaining parts of the fruit (peel, segment walls, and the pressed juice vesicles) constituted the pomace. The dehydrated naranjita pomace (DNP) was obtained through the optimized hot air drying process reported by Delgado-Nieblas et al. (2017). The raw materials were ground individually (hammer-mill, Pulvex, model 200, Mexico City, Mexico), with a 1.0 mm round-holes mesh and sieved to obtain products ≤420 µm (passed mesh 40). The ground raw materials were mixed to obtain a “base-mixture”, using a constant ratio of wheat-bran, oat-bran, yellow-corn grits and powdered malt of 54: 35: 8: 3, to which different concentrations of dehydrated naranjita pomace were added, according to the experimental design (Table 1).
Experimental design for the extrusion study.

| Assay | Code | Actual ET (°C) | Actual DNP (%) |
|-------|------|----------------|----------------|
| 1     | 1    | 95.00          | 2.50           |
| 2     | 1    | 125.00         | 2.50           |
| 3     | 1    | 95.00          | 11.50          |
| 4     | 1    | 125.00         | 11.50          |
| 5     | 1    | 88.79          | 9.00           |
| 6     | 1    | 131.21         | 7.00           |
| 7     | 0    | 110.00         | 0.64           |
| 8     | 0    | 110.00         | 13.36          |
| 9     | 0    | 110.00         | 7.00           |
| 10    | 0    | 110.00         | 7.00           |
| 11    | 0    | 110.00         | 7.00           |
| 12    | 0    | 110.00         | 7.00           |
| 13    | 0    | 110.00         | 7.00           |

ET = extrusion temperature; DNP = dehydrated naranjita pomace.

Color parameters $b^*$ and $L^*$

These parameters were measured using a tristimulus colorimeter (Minolta, CR-210, Tokyo, Japan) in raw materials, in breakfast cereals obtained in the different treatments of the experimental design and in a control treatment without naranjita. The samples of breakfast cereals were ground to a smaller particle size than 250 μm and placed in petri dishes, four equally spaced readings were taken, and the mean value of $L^*$ and $b^*$ was reported. Three repetitions per treatment were done.

Soluble dietary fiber (SDF)

This measurement was made in quadruplicate according to AOAC (2005) method 985.29 in the axial and central treatments (±a) of the experimental design and raw materials, using an enzymatic-kit TDF-100A (Sigma-Aldrich, St. Louis, MO, USA), composed of the enzymes: heat-stable amylase, protease, and amyloglucosidase. The soluble fraction was precipitated with 95% ethanol, separated by filtration (Martin-Cabrejas, Esteban, Lopez-Andreu, Waldron, & Selvendran, 1995) and washed with ethanol and acetone.

β-glucans content

These measurements were carried out in two samples to observe the effect of extrusion process on these fiber compounds. Both samples, an extruded (ES) at ET = 125°C, and a non-extruded (NES) presented the same composition (DNP = 9.21%) (ET and DNP conditions obtained in a previous study not published yet). A Megazyme commercial-kit (Mixed-Linkage) was used, according to the methodology reported by the AOAC (2005) method 995.16. These analyses were performed in quadruplicate.

Extracts for measurement of total phenolic compounds and antioxidant activity

These extracts were obtained according to the methodology reported by Rocha-Guzmán, González-Laredo, Ibarra-Pérez, Nava-Berúmen, and Gallegos-Infante, 2007, with some changes. Acetonic extracts were made with a solution (acetone 70% – water 30% v/v) and concentrated under vacuum conditions with a rotary evaporator Heidolph (model Laborota 4011, Schwabach, Germany), at a temperature of 40°C.

Total phenolic compounds (TPC)

This determination was carried out using the Folin–Ciocalteu spectrophotometric method according to the methodology reported by Heimler, Vignolini, Dini, Vincieri, and Romani (2006). The absorbance measurements were read at a wavelength of 760 nm (Model 10 spectrophotometer, UV GENESYS, Series AQ7 2H7G229001, USA). Three repetitions per treatment were done and the content of TPC of breakfast cereals was expressed in mg of gallic acid/g dry sample.

Antioxidant activity by DPPH method

The scavenging capacity of the 2, 2-diphenyl-1-picrylhydrazyl (DPPH) radical was determined after an incubation period of 60 min. The procedure was performed according to the
Antioxidant activity by ABTS method

This determination for the radical cation ABTS $^+$ (2,2'-azinobis (3-ethylbenzothiazoline-6-sulfonic acid)) was performed according to the spectrophotometric method reported by Re et al. (1999), measuring the absorbance at a wavelength of 734 nm, and comparing with a previously performed Trolox curve. Three repetitions per treatment were done and the antioxidant activity of breakfast cereals was reported in μmol of Trolox equivalents (μmol TE)/g dry sample.

Experimental design and data analysis

For the analysis of data, a central composite rotatable experimental design with a value α = 1.414 was used (Table 1). Factors were the extrusion temperature (ET, °C) and the dehydrated naranjita pomace content (DNP, %), presenting five levels each factor.

The model applied was the following:

$$ Y_i = b_0 + b_1 X_1 + b_2 X_2 + b_{11} X_1^2 + b_{22} X_2^2 + b_{12} X_1 X_2$$  \(2\)

Where $Y_i$ is the variable of response, $X_1$ is the factor ET, $X_2$ is the factor DNP, and $b_0$, $b_1$, $b_2$, $b_{11}$, $b_{22}$, and $b_{12}$ are the regression coefficients. The analysis of data was performed using the Design Expert statistical software Version 7.1.6 (Stat-Ease, Minneapolis, USA), using RSM graphics for Figures 1, 3, 4, 5 and 6. Also, the Pearson correlations were carried out by mean of the software (Statistica 7.0 Statsoft, Tulsa, USA).

Results and discussion

Proximate composition of raw materials and breakfast cereals

The chemical composition (dry base) of the raw materials used for the production of breakfast cereals, such as wheat-bran, oat-bran, yellow corn grits, and dehydrated naranjita pomace, respectively, was as follows: ash, 9.19 ± 0.18%, 2.36 ± 0.03%, 0.52 ± 0.02% and 4.19 ± 0.04%; fat, 2.23 ± 0.02%, 5.42 ± 0.09%, 1.01 ± 0.09% and 0.61 ± 0.05%; protein, 13.42 ± 0.25%, 12.83 ± 0.52 ± 0.02% and 4.19 ± 0.04%; carbohydrates, 75.16%, 79.4%, 91.82% and 88.64%. Likewise, these materials presented moisture content values of 8.28 ± 0.08%, 9.73 ± 0.08%, 12.95 ± 0.04%, and 16.47 ± 0.06%. The moisture, ash, protein and total carbohydrates values in wheat-bran were close to those reported by Onipe, Jideani, and Beswa (2015), while the fat content was similar to that reported by Apprich et al. (2014). Also, the oat-bran presented values of ash and fat close to those reported by Talukder and Sharma (2010), while the protein content was slightly lower. Furthermore, the yellow corn grits presented a composition similar to that reported by Pérez et al. (2008) for protein, fat, and ash. On the other hand, the dehydrated naranjita pomace presented a higher moisture level, lower protein content and similar values of ash and fat to those reported by Delgado-Nieblas et al. (2017). These differences in moisture and protein content could be due to the fact that the season and year of harvest were different. Likewise, the dehydrated naranjita pomace presented a value of soluble dietary fiber of 12.03 ± 0.32%, total phenolic compounds of 16.21 ± 1.16 mg GAE/g db, antioxidant activity (DPPH) of 25.53 ± 0.71 μmol TE/g db, and antioxidant activity (ABTS) of 31.12 ± 1.23 μmol TE/g db. Also, the chemical composition (dry base) of the breakfast cereals obtained at ET = 125°C and DNP = 9.21% was as follows: protein 8.001 ± 1.23%, fat 2.35 ± 0.13%, ash 5.46 ± 0.04%, carbohydrates 84.19%, whereas showed a moisture value of 4.34 ± 0.06%. The breakfast cereals presented similar levels of fat and ash but lower protein than an extruded product made with high content of wheat-bran (Robin et al., 2011).

Regression coefficients and statistical analysis

The statistical information for the analyzed responses in breakfast cereals, such as bulk density (BD), breaking stress (BS), water solubility index (WSI), color parameter b*, total phenolic compounds (TPC) and antioxidant activity (DPPH and ABTS) is presented in Table 2. The variables were fitted to the second-order model (Eq.(2)), obtaining $R^2_{\text{adjusted}}$ values greater than 0.73 for all

Figure 1. Effect of extrusion temperature and dehydrated naranjita pomace content on bulk density (a) and breaking stress (b) of breakfast cereals. 

Figura 1. Efecto de la temperatura de extrusión y del contenido de subproductos de naranjita deshidratados sobre densidad aparente (a) y resistencia a la ruptura (b) de los cereales para desayuno.
the variables of response, the obtained mathematical models were significant ($p < 0.05$), and showed no lack of fit ($p > 0.05$), except antioxidant activity (ABTS). According to the statistical analysis of data (Table 2), the extrusion temperature (ET) factor presented a significant effect ($p < 0.05$) in its linear term on the BD, BS, WSI, color $b^*$ and TPC, whereas, this factor showed a significant effect ($p < 0.05$) in its quadratic term on the BS, WSI, TPC and antioxidant activity (DPPH and ABTS). Also, the dehydrated naranjita pomace (DNP) content, as studied factor, presented highly significant effect ($p < 0.001$) in its linear term on the WSI, color $b^*$, TPC and antioxidant activity (DPPH and ABTS), whereas, this factor showed a significant effect ($p < 0.05$) in its quadratic term on the BD and antioxidant activity (DPPH). In the analysis of interactions, it was found that the interaction ET-DNP showed a significant effect ($p < 0.05$) on the antioxidant activity (DPPH), whereas, the interaction ET-DNP$^2$ presented highly significant effect ($p < 0.001$) on the color $b^*$ and TPC.
The BD has been inversely correlated to the expansion of extruded foods (Singh, Sekhon, & Singh, 2007). On the other hand, in Figure 1(a), it can be observed that at low ET, as the DNP levels were augmented, the BD values increased. This behavior could be due to the high levels of soluble dietary fiber (pectins) in DNP, which interfere within the gelatinization process by competing with starch for water, decreasing the water available for expansion and thus increasing the BD. According to Garau, Simal, Rossello, and Femenia (2007), this type of dietary fiber has a high water retention capacity and is present in high amounts in citrus by-products, such as the peel. Also, Lue, Hsieh, and Huff (1991) reported that the fiber can make the air cells collapse during their formation, reducing the capacity to retain air inside, diminishing thus the expansion and increasing the BD.

Breaking stress (BS)

The texture of extruded breakfast cereals depends on the structural changes of polymers (starch, fiber) during the extrusion process. The texture is an important quality factor for the acceptance of these products by consumers (Robin & Palzer, 2015). The effect of ET and DNP on the BS values of breakfast cereals is shown in Figure 1(b). It is observed that the highest BS values (>9 MPa) were found at high ET (>121°C) and low DNP (<3.5%). BS had a high Pearson correlation with ET factor ($r = 0.74, p = 0.014$) and bulk density ($r = 0.93, p = 0.001$). This could be due to elevated values of bulk density and BS presented in the breakfast cereals at higher ET levels which are related to a lower expansion. In these conditions, the products presented a compact hard structure with a reduced tendency to rupture due to less porosity. The behavior showed in the present study is similar to that reported by Hsieh, Peng, and Huff (1990), who mentioned that in extruded products, the higher the bulk density, the higher the breaking strength. Also, the previous authors reported that the products with the lowest bulk density values (higher expansion) presented larger air cells with thin walls and showed the lowest breaking strength.

Bulk density (BD)

The bulk density (BD) is an important quality characteristic in commercial extruded products since a great quantity of these, are filled based on weight, rather than volume (Brennan, Monro, & Brennan, 2008). In Figure 1(a) is observed that the highest BD values (>900 kg/m$^3$) of breakfast cereals were found at high ET (>121°C) and low DNP (<3.5%), and Figure 2 presents photographs showing the visual effect of the study factors. This response had a high Pearson correlation with the ET factor ($r = 0.79, p = 0.001$). This behavior may be due to that in higher levels of ET, the mass was more plastic and less viscous inside the extruder (Carvalho, Ascheri, & Cal-Vidal, 2002), resulting in shorter residence times. This could have caused a lower starch gelatinization, in consequence, a reduced water-trapping capacity, a low expansion and breakfast cereals with higher BD. In the same way, the lubricating effect of the moisture content fixed at $30 \pm 1.0\%$ combined with high ET levels could have caused a greater fluidity inside the extruder. This could have resulted in diminished mechanical damage, collapsed air bubbles, and a compacted, hard and less expanded product. The BD has been inversely correlated to the expansion of extruded foods (Singh, Sekhon, & Singh, 2007). On the other hand, in Figure 1(a), it can be observed that at low ET, as the DNP levels were augmented, the BD values increased. This behavior could be due to the high levels of soluble dietary fiber (pectins) in DNP, which interfere within the gelatinization process by competing with starch for water, decreasing the water available for expansion and thus increasing the BD. According to Garau, Simal, Rossello, and Femenia (2007), this type of dietary fiber has a high water retention capacity and is present in high amounts in citrus by-products, such as the peel. Also, Lue, Hsieh, and Huff (1991) reported that the fiber can make the air cells collapse during their formation, reducing the capacity to retain air inside, diminishing thus the expansion and increasing the BD.

Water solubility index (WSI)

The WSI is a parameter that indicates the molecular degradation caused in starch granules and fiber during the extrusion process (Rashid et al., 2015). In Figure 3(a) is shown an increase in WSI values of breakfast cereals as DNP levels were increased, in all ET range. The WSI response had a high Pearson correlation with the DNP as the study factor ($r = 0.94; p = 0.001$). This behavior could be due to the high WSI value (49.62 ± 0.01) of the raw material dehydrated naranjita pomace which was higher in relation to the other raw materials used for the production of breakfast cereals. The wheat-bran presented a value of 15.73 ± 0.34%, while the oat-bran showed a value of 10.79 ± 0.29%. Therefore, in the obtained extruded treatments, as the levels of DNP were elevated, the WSI increased too. Similarly, this may be due to the important content of total dietary fiber present in the raw material dehydrated naranjita pomace (43.74% ± 0.9). Therefore, at high levels of DNP, high depolymerization of insoluble dietary fiber could have been presented due to the extrusion process (heat, shear forces), producing low molecular weight compounds with greater water solubility, as reported by Larrea, Chang, and Martinez-Bustos (2005). In the present
work, a comparison of WSI values between the breakfast cereals with the addition of DNP and a commercial breakfast cereal (CBC) was done. It was found that the CBC presented a WSI value of 29.72 ± 1.16%, which was higher than those presented by the breakfast cereals with the addition of DNP, which were in a range from 13.1% to 17.8%. This behavior could be due to the high content of water-soluble sugars added to CBC during their production. The low WSI values of the breakfast cereals added with DNP are a desirable property since these products are consumed mostly mixed with liquid milk, a condition in which the cereal should not become excessively soft. According to Ruiz-Armenta et al. (2014) the extruded breakfast cereals with high WSI values are considered undesirable by consumers since they present high levels of dextrinization, stickiness, and flaccidity when immersed in milk.

**Color parameters b* and L**

Color is an important property for extruded products being consumed mostly mixed with liquid milk, a condition in which the cereal should not become excessively soft. According to Ruiz-Armenta et al. (2014) the extruded breakfast cereals with high WSI values are considered undesirable by consumers since they present high levels of dextrinization, stickiness, and flaccidity when immersed in milk.

### Table 2. Coeficientes de regresión de los modelos, niveles de significancia y análisis de varianza para las respuestas estudiadas en cereales para desayuno extrudidos.

|                    | Bulk density (kg/m³) | Breaking stress (MPa) | WSI (%) | Color parameter b* | TPC (mg GAE/g) | AOA (DPPH) (µmol Te/g) | AOA (ABTS) (µmol Te/g) |
|--------------------|----------------------|-----------------------|---------|-------------------|----------------|------------------------|------------------------|
| intercept          | −867.25              | −61.29                | 35.23   | 1.75              | −1.82          | 52.49                  | 53.45                  |
| linear             | ET                   | 26.13                 | 1.17    | −0.35             | 0.16           | 0.04                   | −0.89                  |
|                    | (0.004)              | (0.003)               | (0.003) | (0.004)           | (0.03)         | (0.242)                | (0.240)                |
|                    | DNP                  | 15.74                 | 0.18    | −0.06             | 6.99           | 0.77                   | −0.46                  |
|                    | (0.18)               | (0.52)                | (< 0.001) | (< 0.001) | (< 0.001) | (< 0.001) | (< 0.001) |
| quadratic          | ET                   | −0.09                 | −0.005  | 0.001             | −              | −                      | 0.0001                |
|                    | (0.08)               | (0.001)               | (0.012) | (0.021)           | (< 0.001)     | (< 0.001)              | 0.0004                |
|                    | DNP                  | 1.19                  | 0.018   | −0.43             | −0.085         | 0.01                   | 0.01                   |
|                    | (0.04)               | (0.15)                | (0.96)  | (0.27)            | (< 0.001)     | (< 0.001)              | (0.209)               |
| interactions       | ET x DNP             | −0.32                 | −0.004  | 0.003             | −0.05          | −0.001                 | 0.003                  |
|                    | (0.14)               | (0.35)                | (0.071) | (0.43)            | (0.73)         | (0.03)                 | (0.524)               |
|                    | ET² x DNP            | −                    | −       | −                 | −              | −0.00004               | −                      |
|                    |                      | −                    | −       | −                 | −              | −                      | −                      |
|                    | ET x DNP²            | −                    | −       | −                 | −              | 0.0007                 | −                      |
|                    | R²                   | 0.81                  | 0.85    | 0.97              | 0.92           | 0.94                   | 0.87                   |
|                    |                      | (0.001)              | (0.001) | (0.001)           | (0.001)       | (0.001)                | (0.001)               |
|                    | CV                   | 3.18                  | 7.09    | 1.65              | 2.68           | 7.20                   | 10.35                  |
|                    |                      | (0.001)              | (0.001) | (0.001)           | (0.001)       | (0.001)                | (0.001)               |
|                    | p of F (model)       | 0.003                 | 0.001   | < 0.001           | < 0.001        | < 0.001                | < 0.001                |
|                    |                      | (0.001)              | (0.001) | (0.001)           | (0.001)       | (0.001)                | (0.001)               |
|                    | Lack of fit          | 0.066                 | 0.160   | 0.903             | 0.956          | 0.178                  | 0.437                  |
|                    |                      | (0.242)              | (0.004) | (0.003)           | (0.15)        | (0.004)                | (0.003)               |

ET = extrusion temperature; DNP = dehydrated naranjita pomace; WSI = water solubility index; TPC = total phenolic compounds; GAE = gallic acid equivalents; AOA = antioxidant activity; TE = Trolox equivalents; CV = coefficient of variation. Numbers within brackets indicate significance levels; empty spaces in table indicate unused model terms.

**Soluble dietary fiber (SDF)**

In Figure 4 is observed the effect of ET on the content of soluble dietary fiber (SDF) of breakfast cereals, showing the higher values of SDF at high levels of ET. This behavior could be due to the fact that elevated levels of ET could have caused the depolymerization of insoluble dietary fiber,
generating compounds of low molecular weight, which have greater solubility. This behavior is similar to that reported by Vasanthan, Gaosong, Yeung, and Li (2002), who studied the effect of the extrusion process on the dietary fiber content in two varieties of oat. These authors related the increase of SDF with the conversion of insoluble dietary fiber to SDF, due to the severity of the process by the high temperatures. On the other hand, in the same Figure 4 is observed the effect of the addition of dehydrated naranjita pomace (DNP) on the SDF values. It can be observed that by increasing the DNP levels from 0.64% to 7%, the values of SDF increased. This behavior may be due to that the dehydrated naranjita pomace is an important source of soluble dietary fiber (12.03 ± 0.32%), presenting a value higher than the values of SDF observed in the other raw materials used for the preparation of the breakfast cereals. Also, the processing by extrusion could have contributed to this increase as previously mentioned. However, by increasing the DNP levels up to 13.36%, the values of SDF decreased. This behavior may be due to the fact that some components present in citrus pomace, such as pectins (Mandalari et al., 2006) could have presented a lubricating effect. Therefore, lower friction within the extruder was presented reducing the severity of the process, diminishing the depolymerization of insoluble dietary fiber, and consequently lowering the SDF formation. Also, it is possible that the combination of the used feed moisture level (30%) and the soluble sugars present at high DNP, could have presented a plasticizing effect, diminishing the glass transition temperature (Tg) values, and reducing the viscosity and residence times inside the extruder. When the Tg is reached, different polymers show rheological changes, becoming less viscous and more fluid materials (Moscicki et al., 2012). Villada, Acosta, and Velasco (2008) mentioned that polymers like starch may interact with non-aqueous plasticizers, among them sugars such as glucose, diminishing its water absorption and generating less viscosity. In the present study, this could have decreased the severity of the extrusion process at high levels of DNP, lowering depolymerization of insoluble dietary fiber, so that less compounds of low molecular weight (soluble) were formed, decreasing the SDF values.

**β-glucans content**

β-glucans content was determined in two samples. Both, an extruded (ES) at ET = 125°C, and a non-extruded (NES) presented the same composition (DNP = 9.21%). This determination was made because of cereal brans, mainly oat-bran, are an important source of this type of soluble dietary fiber. It was found that the NES presented a β-glucan content of 2.46 ± 0.10 g/100g db, while ES showed a content of 1.42 ± 0.05 g/100g db. The extrusion process diminished the content of β-glucans probably due to the severe processing conditions (such as high temperature, pressure and shear force) used to obtain the breakfast cereals. However, the content of β-glucans found in the ES obtained in the present study can be considered as an acceptable level. Holguín-Acuíña et al. (2008) reported that the US Food and Drug Administration (FDA) recognizes the role of fiber consumption in the risk reduction of cardiovascular diseases, mentioning that those products that contain 0.75 g β-glucans per serving are allowed to exhibit a health claim that the product “will reduce the risk of coronary heart disease”. In this study, the obtained breakfast cereals presented a content of 0.57 g of β-glucans per serving (40 g), which is close to the previously mentioned value.

### Total phenolic compounds (TPC)

The effect of extrusion temperature and dehydrated naranjita pomace, as study factors on the TPC values of breakfast cereals, is shown in Figure 5. It is observed that the highest values of TPC (1.3 mg GAE/g db) were found in conditions of high ET and DNP. This response had a direct Pearson correlation with the DNP factor (r = 0.88; p < 0.001). The highest values of TPC observed at high levels of DNP (>10.5%) could have been due to the high content of TPC present in the dehydrated naranjita pomace, which was higher than those found in the other raw materials used to make the breakfast cereals. Citrus peels are by-products of juice extraction process and are treated as waste of little economic value by industry. However, they are important sources of antioxidants such as phenolic compounds (phenolic acids and flavonoids), which are important to human nutrition (Rathod & Annapure, 2017). Also, in the present study was observed that by increasing the ET levels, the TPC values were elevated. This could be due to the fact that the high extrusion temperatures could have caused the release of some phenolic compounds bound to cell walls in the raw materials (wheat bran, oat bran, and corn grits). Phenolic acids, mainly ferulic acid, p-coumaric acid, and vanillic acids, are the main compounds present in the bran layer of grains, being mostly covalently bound with insoluble polymers (Rufián-Henares & Delgado-Andrade, 2009). Zielinski, Kozlowska, and Lewczuk (2001) reported that in some foods exposed to thermal processing, the phenolic acids can be released from the cell walls, causing an increase in the TPC values. Also, in the present work was determined the total phenolic compounds (TPC) content in the breakfast cereals without the addition of DNP, and in the breakfast cereals with 9.21% of DNP, both obtained at an ET of 125°C. The product with the addition of DNP presented a TPC content of 1.07 ± 0.08 mg GAE/g db, whereas the breakfast cereals without DNP had a value of 0.52 ± 0.03 mg GAE/g db. The above results indicate the importance of this pomace as a source of phenolic compounds for the purpose of improving the nutrimental/nutraceutical potential of the obtained breakfast cereals.

### Antioxidant activity by the DPPH method

In Figure 6(a) is presented the effect of the study factors ET and DNP on the antioxidant activity (DPPH) values of breakfast cereals. The highest values (>5 μmol TE/g db) were presented at high levels of ET and DNP. This response had a direct Pearson correlation with the DNP factor (r = 0.90, p = 0.001). This behavior may be due to the high antioxidant activity presented by the dehydrated naranjita pomace (25.53 ± 0.71 μmol TE/g db), which can be attributed to the presence of some compounds with important antioxidant activity, such as flavonoids. According to Anagnostopoulou, Kefalas, Papageorgiou, Assimopoulou, and Boskou (2006) the scavenging capacity of the DPPH radical in citrus by-products such as orange (Citrus sinensis) may be due to its high content of phenolic compounds such as phenolic acids or flavonoids. Similarly, Senevirathne, Jeon, Ha, and Kim (2009) reported that the compounds heptamethoxyflavone, nobiletin, hesperidin, and
naranuritin were the main phenolic compounds present in citrus fruit by-products, and their antioxidant capacity may be due to their high capacity to donate hydrogen atoms. On the other hand, in the present study, the increase in antioxidant capacity when ET levels were increased could be due to a high release of phenolic compounds which has been reported to have important antioxidant capacity. Brennan, Brennan, Derbyshire, and Tiwari (2011) reported that some factors used in the extrusion process, such as temperature, can have a positive influence on the antioxidant capacity of extruded products. These authors mentioned that the positive effect can occur through the release of phenolic acids from the matrix of the cell wall, which may have a greater antioxidant potential. Also, the high antioxidant capacity in conditions of high ET could be due to the formation of compounds generated in the Maillard reaction which can occur at high temperatures (Vega-Galvés et al., 2009). The values of antioxidant activity (DPPH) found in the present work are close to those reported by Leyva-Corral et al. (2016) in an extruded cereal produced with oat flour, potato starch, and apple pomace, who reported values of antioxidant activity (DPPH) from 5.06 to 5.18 μmol TE/g db.

Antioxidant activity by the ABTS method

The effect of ET and DNP on the antioxidant activity (ABTS) of breakfast cereals is shown in Figure 6(b). The highest values (>6 μmol TE/g db) were obtained at high levels of DNP, in all studied range of ET. This response had an important Pearson correlation with the DNP factor (r = 0.81; p = 0.004) and with the responses TPC (r = 0.79; p = 0.007) and antioxidant activity (DPPH) (r = 0.77; p = 0.009). This correlation between TPC and antioxidant activity has been reported by Zielinski and Kozłowska (2000) in cereals grains, such as wheat and oat, which were used in the present study. The previous authors mentioned that these cereals have an important capacity to scavenge the radical ABTS⁺, with potential effects in the inhibition of lipid peroxidation. These authors mentioned that the antioxidant potential and bioavailability of cereal antioxidants may depend on species, as well as grain fractions (bran, flour, or whole grain), and processing conditions. The behavior presented in the present study may be due to the high antioxidant activity presented by the dehydrated naranjita pomace attributed to its high content of bioactive compounds. The highest values of antioxidant activity (ABTS) found in the present work are similar to those reported by Sharma, Dar, Nayik, and Kaur (2016) in a breakfast cereal porridge made with bran obtained from wheat, oat, and rice. These authors applied a conventional method using acetone solvent for extraction, as in the present work, reporting values of antioxidant activity (ABTS) of 6.16 ± 1.42 μmol TE/g db. In addition to the antioxidant, nutritional and physicochemical properties of the breakfast cereals with the addition of dehydrated naranjita pomace, its sensorial acceptability evaluated by consumers is of great importance. Delgado-Nieblas et al. (2017) reported, in a previous study, that the breakfast cereals with the addition of dehydrated naranjita pomace presented acceptable sensory properties (color, flavor, and texture) evaluated by panelists when compared to a control (breakfast cereals without naranjita). In such work is mentioned that this finding is positive due to the important levels of phytochemical compounds provided by the naranjita pomace, which could improve the antioxidant activity, without affecting the sensory acceptability of the breakfast cereals.

Conclusions

The extrusion temperature and the content of dehydrated naranjita pomace as studied factors presented a significant effect on the different evaluated responses. The models obtained for all the variables of response were significant (p < 0.05), with values of R² adjusted ≥ 0.74, without presenting lack of fit (p > 0.05), except AOA (ABTS). Further analysis showed the importance of the dehydrated naranjita pomace as a source of fiber, phenolic compounds and antioxidant activity in the production of breakfast cereals (BC) by an extrusion process. In addition, our findings suggest that it is possible to combine cereal brans and citrus pomace to obtain BC rich in bioactive compounds. Based on the results of this work, it is inferred and recommended to use extrusion temperature levels between 121°C and 131°C, and DNP levels between 7.1% and 9.6%, in order to obtain breakfast cereals with acceptable physicochemical, nutritional and antioxidant properties. The consumption of this type of breakfast cereals presents potential benefits in human health.

Disclosure statement

No potential conflict of interest was reported by the authors.

Acknowledgments

Authors thank Universidad Autónoma de Sinaloa for the financial support through PROFAPI2014/239 for the development of this study. They also thank M.Sc. Xóchitl Ariadna Ruiz Armenta for technical assistance in laboratory analysis.

Funding

This work was supported by the Universidad Autónoma de Sinaloa [PROFAPI2014/239].

ORCID

Carlos Delgado-Nieblas http://orcid.org/0000-0002-6202-090X
José de Jesús Zazueta-Morales http://orcid.org/0000-0001-6978-0752
Ernesto Aguilar-Palazuelos http://orcid.org/0000-0002-3805-8422
Armando Carrillo-López http://orcid.org/0000-0003-3541-8493
Irma Leticia Camacho-Hernández http://orcid.org/0000-0002-8397-2029
Armando Quintero-Ramos http://orcid.org/0000-0003-2689-2601

References

Aguilar-Palazuelos, E., Zazueta-Moraes, J. D. J., Jiménez-Arévalo, O. A., & Martínez-Bustos, F. (2007). Mechanical and structural properties of expanded extrudates produced from blends of native starches and natural fibers of henequen and coconut. Starch-Stärke, 59(11), 533–542.
Anagnostopoulou, M. A., Kefalas, P., Papageorgiou, V. P., Assimopoulou, A. N., & Bokou, D. (2006). Radical scavenging activity of various extracts and fractions of sweet orange peel (Citrus sinensis). Food Chemistry, 94(1), 19–25.
Anderson, R. A., Conway, H. F., Pfeifer, V. F., & Griffin, E. L. (1969). Gelatinization of corn grits by roll-and extrusion-cooking. Cereal Science Today, 14, 4–7, 11, 12.
Andersson, A. A., Andersson, R., Jonsäll, A., Andersson, J., & Fredriksson, H. (2017). Effect of different extrusion parameters on...
dietary fiber in wheat bran and rye bran. Journal of Food Science, 82 (6), 1344–1350.

Anton, A. A., & Luciano, F. B. (2007). Instrumental texture evaluation of extruded snack foods: A review. CYTA-Journal of Food, 5(4), 245–251.

AOAC. (2005). Official Methods of Analysis (18th ed.). Gaithersburg, MD: Association of Official Analytical Chemists.

Apprich, S., Tiranpanal, O., Hell, J., Reisinger, M., Bohmddorfer, S., Siebenhandl-Ehn, S., & Knefel, W. (2014). Wheat bran-based biofinery - 2: Valorization of products. LWT-Food Science and Technology, 56 (2), 222–231.

Brand-Williams, W., Cuvelier, M. E., & Berset, C. L. W. T. (1995). Use of a free radical method to evaluate antioxidant activity. LWT-Food Science and Technology, 28(1), 25–30.

Brennan, C., Brennan, M., Derbyshire, E., & Tiwari, B. K. (2011). Effects of extrusion on the polyphenols, vitamins and antioxidant activity of foods. Trends in Food Science & Technology, 22(10), 570–575.

Brennan, M. A., Derbyshire, E., Tiwari, B. K., & Brennan, C. S. (2013). Ready-to-eat snack products: The role of extrusion technology in developing consumer acceptable and nutritious snacks. International Journal of Food Science & Technology, 48(5), 893–902.

Brennan, M. A., Mono, J. A., & Brennan, C. S. (2008). Effect of inclusion of soluble and insoluble fibres into extruded breakfast cereal products made with reverse screw configuration. International Journal of Food Science & Technology, 43(12), 2278–2288.

Carvalho, R. D., Ascheri, J. L. R., & Cal-Vidal, J. (2002). Efeito dos parâmetros de extração nas propriedades físicas de pellets (3G) de misturas de farinhas de trigo, arroz e banana. Ciência e Agrotecnologia, 26(5), 1006–1018.

Charunuch, C., Limsangouan, N., Prasert, W., & Wongkrajang, K. (2014). Optimization of extrusion conditions for ready-to-eat breakfast cereal enhanced with defatted rice bran. International Food Research Journal, 21(2), 713–722.

Delgado-Nieblas, C. I., Zazueta-Morales, J. J., Ahumada-Aguilar, J. A., Aguilar-Palazuelos, E., Carrillo-López, A., Jacobo-Valenzuela, N., & Telis-Romero, J. (2017). Optimization of an air-drying process to obtain a dehydrated naranjita (Citrus mitis B.) pomace product with high bioactive compounds and antioxidant capacity. Journal of Food Process Engineering, 40(1), 1–13.

Drzíková, B., Dongowski, G., Gebhardt, E., & Habel, A. (2005). The composition of dietary fibre-rich extrudates from oat affects bile acid binding and fermentation in vitro. Food Chemistry, 90(1–2), 181–192.

Gandhi, N., & Singh, B. (2015). Study of extrusion behaviour and porridge making characteristics of wheat and guava blends. Journal of Food Science and Technology, 52(5), 3030–3036.

Garau, M. C., Simal, S., Rossello, C., & Femenia, A. (2007). Effect of air-drying temperature on physico-chemical properties of dietary fibre and antioxidant capacity of orange (Citrus aurantium v. Canotena) by-products. Food Chemistry, 104(3), 1014–1024.

Gulati, P., Weier, S. A., Santra, D., Subbiah, J., & Rose, D. J. (2016). Effects of feed moisture and extruder screw speed and temperature on physical and chemical characteristics and antioxidant activity of extruded proso millet (Panicum millaceum) flour. International Journal of Food Science & Technology, 51(1), 114–122.

Handelman, G. J., Cao, G., Walter, M. F., Nightingale, Z. D., Paul, G. L., Brennan, M. A., Chun, J., & Blumberg, J. B. (1999). Antioxidant capacity of oat (Avena sativa L.) extracts. 1. Inhibition of low-density lipoprotein oxidation and oxygen radical absorbance capacity. Journal of Agricultural and Food Chemistry, 47(12), 4888–4893.

Heilmä, L., Vignolini, B., Dini, M. G., & Romani, A. (2006). Antiradical activity and polyphenol composition of local brassicaeaeedible varieties. Food Chemistry, 99(3), 464–469.

Holguín-Acuña, A. L., Carvajal-Millán, E., Santana-Rodríguez, V., Rascón-Chu, A., Márquez-Escalante, J. A., Ponce de León-Renova, N. E. P., & Gastelum-Franco, G. (2008). Maize bran/oat flour extruded breakfast cereal: A novel source of complex poly saccharides and an antioxidant. Food Chemistry, 111(3), 654–657.

Hsieh, F., Rashid, S., Rakha, A., Anjum, F. M., Ahmed, W., & Sohail, M. (2011). Effect of extrusion on the antioxidant capacity and color attributes of expanded extrudates prepared from purple potato and yellow pea flour mixtures. Journal of Food Science, 76(6), C874–C883.

Onipe, O. O., Jideani, A. I., & Beswa, D. (2015). Composition and functionality of wheat bran and its application in some cereal food products. International Journal of Food Science & Technology, 50(12), 2509–2518.

Pérez, A. A., Drago, S. R., Carrara, C. R., De Greef, D. M., Torres, R. L., & Gonzalez, R. (2008). Extrusion cooking of maize/soybean mixture: Factors affecting expanded product characteristics and flour disper sion viscosity. Journal of Food Engineering, 87(3), 333–340.

Rashid, S., Rakha, A., Anjum, F. M., Ahmed, W., & Sohail, M. (2015). Effects of extrusion cooking on the dietary fibre content and water solubility index of wheat bran extrudates. International Journal of Food Science & Technology, 50(7), 1533–1537.

Rathod, R. P., & Annappare, U. S. (2017). Antioxidant activity and polyphenolic compound stability of lentil-orange peel powder blend in an extrusion process. Journal of Food Science and Technology, 54(4), 954–963.

Re, R., Pellegrini, N., Protaggente, A., Pannala, A., Yang, M., & Rice-Evans, C. (1999). Antioxidant activity applying an improved ABTS radical cation decolorization assay. Free Radical Biology and Medicine, 26 (9–10), 1231–1237.

Robin, F., & Palzer, S. (2015). Modifying food texture: Texture of breakfast cereals and extruded products. Cambridge, UK: Woodhead Publishing.

Robin, F., Théodulcz, C., Gianfrancesco, A., Pineau, N., Schuchmann, H. P., & Paliservice, S. (2011). Effect of extrusion and moisture on the physical and sensorial characteristics of experimental breakfast cereal. Carbohydrate Polymers, 85(1), 65–74.

Rocha-Guzmán, N. E., González-Laredo, R. F., Ibarra-Pérez, F. J., Nava-Berón, F., Ríos-Tejeda, R., & Vargas, R. E. (2015). Effect of extrusion on the antioxidant activity of snacks added with bagasse of naranjita (Citrus bergamia R. R. Aus). CYTA-Journal of Food, 17(2), 171–178.

Senevirathne, M., Mekal, Y. J., Ha, J. H., & Kim, S. H. (2009). Effective drying of citrus by-product by high speed drying: A novel drying technique and their antioxidant activity. Journal of Food Engineering, 92(2), 157–163.

Sharma, S., Dar, B. N., Nayak, G. K., & Kaur, G. (2016). Total phenolic antioxidant and antioxidant activity of cereal bran enriched ready to eat breakfast cereal porridge. Current Nutrition & Food Science, 12(2), 142–149.

Singh, B., Sekhon, K. S., & Singh, N. (2007). Effects of moisture, temperature and level of pea grits on extrusion behaviour and product characteristics of rice. Food Chemistry, 100(1), 198–202.
Talukder, S., & Sharma, D. P. (2010). Development of dietary fiber rich chicken meat patties using wheat and oat bran. *Journal of Food Science and Technology, 47*(2), 224–229.

Vasanthan, T., Gaosong, J., Yeung, J., & Li, J. (2002). Dietary fiber profile of barley flour as affected by extrusion cooking. *Food Chemistry, 77*(1), 35–40.

Vega-Gálvez, A., Di Scala, K., Rodríguez, K., Lemus-Mondaca, R., Miranda, M., López, J., & Perez-Won, M. (2009). Effect of air-drying temperature on physico-chemical properties, antioxidant capacity, colour and total phenolic content of red pepper (*Capsicum annuum*, L. var. Hungarian). *Food Chemistry, 117*(4), 647–653.

Villada, H. S., Acosta, H. A., & Velasco, R. J. (2008). Investigación de amilóides termoplásticos, precursores de productos biodegradables. *Información Tecnológica, 19*(2), 3–14.

Wani, S. A., & Kumar, P. (2016). Effect of extrusion on the nutritional, antioxidant and microstructural characteristics of nutritionally enriched snacks. *Journal of Food Processing and Preservation, 40*(2), 166–173.

Yu, M. W., Lou, S. N., Chiu, E. M., & Ho, C. T. (2013). Antioxidant activity and effective compounds of immature calamondin peel. *Food Chemistry, 136*(3–4), 1130–1135.

Zielinski, H., & Kozłowska, H. (2000). Antioxidant activity and total phenolics in selected cereal grains and their different morphological fractions. *Journal of Agricultural and Food Chemistry, 48*(6), 2008–2016.

Zielinski, H., Kozłowska, H., & Lewczuk, B. (2001). Bioactive compounds in the cereal grains before and after hydrothermal processing. *Innovative Food Science & Emerging Technologies, 2*(3), 159–169.