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**Physical, phytochemical and sensory characteristics of extruded high-fiber breakfast cereals prepared by combining carrot by-products with wheat and oat bran**

**Características físicas, fitoquímicas y sensoriales de cereales para desayuno altos en fibra producidos combinando subproductos de zanahoria con salvado de trigo y avena**

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**Abstract**

High-fiber breakfast cereals are mainly produced by extrusion, and to improve its nutritional/nutraceutical properties, the addition of raw materials rich in bioactive compounds has been suggested. The aim of this study was to evaluate the physical, phytochemical and sensory characteristics of extruded high-fiber breakfast cereals (HFB) prepared from carrot by-products and bran as affected by feed moisture (FM, 19.34-30.66%) and carrot pomace content (CPC, 5.51-22.49%), under a central composite rotatable experimental design. As FM increased, flexural modulus (FMO, 54-89 MPa), soluble dietary fiber (SDF, 0.64-4.18%), bound phenolic compounds (BPC, 0.02-0.25 mg GAE/g), and DPPH antioxidant activity from BPC extracts (2.07-2.29  $\mu$ mol TE/g) increased, whereas expansion index (EI, 1.09-0.89) diminished. Also, As CPC augmented, FMO (54-83 MPa), water solubility index (WSI, 14.21-18.84%), color b\* (Cb\*, 26.8-28.8), insoluble dietary fiber (IDF, 19.43-24.71%), SDF (1.12-3.98%), free phenolic (FP, 0.56-0.71 mg GAE/g), and free flavonoids compounds (FFC, 0.05-0.31 mg QE/g) increased, whereas BPC decreased (0.25-0.14 mg GAE/g). The HFB presented good sensory acceptability ( $\geq 69\%$ ). These extruded products had appropriate physical, phytochemical, and sensory characteristics, the consumption of which has potential nutraceutical benefits. **Keywords:** breakfast cereals, extrusion, carrot pomace, nutraceutical properties, sensory properties.

**Resumen**

Los cereales para desayuno altos en fibra son producidos principalmente por extrusión y, para mejorar sus propiedades nutricionales/nutracéuticas, se ha sugerido la adición de materias primas ricas en compuestos bioactivos. El objetivo del presente estudio fue evaluar las características físicas, fitoquímicas y sensoriales de cereales para desayuno extrudidos altos en fibra (CDAF) producidos a partir de subproductos de zanahoria y salvado, como son afectadas por el contenido de humedad (CH, 19.34-30.66%) y el contenido de subproductos de zanahoria. (CSN, 5.51-22.49%), utilizando un diseño experimental central compuesto rotatable. Cuando aumentó el CH, se incrementaron el módulo de flexión (MFL, 54-89 MPa), fibra dietaria soluble (FDS, 0.64-4.18%), compuestos fenólicos ligados (CFLG, 0.02-0.25 mg EAG/g) y actividad antioxidante (DPPH) del extracto de CFLG (2.07-2.29  $\mu$ mol ET/g), mientras que, el índice de expansión (IE, 1.09-0.89) disminuyó. Asimismo, cuando se incrementó el CSN, aumentó MFL (54-83 MPa), índice de solubilidad en agua (ISA, 14.21-18.84%), color b\* (Cb\*, 26.8-28.8), fibra dietaria insoluble (FDI, 19.43-24.71%), FDS (1.12-3.98%), compuestos fenólicos libres (CFL, 0.56-0.71 mg EAG/g) y flavonoides libres (FL, 0.05-0.31 mg EQ/g), mientras que CFLG (0.25-0.14 mg EAG/g) disminuyeron. Los CDAF presentaron buena aceptabilidad sensorial ( $\geq 69\%$ ). Estos productos extrudidos mostraron adecuadas características físicas, fitoquímicas y sensoriales, cuyo consumo tiene potenciales beneficios nutraceuticos.

**Palabras clave:** cereales para desayuno, extrusión, subproductos de zanahoria, propiedades nutraceuticas, propiedades sensoriales.

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## 1 Introduction

The extrusion is a versatile technology that combines several unit operations such as mixing, cooking, shearing, shaping, and forming. This process is widely used for the production of ready-to-eat breakfast cereals, snacks, among other foods (Oliveira *et al.*, 2017). The processing conditions that are commonly used during extrusion involve high barrel temperatures and high pressures applied for short times, causing changes in the food properties that result in a better nutritional quality based on a higher digestibility and a lower level of anti-nutritional factors (Patil *et al.*, 2016). These changes are dependent of different extrusion variables used for their production, such as feed moisture, screw speed, as well as the properties of the raw materials employed for their elaboration. Additionally, these variables can influence properties such as expansion, texture, water solubility index, color, dietary fiber and sensory acceptability, all of them referred to as quality parameters in this type of foods (Borah *et al.*, 2016). Likewise, it is important the effect of the extrusion process on food phytochemicals, such as phenolic compounds. These compounds occur in foods in both free and bound forms, and their presence has been related to an increased antioxidant activity with potential benefits in human health (Zeng *et al.*, 2016). In recent years, the demand for fiber-rich foods has increased due to the health benefits associated with their consumption. Extrusion technology is highly used for the production of these products, and some types of bran, such those obtained from wheat and oat, can be used as raw materials (Oladiran *et al.*, 2018). The bran that is found mostly in the outer layers of the whole cereal grains, contains high levels of bioactive compounds and dietary fiber, whose consumption has shown important advantages in comparison to refined cereals (Gong *et al.*, 2018). The addition of wheat bran for the production of extruded high-fiber breakfast cereals can increase their dietary fiber content, mostly the insoluble type. Additionally, this wheat-milling by-product provides important contents of phenolic compounds, proteins, vitamins and minerals (Rashid *et al.*, 2015). Also, Călinoiu and Vodnar (2018) reported that oat-bran is an important source of soluble dietary fiber, mainly  $\beta$ -glucan compounds, with beneficial impacts on reducing cholesterol and glycemic index, and presenting potential benefits in reducing chronic

diseases in humans. Also, as an alternative to improve the nutritional quality and as well as generating innovations in shape, texture and color of these extruded products, the addition of by-products from fruits and vegetables in the production of cereal-based foods by extrusion has been recommended (Offiah *et al.*, 2018). These by-products with high content of phytochemical compounds, and dietary fiber, are generally dehydrated and milled prior to be added as ingredients in the production of functional foods (Saleh *et al.*, 2019). Among the vegetables that can be used to improve the nutritional/nutraceutical properties of high-fiber breakfast cereals, the carrot excels. The carrot (*Daucus carota* L.) is one important root vegetable that is an important source of bioactive compounds, such as carotenoids and phenolic compounds. The phenolic compound mostly occurring in carrot is chlorogenic acid, followed by important levels of flavonoids such as kaempferol, quercetin and luteolin (Ergun and Süslüoğlu, 2018), whose presence in foods confers them a greater antioxidant activity. The carrot pomace is a rich-in-insoluble-fiber by-product that is derived from the juice extraction process and have also a high content of soluble fiber when compared to pomace obtained from fruits such as apple, cabbage, and strawberry. This pomace can easily be added as an ingredient in the production of functional foods, without introducing unpleasant flavors (Yadav *et al.*, 2018) and has been used as an additive in the production of foods as sponge cakes, improving its color and sensory properties (Salehi *et al.*, 2016). In the same way, the carrot pomace has been added for the preparation of pasta (Gull *et al.*, 2015; Jalgaonkar *et al.*, 2018), biscuits (Gayas *et al.*, 2012; Baljeet, *et al.*, 2014), extruded rice-based snacks (Upadhyay *et al.*, 2010; Alam *et al.*, 2015), extruded corn-based snacks (Kaisangsri *et al.*, 2016; Samard *et al.*, 2017), cookies (Kumar *et al.*, 2011), batter and cakes (Majzoobi *et al.*, 2017), and nixtamalized corn flour tortillas (Santana-Gálvez *et al.*, 2016). During extrusion processing, ingredients such as carrot pomace, or other milling by-products such as wheat-bran and oat-bran can be transformed by the effect of barrel temperature and shear stress generated in the extruder chamber, causing in the pericarp fiber the releasing of bioactive compounds, such as polyphenols or flavonoids (Güven *et al.*, 2018). Also, changes in complex polysaccharides can occur, releasing low molecular weight compounds, increasing the soluble dietary fiber (Rashid *et al.*, 2015) from the ingredients under study, and impacting the biological, functional

and acceptability properties of the extruded cereals. However, no scientific information has been found on the use of carrot pomace mixed with wheat bran and/or oat bran for the production of high-fiber breakfast cereals by extrusion, which allows us to know the effect of the extrusion process conditions on the functional quality and acceptability characteristics of the extruded cereals. Therefore, the objective of the present work was to determine the physical, phytochemical and sensory characteristics of extruded high-fiber breakfast cereals prepared by combining carrot by-products with wheat and oat bran.

## 2 Material and methods

### 2.1 Raw materials

The breakfast cereals were produced using as raw materials wheat-bran (purchased in a local market, Culiacan, Mexico), oat-bran (purchased at the Mother Nature Company, Zapopan, Mexico), yellow corn grits (Industrial de Alimentos S.A., Mexico City, Mexico), powdered malt extract (Complementos Alimenticios S.A. de C.V. Mexico City, Mexico) and dehydrated carrot pomace (obtained as a partially humid by-product of the carrot juice artisan production in Culiacan, Mexico). The partially humid carrot pomace, added with 1.5% sucrose disolution for color retention (according to preliminary tests), was dehydrated in a hot air dryer at  $70 \pm 2^\circ\text{C}$  for 110 min. All raw materials were separately ground using a grinder Pulvex (model 200, Mexico City, Mexico), and sieved to a particle size  $\leq 420 \mu\text{m}$ . The extruded breakfast cereals were formulated using a "base blend" composed of wheat-bran, oat-bran, and yellow corn grits (52.7:36.0:11.3%, respectively). This base blend was added with a malt extract content of 4%, and a dehydrated carrot pomace content (CPC) in a range of 5.51 to 22.49%, in according to the experimental design (Table 1).

### 2.2 Production of breakfast cereals by extrusion

For the production of breakfast cereals, the aforementioned mixtures of raw materials were adjusted to a feed moisture content (FM) in the range of 19.34 to 30.66%, according to the experimental design (Table 1). Subsequently, these materials were stored for 12 h in polyethylene bags at  $4-6^\circ\text{C}$ ,

before being processing by extrusion. After that, the temperature of the samples was allowed to become steady at room temperature, and then the samples were fed at a feed rate of  $57 \pm 2 \text{ g/min}$ , using a forced flow conical feeder, into a single-screw extruder (Brabender brand, model 20DN / 8-235-00, Germany), of a 2:1 compression ratio at a constant speed of 110 rpm, and a 2-mm-diameter circular die. During the extrusion process the barrel temperatures in the feeding, mixing and exit zones, were maintained constant, being  $75$ ,  $130$  and  $125^\circ\text{C}$ , respectively. After leaving the extruder, the breakfast cereals were dehydrated at a temperature of  $52 \pm 1^\circ\text{C}$  for 24 h, until reaching moisture levels of 4-5%, and subsequently cut into pieces of 5 cm in length, or ground and sieved to particle size  $\leq 420 \mu\text{m}$ . Then, these materials were stored in black plastic bags at refrigeration conditions ( $4-6^\circ\text{C}$ ) until analyzes.

### 2.3 Proximate composition

This analysis was performed in the extruded breakfast cereals obtained in the best processing conditions, and in a commercial product (control), according to the procedures described by the AOAC (2005), determining ash (923.03), fat (920.39), moisture (925.10), crude fiber (962.09), protein (960.52), whereas the carbohydrate levels were obtained by difference. Three measurements were carried out for each response.

### 2.4 Expansion index (EI)

This determination was made to the extruded breakfast cereals according to the methodology reported by Gujska and Khan (1990). The EI was obtained by dividing the diameter of the breakfast cereals by the diameter of the output die of the extruder. Fifteen measurements per treatment were performed.

### 2.5 Flexural modulus (FMO)

This determination was carried out in the breakfast cereals obtained under different treatments of the experimental design, according to the procedure described by Aguilar-Palazuelos *et al.* (2007). These analyzes were done employing a universal texturometer (INSTRON 3342, Norwood, MA, USA) using a 500 N load cell, with a descent speed of 4 mm/min. A flexion test in three points was performed,

and the FMO calculated using the equation (1).

$$FMO = \frac{(P)(L^3)}{(D_{\max})(48)(I)} \quad (1)$$

Where,  $FMO$  = flexural modulus (MPa),  $D_{\max}$  = maximum deflection at the center (m),  $P$  = strength measured in the point of maximum deflection (N),  $L$  = length between the load point and the support base (m),  $I$  = moment of inertia of a cylinder (m<sup>4</sup>).

## 2.6 Water solubility index (WSI)

This analysis was performed in triplicate on raw materials, breakfast cereals obtained in each treatment of the experimental design, and on commercial products, according to the methodology reported by Anderson *et al.* (1969). In this determination, 0.25 grams (wb) of sample were mixed with 12 mL of water and stirred (Vari-Mix Aliquot Mixer, Dubuque, Iowa, USA) during 30 min. Subsequently, the tubes were centrifuged (Eppendorf 5804R, Hamburg, Germany) to 4500×g during 30 min, at 25°C. After that, the supernatant was decanted and evaporated. The WSI was calculated by dividing the weight of dried solids recovered after supernatant evaporation by the weight of dried solids of initial sample and was reported as percentage.

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

These determinations were done in the raw materials and breakfast cereals obtained in the different extrusion conditions using a tristimulus colorimeter (Minolta, CR-210, Tokyo, Japan). Four equally spaced readings were performed on grinded samples (particle size  $\leq 250 \mu\text{m}$ ) after being scattered evenly in a petri dish. Four measurements per treatment were carried out.

## 2.8 Dietary fiber (DF)

The analysis of DF was carried out according to the methodology described by AOAC (2005), method 985.29, in raw materials, and axial and central treatments of the experimental design. This test is based on the removal of starch and proteins by means of an enzymatic kit TDF-100A (Sigma-Adrich, St. Louis, MO, USA). The kit consists of three enzymes: a heat-stable amylase, a protease and an amyloglucosidase, and the soluble and insoluble dietary fibers were obtained by filtration.

## 2.9 Extraction of free and bound phenolic compounds

The procedure described by Adom and Liu (2002) with some modifications was used to obtain the free and bound fraction of phenolic compounds. The free fraction of phenolic compounds was obtained by adding 10 mL of 80% ethanol to one gram of defatted sample, stirring for 10 min, and then centrifuging to 3000×g, at 10°C during 10 min. The supernatant was recovered, and the above procedure was repeated four times. On the other hand, the bound fraction of phenolic compounds was obtained from the residue generated from the extraction of free phenolic compounds. A mixture of this residue with 10 mL of NaOH 2N was stirred and heated in a water bath at 95°C for 30 min. After that, the mixture was stirred and cooled to room temperature (25°C). Subsequently, 2 mL of concentrated HCl were added and stirred for 10 min and 10 mL of ethyl acetate were added, being centrifuged (3000×g/10°C/10 min) and the supernatant recovered four times. For each treatment of the experimental design, the supernatants of the two fractions (free and bound) were concentrated, in a separate form, at a temperature of 45°C using an evaporator Heidolph (model Laborota 4011, Schwabach, Germany). They were then reconstituted with 50% methanol to perform the measurements of phenolic compounds and antioxidant activity (DPPH).

## 2.10 Determination of free and bound phenolic compounds

The determination of free (FPC) and bound phenolic (BPC) compounds was carried out using the Folin-Ciocalteu spectrophotometric method, according to the procedure described by Heimler *et al.* (2006), with some modifications. Four repetitions per each treatment of the experimental design were performed, measuring the absorbance at 760 nm with a Model 10 spectrophotometer, UV-GENESYS, Series AQ7-2H7G229001, USA. The results of FPC and BPC were reported in mg gallic acid equivalent (GAE)/g db.

## 2.11 Flavonoids

This determination was carried out according to the methodology reported by Dewanto *et al.* (2002). 200  $\mu\text{L}$  of the extract of free phenolic compounds were placed in a 2 mL eppendorf tube, then 1000  $\mu\text{L}$  of distilled water and 60  $\mu\text{L}$  of NaNO<sub>2</sub> were added and, after 6 min, 120  $\mu\text{L}$  of AlCl<sub>3</sub> were added. Also,

400  $\mu\text{L}$  of NaOH 1M and finally 220  $\mu\text{L}$  of distilled water were added to complete a volume of 2 mL. The absorbance at 550 nm was measured in a Thermo Spectronic spectrophotometer (model GENESYS 10 UV, series 2H7G229001, USA), and the flavonoid content was expressed in mg equivalents quercetin (EQ)/g db). Three repetitions were performed per treatment.

### 2.12 Antioxidant activity (AOA, DPPH $\cdot$ )

The AOA provided by the free and bound extracts was determined by the DPPH $\cdot$  (2, 2-diphenyl-1-picrylhydrazyl) method, according to the procedure described by Brand-Williams *et al.* (1995). The absorbance of the DPPH $\cdot$  radical was adjusted with methanol in a range of 0.76-0.78 and read at 515 nm using a Thermo Spectronic spectrophotometer (model GENESYS 10 UV, series 2H7G229001, USA). Subsequently, 2900  $\mu\text{L}$  of DPPH $\cdot$  radical and 100  $\mu\text{L}$  of free or bound extracts were mixed and incubated for 30 min in dark, and the absorbance was read. Four repetitions were carried out in the different treatments, and the results were reported as  $\mu\text{mol}$  trolox equivalents (TE)/g db.

### 2.13 Sensory study

The sensory acceptability of both breakfast cereals, the added with carrot pomace obtained in the best processing conditions (FM = 24.48% and CPC = 20.21%), and a commercial breakfast cereal (control),

was assessed. For that purpose, 80 untrained panelists of both genders, over 18 years old performed the evaluation. A test of general acceptability was carried out, by means of a hedonic scale of 9 points, where 1 = dislike extremely, 2 = dislike very much, 3 = dislike moderately, 4 = dislike slightly, 5 = no like nor dislike, 6 = like slightly, 7 = like moderately, 8 = like very much, 9 = like extremely), according to the procedure reported by Delgado-Nieblas *et al.* (2018).

### 2.14 Statistical analysis

The data for the different variables of response were analyzed with a central composite rotatable experimental design with a value  $\alpha = 1.414$ . The independent variables were feed moisture content (FM, %) and the carrot pomace content (CPC, %), with five levels each factor (Table 1).

The second-order polynomial is shown in equation (2):

$$Y_i = b_0 + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2 \quad (2)$$

Where,  $Y_i$  is a generic response,  $X_1$  is the feed moisture content,  $X_2$  is the carrot pomace content, and  $b_0$ ,  $b_1$ ,  $b_2$ ,  $b_{11}$ ,  $b_{22}$ , and  $b_{12}$  are the regression coefficients. The multiple regression analysis and Fisher's LSD test to compare means of the sensory study were carried out using the statistical program Design Expert (Stat-Ease, 2008) version 7.1.6. In addition, Pearson correlations were performed with the statistical program Statistica 7.0 (Statsoft, 2004).

Table 1. Experimental design for the extrusion study.

Assay	Coded levels		Actual levels	
	X1	X2	FM (%)	CPC (%)
1	-1	-1	21	8
2	1	-1	29	8
3	-1	1	21	20
4	1	1	29	20
5	-1.414	0	19.34	14
6	1.414	0	30.66	14
7	0	-1.414	25	5.51
8	0	1.414	25	22.49
9	0	0	25	14
10	0	0	25	14
11	0	0	25	14
12	0	0	25	14
13	0	0	25	14

FM = feed moisture content; CPC = carrot pomace content.



### 3 Results and discussion

#### 3.1 Proximate composition of the breakfast cereals

The chemical composition (in dry basis) of the extruded high-fiber breakfast cereal obtained at FM = 24.48% and CPC = 20.21% (best processing conditions) presented a protein content of  $13.2 \pm 0.03\%$ , fat  $0.97 \pm 0.02\%$ , crude fiber  $7.28 \pm 0.01$ , ash  $5.99 \pm 0.15\%$ , and carbohydrates  $72.56\%$ , and a moisture content of  $4.86 \pm 0.01\%$ . In comparison, a commercial cylindrical-shaped high-fiber breakfast cereal showed a protein content of  $14.31 \pm 0.03\%$ , fat  $2.21 \pm 0.02\%$ , crude fiber  $6.36 \pm 0.12$ , ash  $4.05 \pm 0.03\%$ , and carbohydrates  $73.07\%$ , with a moisture content of  $4.17 \pm 0.02\%$ . The highest values of ashes in the breakfast cereal with addition of carrot pomace in relation to the commercial product may be due to the outstanding content of minerals found in carrot pomace (Gull *et al.*, 2015). Also, the lower contents of protein and fat in carrot pomace contributed to lower values of these components in the breakfast cereals obtained from this by-product. In addition, wheat bran, which was the main ingredient of commercial breakfast cereal provides important levels of protein

and fat (Talukder and Sharma, 2010), which allowed to increase the values for these properties in the commercial breakfast cereal.

#### 3.2 Regression coefficients and statistical analysis

In the Table 2 and Table 3 is presented the statistical information for the variables of response analyzed in the extruded breakfast cereals with addition of carrot pomace. The statistical information for the expansion index (EI), flexural modulus (FMO), water solubility index (WSI), and color parameters  $b^*$  and  $L^*$  is shown in Table 2. Also, the information for the responses free phenolic compounds (FPC), bound phenolic compounds (BPC), antioxidant activity from free phenolic compounds (FAOA-DPPH) and antioxidant activity from bound phenolic compounds (BAOA-DPPH) is presented in Table 3. The adjusted second order model resulted in  $R^2$  values  $\geq 0.65$  for all response variables (except FMO). The obtained mathematical models were significant ( $p < 0.05$ ) and showed no lack of fit ( $p > 0.05$ ). The feed moisture (FM) factor presented a significant effect ( $p < 0.05$ ), in its linear term, on the FMO, color  $b^*$ , BPC, FAOA-DPPH and BAOA-DPPH.

Table 2. Regression coefficients of the models, significant levels and analysis of variance for the studied responses in extruded breakfast cereals.

	EI	FMO (MPa)	WSI (%)	Color parameter $b^*$	Color parameter $L^*$
<b>Intercept</b>	1.01	78.13	17.52	28.54	77.67
<b>Linear</b>					
FM	0.0011 (0.955)	13.09 (0.008)	0.009 (0.92)	-1.37 (0.005)	-0.82 (0.172)
CPC	-0.0033 (0.811)	4.53 (0.143)	1.60 (< 0.001)	0.73 (0.029)	-1.13 (0.032)
<b>Quadratic</b>					
FM	0.024 (0.168)	-6.93 (0.118)	—	1.32 (0.003)	0.60 (0.222)
CPC	—	—	-0.58 (0.006)	-0.39 (0.084)	-0.69 (0.171)
<b>Interactions</b>					
FM x CPC	—	4.97 (0.239)	0.26 (0.077)	0.62 (0.043)	1.02 (0.107)
FM <sup>2</sup> x CPC	—	—	—	0.90 (0.040)	—
FM x CPC <sup>2</sup>	-0.087 (0.018)	—	—	1.66 (0.008)	2.57 (0.021)
$R^2$	0.65	0.60	0.97	0.96	0.76
CV	3.68	10.02	1.46	1.26	1.27
$p$ of $F_{(model)}$	0.03	0.04	< 0.001	0.006	0.04
Lack of fit	0.087	0.331	0.453	0.468	0.554

FM = feed moisture content; CPC = carrot pomace content; EI = expansion index; FMO = flexural modulus; WSI = water solubility index; CV = coefficient of variation. Numbers within brackets indicate significance levels; dashes indicate terms of model non-used.

Table 3. Regression coefficients of the models, significant levels and analysis of variance for the studied responses in extruded breakfast cereals.

	FPC (mg GAE/g)	BPC (mg GAE/g)	FAOA (DPPH) ( $\mu\text{mol TE/g}$ )	BAOA (DPPH) ( $\mu\text{mol TE/g}$ )
<b>Intercept</b>	0.68	0.17	2.07	2.26
<b>Linear</b>				
FM	—	0.053 (< 0.001)	-0.054 (0.025)	0.032 (0.043)
CPC	0.049 (< 0.001)	-0.043 (< 0.001)	0.030 (0.163)	-0.016 (0.256)
<b>Quadratic</b>				
FM	—	-0.031 (0.002)	-0.20 (< 0.001)	-0.034 (0.040)
CPC	-0.020 (0.005)	0.011 (0.047)	-0.063 (0.017)	-0.026 (0.101)
<b>Interactions</b>				
FM x CPC	—	—	—	-0.068 (0.007)
R <sup>2</sup>	0.89	0.96	0.89	0.67
CV	2.25	7.83	2.90	1.65
<i>p</i> of $F_{(model)}$	< 0.001	< 0.001	< 0.001	0.018
Lack of fit	0.912	0.763	0.265	0.145

FM = feed moisture content; CPC = carrot pomace content; FPC = free phenolic compounds; BPC = bound phenolic compounds; FAOA = free antioxidant activity; BAOA = bound antioxidant activity; CV = coefficient of variation. Numbers within brackets indicate significance levels, dashes indicate terms of model non-used.

In the same way, FM showed a significant effect ( $p < 0.05$ ) in its quadratic term on the color  $b^*$ , BPC, FAOA-DPPH and BAOA-DPPH. On the other hand, the carrot pomace content (CPC) factor, presented significant effect ( $p < 0.05$ ) in its linear term on the WSI, color  $b^*$ , color  $L^*$ , FPC and BPC, whereas CPC showed a significant effect ( $p < 0.05$ ) in its quadratic term on WSI, FPC, BPC, and FAOA-DPPH. In the analysis of interactions, the interaction FM-CPC showed a significant effect ( $p < 0.05$ ) on the color  $b^*$  and BAOA-DPPH, whereas the interaction FM-CPC<sup>2</sup> presented significant effect ( $p < 0.05$ ) on EI, color  $b^*$ , and color  $L^*$ , whereas the interaction FM<sup>2</sup>-CPC presented significant effect ( $p < 0.05$ ) on the color  $b^*$ .

### 3.3 Expansion index (EI)

The expansion index is a physical property of extruded products that can be influenced by different factors during the extrusion process, such as feed moisture content, extrusion temperature, screw speed and feed rate. Additionally, the dietary fiber provided by the raw materials used to produce the extruded foods can have important effect on this parameter (Ying *et al.*, 2017). High fiber breakfast cereals with intermediate expansion values are desired, because products with high expansion are brittle, while those with low expansion are hard to chew. In Figure 1a is presented the effect of feed moisture content (FM) and carrot pomace content (CPC) on the expansion index values

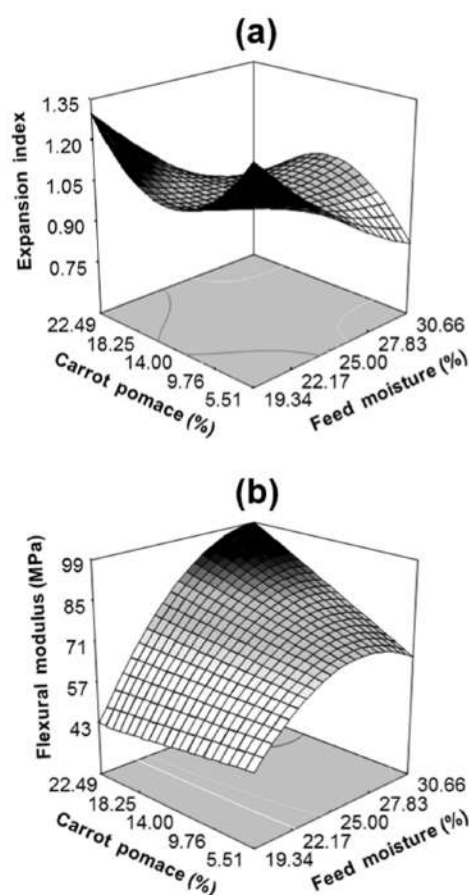


Fig. 1. Effect of feed moisture and carrot pomace content on expansion index (a) and flexural modulus (b) of extruded breakfast cereals.



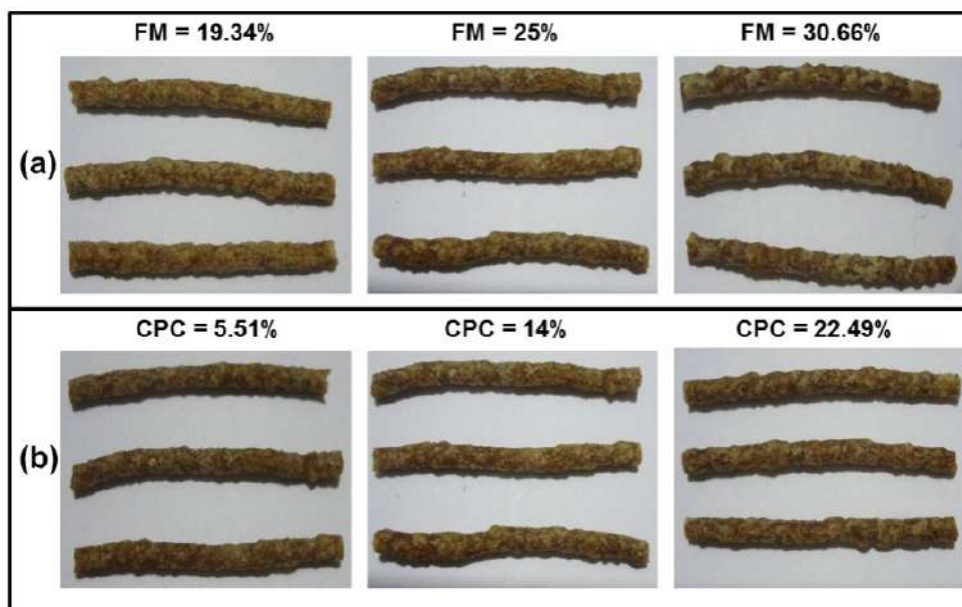


Fig. 2. Photographs showing the visual effect of feed moisture (a, 19.34%, 25%, 30.66%), at CPC = 14% and of carrot pomace content (b, 5.51%, 14%, 22.49%), at FM = 25% on expansion index of breakfast cereals.

of breakfast cereals produced by the extrusion process, and Figure 2 shows photographs of the breakfast cereals obtained at different extrusion conditions. The highest values of EI ( $> 1.1$ ) were presented at low FM levels ( $< 22\%$ ) throughout the CPC range. The EI presented a negative correlation with FM ( $r = -0.58$ ,  $p = 0.03$ ). This behavior may be due to the high friction inside the extruder barrel generated at low FM levels. The structure of starch and fiber could have been modified causing a greater retention of water that favored the expansion of the breakfast cereals when leaving the extruder through the die. Charunuch *et al.* (2011) reported a similar behavior in breakfast cereals made from rice, with addition of sweet potato, purple cabbage, and beet, where the highest values of EI were presented at low FM. Also, Oliveira *et al.* (2017) obtained the highest values of expansion at low FM levels in extruded breakfast cereals produced from whole grain wheat flours and corn flours, reporting expansion values from  $1.03 \pm 0.16$  to  $2.45 \pm 0.18$ , which are slightly higher than those found in the present study. Similarly, Borah *et al.* (2015) reported a significant increase in EI as FM decreased in breakfast cereals produced from rice and banana. In the present work, it can be observed that at high levels of FM, in combination with high and low CPC, the EI values decreased possibly due to a plasticizing effect as a consequence of the high levels

of FM and the sugar components of the carrot pomace. According to Anderson and Simsek *et al.* (2019), the plasticizers limit the interaction of polymers with water. This could have generated a lesser degree of modification in the structure of the extruded materials due to shorter residence times within the extruder, resulting in less water absorption, collapsed air cells, and reduced EI values.

### 3.4 Flexural modulus (FMO)

Texture in extruded breakfast cereals is a quality parameter, which can have an important influence on the consumer acceptability of these products (Dos Santos *et al.*, 2019), whose high hardness and low expansion are related to high values of FMO. The effect of FM and CPC on the FMO values of extruded breakfast cereals is shown in the Figure 1b. The lowest values of FMO ( $< 60$  MPa) were presented at low FM levels ( $< 22\%$ ) throughout the CPC range. FMO presented a positive Pearson correlation with the factor of study FM ( $r = 0.69$ ,  $p = 0.03$ ). This behavior could be due to the fact that as FM levels increased, the expansion values decreased, producing breakfast cereals with higher hardness values and elevated levels of FMO. Furthermore, under conditions of high FM and CPC, breakfast cereals with higher FMO values were obtained. This could have been

due to the high fiber values present in high CPC. According to Aguilar-Palazuelos *et al.* (2007), the fiber presents high water absorption, and during the extrusion process the distribution of water is diminished, resulting in reduced expansion and high FMO and mechanical strength values. Also, Oliveira *et al.* (2017) reported that in extruded breakfast cereals the presence of dietary fiber increases the hardness, mainly at high moisture contents. These authors attributed this behavior to a reduced elasticity and weakening of the cellular structure, due to interactions between starch and fiber. In addition, Chanvrier *et al.* (2014) mentioned that the addition of fiber in extruded cereals affects the internal structure of these products, decreasing porosity and expansion. In the present study, the higher expansion values in breakfast cereals were presented under low FM conditions. According to Hsieh *et al.* (1990), in extruded products with high expansion, larger air cells with thinner walls are formed, which decreases the force necessary to produce the deformation.

### 3.5 Water solubility index (WSI)

The WSI reflects the level of macromolecular degradation due to the extrusion process (Gopirajah and Muthukumarappan, 2018) in materials such as fiber and starch. In Figure 3a is observed the effect of FM and CPC on WSI of extruded breakfast cereals. It can be observed that the highest values of WSI ( $> 17.22\%$ ) were presented at high levels of CPC ( $> 14\%$ ) throughout the range of FM. The variable of response WSI presented a positive Pearson correlation with the factor CPC ( $r = 0.95$ ,  $p = 0.001$ ). This behavior could be due to the high values of WSI presented by the carrot pomace raw material ( $33.19 \pm 0.42\%$ ) in relation to the rest of materials used for the production of the breakfast cereals. The wheat bran presented a value of  $15.22 \pm 0.33\%$ , oat bran  $6.82 \pm 0.42\%$ , and yellow corn grits  $0.84 \pm 0.06\%$ . Therefore, as CPC was increased, the WSI was elevated. Also, the extrusion variables (barrel temperature, shear) could have caused an increase in the WSI, since this process has been reported to convert insoluble dietary fiber to soluble dietary fiber (Rashid *et al.*, 2015). The WSI values obtained in the present study for the breakfast cereals are close to those reported by Yao *et al.* (2011) in breakfast cereals made with whole oat flour and wheat starch, who reported values from 13.8 to 18.0%. Also, it can be observed that by combining high levels of FM and CPC, high WSI values were obtained. This may be due to the gelatinization of the starches

promoted by the high levels of FM, allowing these materials to depolymerize in smaller molecules which possess a greater water solubility. According to Ding *et al.* (2005), the starch gelatinization is presented by effect of the extrusion process due to the interaction of this polymer with water and heat, presenting changes in the granular material. Likewise, a comparison was made of WSI values in breakfast cereals with addition of carrot pomace (BCP, obtained at FM = 24.48% and CPC = 20.21%) in relation to a breakfast cereal control (BCC) without carrot, and a commercial breakfast cereal (BCOM). BCOM had the highest WSI value ( $28.49 \pm 0.28$ ), followed by BCP with  $17.56 \pm 0.27\%$ , while BCC showed a value of  $11.03 \pm 0.33\%$ . The low WSI levels of the BCP compared with a commercial product (BCOM) is a positive finding, since low WSI values are recommended, because these products are consumed accompanied with liquid milk. In these conditions the breakfast cereals must not be excessively soft, which allows a faster disintegration. Charunuch *et al.* (2014) reported that high levels of WSI cause important dextrinization, stickiness and softness levels, when the cereals are placed in liquid milk. These are undesirable characteristics for breakfast cereals consumed by people.

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

The effect of FM and CPC on the color parameter  $b^*$  of extruded breakfast cereals is shown in Figure 3b. It is observed that the highest  $b^*$  values ( $> 29$ ) were obtained by combining high levels of FM ( $> 28\%$ ) and CPC ( $> 15\%$ ). The color parameter  $b^*$  presented a positive correlation with the response WSI ( $r = 0.81$ ,  $p = 0.008$ ), and with the factor of study CPC ( $r = 0.79$ ,  $p = 0.01$ ). This behavior could have been due to the higher levels of carotenoids in carrot pomace (Baljeet *et al.*, 2014) in relation to the other raw materials used to produce the breakfast cereals. Likewise, high levels of FM could have presented a plasticizing effect, decreasing the residence time of the materials inside the extruder, allowing a lower exposure of the carotenoids to the heating and shear forces, and therefore a greater retention of the color  $b^*$ . Also, high values of the color  $b^*$  were presented in conditions of high CPC and low FM ( $< 22\%$ ). This could be partially due to the releasing of carotenoids as a consequence of an increased friction presented inside the extruder at low FM levels. According to Basto *et al.* (2016), the heating and shearing produced during the extrusion process can cause a disruption in the cell

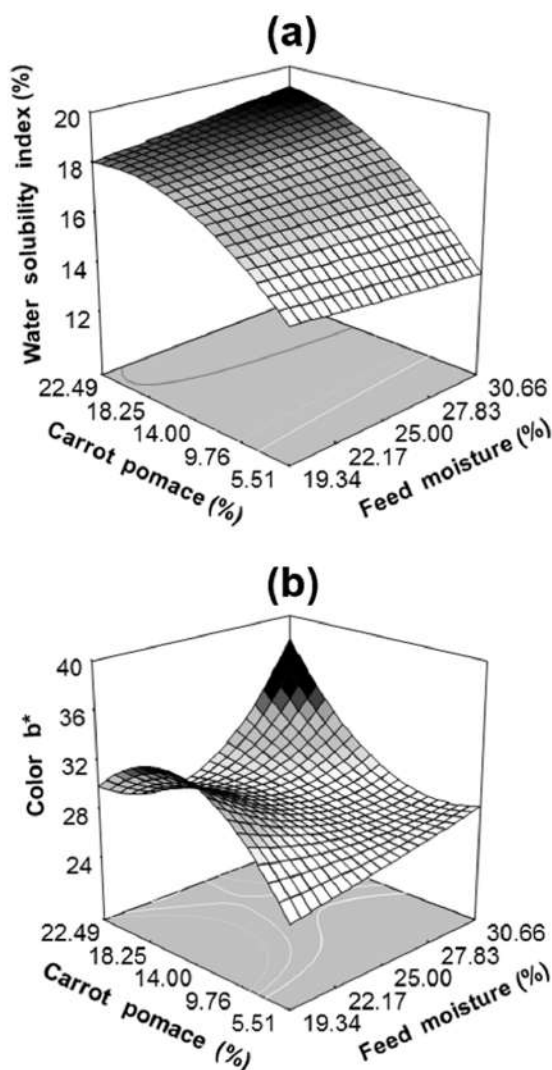


Fig. 3. Effect of feed moisture and carrot pomace content on water solubility index (a) and color  $b^*$  (b) of extruded breakfast cereals.

wall, thus facilitating the extractability of carotenoids. In the present work were obtained  $b^*$  values from 26.8 to 33.2, which were higher than those reported by Gull *et al.* (2015) in extruded pasta with addition of carrot pomace, who reported values from 9.97 to 24.76. On the other hand, the lower values of  $L^*$  ( $< 74$ ) (Figure not shown) were presented by combining low FM ( $< 25\%$ ), with high ( $> 15\%$ ) and low ( $< 9\%$ ) levels of CPC. This could be due to Maillard darkening reactions, which are presented during the extrusion process (Camire *et al.*, 1997) mostly at low moisture levels, due to the friction generated inside the extruder. The above mentioned, could have allowed greater darkening of the samples, decreasing the  $L^*$  values.

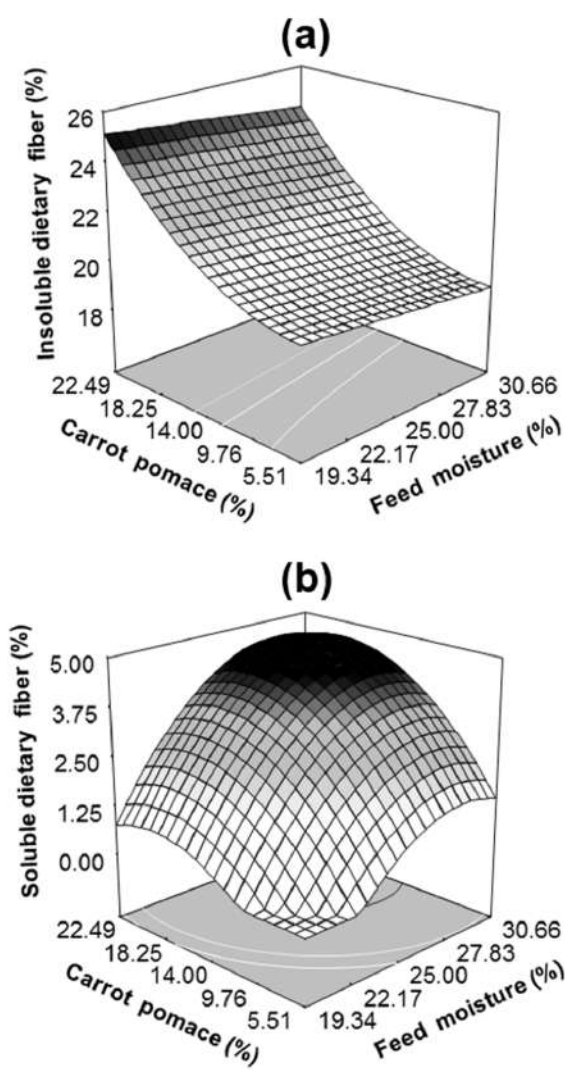


Fig. 4. Effect of feed moisture and carrot pomace content on insoluble dietary fiber (a) and soluble dietary fiber (b) of extruded breakfast cereals.

However, when low levels of FM were combined with intermediate levels of CPC, high values of  $L^*$  ( $> 80$ ) were obtained. This could be due to the fact that in these same conditions' high values of color  $b^*$  parameter were presented as previously mentioned, increasing the yellow color in the breakfast cereals, which is a color that has a high capacity to reflect light (Weiss *et al.*, 2012), elevating the luminosity, and increasing  $L^*$  values.

### 3.7 Dietary fiber

In Figure 4a is shown the effect of FM and CPC on the insoluble dietary fiber (IDF) values of the



breakfast cereals. It can be observed that as CPC levels were increased throughout the FM range, IDF values augmented ( $> 22\%$ ). This may be due to the high content of insoluble dietary fiber found in the raw material carrot pomace ( $41.28 \pm 4.46\%$ ), in relation to the “base mixture” composed by wheat-bran, oat bran and yellow corn grits, that was used for the production of breakfast cereals which presented a value of  $27.90\%$ . Similarly, a slight reduction in IDF values were observed as FM increased, mainly at high CPC levels. This could have caused a degradation of insoluble fiber polymers, producing compounds of lower molecular weight. The behavior coincides with that reported by Rashid *et al.* (2015), who studied the effect of the extrusion process on the dietary fiber content of extruded products made from wheat bran. These authors reported that an increase in the FM levels from 20 to  $26\%$  allowed a reduction in the IDF and an increase in the soluble dietary fiber (SDF). The above authors reported that the processing by extrusion could have caused a decrease in the molecular weight of polymers, generating small fragments with higher water solubility and increasing SDF. In the present study, a similar trend can be observed in Figure 4b, showing the highest values of SDF by combining high levels of FM with high CPC. A similar behavior was observed by Martín-Cabrejas *et al.* (1999), who studied the effect of the extrusion process on the dietary fiber content of extruded beans, reporting redistribution from IDF to SDF during this process. Also, these authors found that as FM levels increased to  $30\%$ , the soluble fiber values increased. Likewise, the high values of SDF ( $> 4\%$ ) at intermediate and high levels of CPC ( $> 14\%$ ) could be due to the higher content of SDF in the raw material carrot pomace ( $6.87 \pm 0.47\%$ ) in relation to the value of SDF presented by the “base mixture” ( $4.66\%$ ) composed by wheat bran, oat bran, and yellow corn grits. Therefore, the important content of soluble dietary fiber provided by carrot pomace, in addition to the levels of soluble fiber provided by oat bran, mainly  $\beta$ -glucans, could contribute to obtain important benefits in the reduction of glycaemic response and blood cholesterol (Brennan *et al.*, 2013), decreasing the risk of suffering cardiovascular diseases.

### 3.8 Free (FPC) and bound (BPC) phenolic compounds

In Figure 5a is shown the effect of FM and CPC on the free phenolic compounds (FPC) of the extruded breakfast cereals. It can be observed that the highest

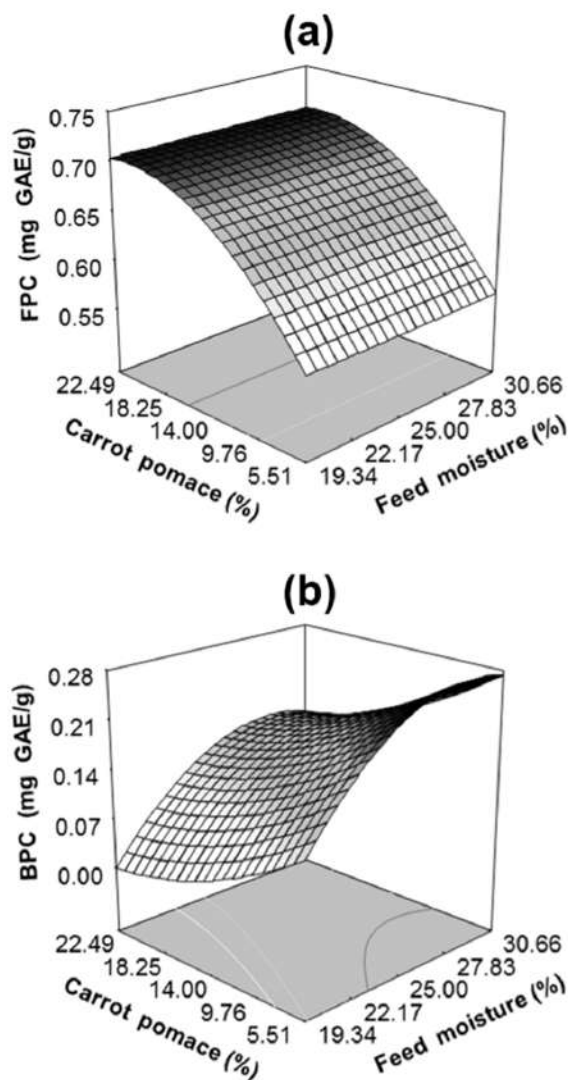


Fig. 5. Effect of feed moisture and carrot pomace content on free phenolic compounds (FPC, a) and bound phenolic compounds (BPC, b) of extruded breakfast cereals.

values of FPC ( $> 0.69$  mg GAE/g) were presented at high levels of CPC throughout the FM range. This behavior could be due to the higher content of FPC in the raw material carrot pomace ( $0.52 \pm 0.056$  mg GAE/g), compared with the other raw materials used for the production of breakfast cereals. The oat bran showed a FPC value of  $0.42 \pm 0.05$  mg GAE/g, while the yellow corn grits showed a value of  $0.045 \pm 0.07$  mg GAE/g. The value of FPC in carrot pomace is slightly higher than the value of total phenolic compounds reported by Jabbar *et al.* (2015) in carrot pomace, who reported a maximum value of  $0.316$  mg GAE/g. Also, in the present study, the highest values

of free flavonoids (0.31 mg quercetin equivalents/g db, Figure not shown), representing approximately 44% of FPC, were obtained in the same conditions where the highest FPC values were obtained (CPC > 12%). The main phenolic compounds in carrot are hydroxycinnamic acid derivatives such as chlorogenic acid, followed by flavonoids such as kaempferol, quercetin and luteolin (Ergun and Süslüoğlu, 2018). Flavonoids are powerful antioxidants that can remove free radicals from the body and prevent the incidence of various diseases (Zheng *et al.*, 2021). Similarly, in this study, the FPC could have increased due to the release of phenolic compounds from cereals by the extrusion process. It has been reported that cereals have a high content of phenolic compounds, where the ferulic acid is the main compound (Adom and Liu, 2002), being mostly linked to cell walls. The FPC response presented a positive correlation with WSI ( $r = 0.96$ ,  $p = 0.031$ ) and the color  $b^*$  parameter ( $r = 0.78$ ,  $p = 0.020$ ), where its highest values were also presented at high CPC. Likewise, In Figure 5b are shown the values of bound phenolic compounds (BPC) as an effect of the FM and CPC factors. It can be observed that the highest BPC values (> 0.25 mg GAE/g) were presented at low levels of CPC (< 9%) in combination with high FM levels (> 25%). The BPC presented a high negative Pearson correlation ( $r = -0.97$ ,  $p = 0.038$ ) with the factor CPC. This could be due to the low amount of BPC in the carrot pomace in relation to the other raw materials used in this study, such as wheat and oat bran, as well as yellow corn grits. According to Călinoiu and Vodnar (2018), in cereal grains the bran is the most important source of phenolic acids and dietary fiber, with high antioxidant potential. The previous authors reported that the hydroxycinnamic phenolic acids (p-coumaric, caffeic, ferulic, and sinapic) are an important part of bran fraction in wheat and oat, being mainly covalently bound to cell wall polymers. Also, in the present study at low levels of CPC, as the FM levels were increased, the BPC were elevated. This could be due to the fact that higher moisture in the samples presented a plasticizing effect inside the extruder. This may have diminished the residence time, shearing forces and rupture of the bound phenolic compounds during the thermomechanical process (Güven *et al.*, 2018). In the present study, the sum of FPC and BPC produced values of total phenolic compounds between 0.71 and 0.88 mg GAE/g. These values were slightly higher than those reported by Leyva-Corral *et al.* (2016) in extruded breakfast cereals produced from oat, potato starch and apple pomace, who reported values of total

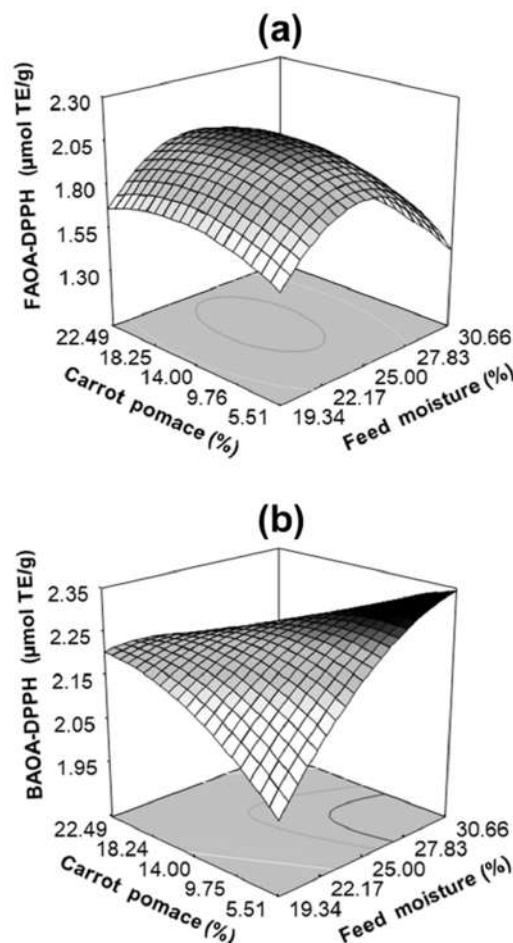


Fig. 6. Effect of feed moisture and carrot pomace content on the free antioxidant activity (FAOA-DPPH, a) and the bound antioxidant activity (BAOA-DPPH, b) of extruded breakfast cereals.

phenolic content between 0.52 and 0.64 mg GAE/g db.

### 3.9 Antioxidant activity (DPPH) from free and bound phenolic compounds

Figure 6a shows the effect of FM and CPC on the antioxidant activity measured from the extracts of free phenolic compounds (FAOA-DPPH) of the breakfast cereals obtained in the present study. It can be observed that the highest values of FAOA-DPPH (> 2  $\mu\text{mol TE/g db}$ ) were obtained at intermediate and high levels of CPC in combination with intermediate levels of FM. This behavior could be due to the higher values of FAOA-DPPH presented by the raw material carrot pomace ( $1.27 \pm 0.07 \mu\text{mol TE/g}$ ), compared with the other raw materials used for the preparation of breakfast cereals. Likewise, the extrusion process



could have released phenolic compounds linked to the cell wall present in materials such as wheat bran, oat bran and yellow corn grits used for the production of breakfast cereals. According to Adom and Liu (2002), corn has the highest antioxidant activity among cereals, followed by wheat and oat. However, at high FM throughout the CPC range a decrease in FAOA-DPPH values was observed. This could be due to a lubricating effect at high FM, reducing the severity of the treatment and the conditions to release phenolic compounds, mainly contained in the cereal fraction. Also, low values of FAOA-DPPH were presented at low FM levels throughout the range of CPC. This could be due to the fact that in these processing conditions high friction could have occurred inside the extruder barrel, causing a greater thermomechanical degradation of the phenolic compounds, and therefore greater reduction of the FAOA-DPPH values. On the other hand, in Figure 6b it is observed that the highest values ( $> 2.3 \mu\text{mol TE/g}$ ) of antioxidant activity from bound phenolic compounds (BAOA-DPPH) were obtained at low levels of CPC ( $< 9\%$ ) and high FM ( $> 28\%$ ). BAOA-DPPH presented a negative correlation of Pearson ( $r = -0.97$ ,  $p = 0.029$ ) with the CPC factor. This behavior could be due to a lower antioxidant capacity of the bound extracts provided by the carrot pomace, since in the processing conditions in which lower levels of carrot pomace were used, higher levels of cereal bran and yellow corn grits were presented. Miller *et al.* (2000) reported that antioxidant compounds in grains are more concentrated in the bran fraction, however its presence in the endosperm fraction is also important. In the same way, in the present work, the occurring behavior coincides with the higher values of bound phenolic compounds obtained under the same extrusion conditions. Likewise, the high values of BAOA-DPPH at high FM levels could be due to a greater fluidity of the materials within the extruder, promoted by the high moisture levels, thus decreasing the thermomechanical damage, and causing a greater retention of phenolic compounds. According to Oniszczuk *et al.* (2019), the phenolic acids present in cereals, mainly cinnamic acid derivatives, have a high capacity to capture free radicals, thus protecting lipids and proteins from oxidation, and presenting ability to chelate metal ions capable of accelerating oxidation reactions. Also, Medina-Torres *et al.* (2021) reported that the phenolic compounds have high biological activity and present the ability to reduce the risk of degenerative diseases.

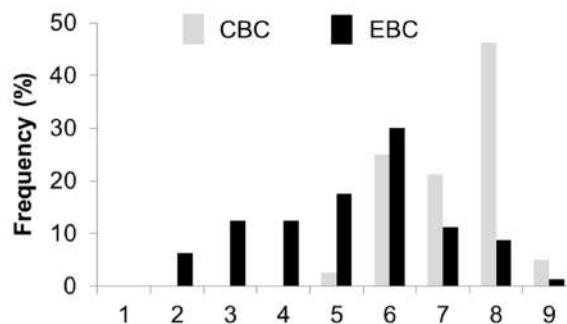


Fig. 7. Frequency analysis of the general acceptability test (1 = dislike extremely; 2 = dislike very much; 3 = dislike moderately; 4 = dislike slightly; 5 = neither like nor dislike; 6 = like slightly; 7 = like moderately; 8 = like very much; 9 = like extremely) in the sensory study of high-fiber breakfast cereals (CBC = commercial product; EBC = extruded breakfast cereal with addition of carrot pomace).

### 3.10 Sensory study

In Figure 7 are shown the results of the general acceptability test for the extruded breakfast cereal (EBC) obtained under the conditions of FM = 24.48% and CPC = 20.21% (best processing conditions), and those of a commercial breakfast cereal (CBC). The results showed that in EBC, 69% of the panelists indicated a degree of acceptance  $\geq 5$ , whereas 31% of them mentioned that they disliked the products. Likewise, the results showed that 100% of the panelists selected values from the hedonic scale  $\geq 5$  in CBC. The mean comparison test indicated that there was significant statistical difference ( $p < 0.05$ ) in the acceptability of the two products, showing CBC a greater level of acceptability. Likewise, when the breakfast cereals general acceptability between men and women was compared, no significant statistical difference was presented ( $p > 0.05$ , data not shown). The obtained results in EBC are considered positive, since panelists commented that the product presented acceptable characteristics for color, texture and flavor, when asked for general comments. The resulting difference between EBC and CBC could be due to the flavor attribute, since the panelists commented that the commercial product had a more pleasant flavor. This could be due to the sugars added to the commercial breakfast cereals during their production process (Mesías *et al.*, 2019), thus improving its acceptability by consumers. However, Dos Santos *et al.* (2019) reported that it is important for the extruded breakfast cereals to be of natural taste, since commercial product taste has been based generally on high amounts

of sugar and artificial flavorings. Therefore, it is recommended to reduce these ingredients in the formulation of breakfast cereals, to reduce obesity, which is a public health problem. The breakfast cereals with the addition of carrot pomace have the advantage of having a high content of bioactive compounds, such as phenolic compounds and carotenoids, with acceptable antioxidant properties. In addition, these products have a significant content of dietary fiber, the consumption of which has potential benefits for human health.

## Conclusions

The feed moisture and the carrot pomace content evaluated as independent variables showed a significant effect ( $p < 0.05$ ) on the different physical, physicochemical and nutritional/nutraceutical responses. The characterization of high-fiber extruded breakfast cereals showed the great relevance of carrot pomace as a source of dietary fiber, phenolic and flavonoids compounds, as well as antioxidant activity. Also, the findings of the present study indicated the importance of wheat and oat bran as a source of bound phenolic compounds. The breakfast cereals presented acceptable sensory properties and no sugar was added. These products showed acceptable physical, physicochemical and nutritional/nutraceutical properties. The consumption of this type of breakfast cereals has potential benefits for human health due to the important content of bioactive compounds provided by the raw materials used for their production.

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