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Effect of nixtamalization extrusion conditions in purple creole corn (*Zea mays* L.) from the state of Guerrero on nutraceutical and functional properties of the optimized corn flour

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Abstract

In Mexico, pigmented creole corn varieties are used for its consumption in a great diversity of foods. Purple corn has a high content of phytochemicals (health promoters). The extrusion process improved the accessibility of these phytochemicals. The aim of this work was to evaluate the effect of extrusion factors on the nutraceutical and functional properties of nixtamalized purple corn flour, and determine the optimal process conditions that allows obtaining a flour with the best characteristics to be used in the preparation of a beverage. The processing factors were temperature (T = 50-170 °C) and screw speed (SS = 50-240 rpm). The properties evaluated were total phenolic compounds (TPC), total anthocyanins (TA) antioxidant activity (AOX), and water solubility index (WSI). The optimization was carried out using the response surface methodology. The optimization yielded the following values: AOX: 676.34 μmol ET/100 g, TPC: 299.61 mg EGA/100 g, TA: 29.9 mg EC3G/100 g and WSI: 6.31%, under process conditions T = 50 °C and SS = 50 rpm. It was demonstrated that it is possible to generate a flour with the use of pigmented corn native to the state of Guerrero, with suitable properties for developed new corn based products.

Keywords: creole purple corn; functional foods; optimization; phytochemicals; extrusion.

Practical Application: The extrusion process is an alternative for corn processing, compared to nixtamalization, it is more environmentally friendly, improves nutritional and technological properties and the phytochemical potential of the food. Antioxidant activity, water absorption index, phenolic compounds and total anthocyanins are indicators of the quality of corn flour and its potential in the food industry. The results obtained in this research can provide a useful guide to develop and optimize innovative pigmented corn-based products.

1 Introduction

Mexico is characterized by a wide diversity of germplasm of corn (*Zea mays* L.). The state of Guerrero occupies the sixth place in corn production at the national level. During 2019-2021, a harvest of one million 335 thousand 918 tons of corn was recorded in the state, being the Costa Chica region the area with the largest planted area. The different conditions of the state of Guerrero (ranging from sea level to 2,880 masl) allows the development of a great diversity of creole corn (González-Mateos et al., 2018).

Unfortunately, the production of pigmented creole corn is not driven by public policies, on the contrary, they cause a joint displacement with transnational corporations that promote the use of hybrid corn (Bello-Pérez et al., 2016). In Guerrero, the pigmented varieties are still being planted on a smaller scale (less than 10% off the total production) since the producers use the creole pigmented corn mainly for self-consumption and not for marketing (Rivera-Castro et al., 2020).

Recently, the issuance of a law that protects, promotes and respects creole corn with its various varieties was approved. This seeks to declare creole corn as a National Food Heritage, and in this way promote its development, productivity, competitiveness and biodiversity. In addition, some researchers in various states have joined efforts to promote the creation of seed banks to promote and protect corn, allowing the identification and selection of those with greater properties that can be used in improvement programs (Velázquez-Cardelas et al., 2018).

According with the literature, purple corn has a high nutritional value. The nutritional composition of creole purple corn grains is superior to that from hybrids white genotypes, with a high starch and protein content predominating. In addition to these macronutrients, purple corn contain micronutrients such as antioxidants, which, although not essential for humans, help maintain good health and have been found to reduce the possibility of contracting chronic degenerative diseases (Lagunas et al., 2022; Rodríguez-Salinas et al., 2020).

Extrusion is a versatile process that allows to obtain instant flour that can be used to make foods such as a breakfast cereals, snacks and precooked flours (Pacheco-Delahaye et al., 2008) The combination of temperature and screw speed promotes the release of phytochemicals from the food matrix and thus being

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more accessible to exert its antioxidant activity with potential health effects (Wang et al., 2013). The extrusion process also leads to the pregelatinization of the starch granules, causing the loss of molecular order and the complete degradation of the polymers with the formation of highly soluble fragments. Flour suspensions precooked by extrusion presenting a low tendency to the formation of lumps, and could being suitable for obtaining powdered products that are quickly prepared such as beverages (Pacheco-Delahaye et al., 2008).

The state of Guerrero is a territory with a great diversity of creole corn, for that we consider it is relevant to study, analyze and identify those materials that show adequate nutritional, nutraceutical and functional characteristics for the development of food products, due there is little scientific information about it. Giving added value to these pigmented corn varieties, thereby contributing to the promotion of their planting and cultivation and improving their commercial level. The aim of this work was to evaluate the effect of extrusion factors on the nutraceutical and functional properties of nixtamalized purple corn flour, and determine the optimal process conditions that allows obtaining a flour with the best characteristics to be used in the preparation of a beverage.

2 Materials and methods

2.1 Vegetal material

The purple creole corn used was cultivated and collected during the spring-summer 2018 cycle, in the experimental garden of the Regional Center for Higher Education of the Costa Chica, of the Autonomous University of Guerrero, located in the municipality of Florencio Villareal in the state from Guerrero, on the Cruz Grande-Ayutla highway, km 0.5, at the geographic coordinates of 16° 44' 02.2" North latitude and 99° 07' 48.5" West longitude; with a warm sub-humid climate (Aw), precipitation 1100 mm, average annual temperature of 25 °C, average relative humidity of 85% at 16 masl (Instituto Nacional de Estadística y Geografía, 2010).

2.2 Sample preparation

The corn kernel was pulverized to a particle size ≤ 0.420 mm using a laboratory grain mill (Model LM3100, Perten Instrument, Stockholm, Sweden). Subsequently, the ground corn was conditioned with distilled water until obtaining a moisture of 15%. Then were packed in polyethylene bags and stored (4 °C/8 h). Before extrusion, the samples were tempered (25 °C/1 h).

2.3 Obtaining extruded purple corn flour

To obtain extruded purple corn flours, the process described by Milán-Carrillo et al. (2012) was used with minor modifications. The extrusion was carried out in a model 20DN single screw extruder (CW Brabender Instruments, Inc., NJ, USA) with a screw diameter of 19 mm; length-diameter ratio 20:1; 1:1 compression ratio and 3mm output socket. The extrusion process was optimized to obtain extruded corn flour with high content of total phenolic compounds (TPC), anthocyanins (TA), antioxidant activity (AOX), and water solubility index (WSI). The response surface methodology, as an optimization tool, was applied on the four response variables. A rotating central composite experimental design was used with two factors [Temperature (T, 50-170 ºC), screw speed (SS, 50-240 rpm)] in five levels; 13 treatments were generated. The 13 treatments were runed, with different T/SS combinations, presented in Table 1. The extrudates from each treatment were collected and cooled to the environment. The dried extrudates were ground (Perten Laboratory Mill, Model 3100) to pass through 80 mesh (0.180 mm), hermetically packed in polyethylene bags and stored at 4 °C until use. These flours were recognized as extruded corn flour. From each of the 13 treatments, which were evaluated for total phenolic compounds, anthocyanins, antioxidant activity, and water solubility index.

2.4 Extraction of free phenolic compounds

The extraction of free phenolic compounds was carried out according to the methodology proposed by Adom & Liu (2002). The free phenolic compounds were extracted from 0.5 g of sample to which 10 mL of 80% ethanol (v/v) were added. The suspension was shaken in a Rotator (OVAN Noria R, USA) at 25 rpm for 10 min, and then centrifuged (3,000 *g*/10 °C/10 min). The supernatant was separated, placed in a conical tube, and concentrated (Apud Vac Concentrator, Thermo Elector Corporation) at 45 °C under low pressure until reaching a final volume of 2 mL. The extract was stored at -20 °C until use. The extraction was carried out in quadruplicate.

2.5 Extraction of bound phenolic compounds

The extraction of bound phenolic compounds was carried out according to the procedure of Adom & Liu (2002). The precipitate obtained in the extraction of free phenolics was digested with 10 mL of 2 M NaOH, oxygen was removed in the presence of N_2 gas and the sample was subjected to heat treatment (water bath at 95 °C/30 min), then stirred for 1 h at room temperature (25 °C). The mixture was neutralized with 2 mL of concentrated HCl, stirred for 2 min in a rotator (OVAN noria R, USA) with speed 25 rpm for 10 min, and immediately afterwards 10 mL of hexane were added to it to remove lipids. The solution was centrifuged (3,000 x *g*/10 °C/10 min) and after that the hexane was removed. The resulting mixture was extracted with 10 mL of ethyl acetate, stirred for 2 min (vortex) and stirred in a rotator (OVAN noria R, USA 2010) with speed 25 for 10 min and centrifuged (3,000 x *g*/10 °C/10 min) The ethyl acetate was removed and stored in a conical tube; this extraction was carried out 4 times. The ethyl acetate fraction was evaporated to dryness (Apud Vac Concentrator, Thermo Elector Corporation). The extracts were reconstituted with methanol and stored at -20 °C until use. The extraction was carried out in quadruplicate.

2.6 Extraction of anthocyanins

The anthocyanin extraction was developed by using the method described by Abdel-Aal & Hucl (1999). The anthocyanin extracts were prepared with 0.1 g of sample and acidified cold ethanol (95% methanol and 1 N HCl, 85:15, v/v). After that, the sample was centrifuged (Model 5415D; Eppendorf AG, Hamburg,

Table 1. Central composite design arrangement for the process optimization and main values for each response variable.

Process factors			Response variables			
Tr	T (°C)	SS (rpm)	TPC mg EGA/100 g	TA mg EC3G/100 g	AOX µmol ET/100 g	WSI %
1	68	78	297.97 ± 2.98	26.04 ± 0.38	591.77 ± 6.01	5.25 ± 0.12
2	152	78	268.06 ± 2.52	9.83 ± 0.21	551.03 ± 6.33	4.92 ± 0.27
3	68	212	289.72 ± 1.54	16.23 ± 0.01	552.90 ± 1.26	4.75 ± 0.32
4	152	212	312.24 ± 5.02	17.58 ± 0.54	602.98 ± 5.49	5.46 ± 0.34
5	50	145	330.53 ± 4.02	18.36 ± 0.20	661.62 ± 6.49	8.96 ± 0.32
6	170	145	311.91 ± 6.91	6.37 ± 0.11	586.90 ± 5.24	5.58 ± 0.33
7	110	50	278.94 ± 3.87	17.69 ± 0.69	553.60 ± 4.25	4.15 ± 0.17
8	110	240	273.27 ± 5.97	16.62 ± 0.01	536.39 ± 4.24	5.19 ± 0.24
9	110	145	276.06 ± 0.88	14.36 ± 0.33	561.30 ± 9.15	4.52 ± 0.29
10	110	145	275.04 ± 1.55	16.38 ± 0.21	567.53 ± 6.49	3.58 ± 0.16
11	110	145	284.09 ± 3.19	16.23 ± 0.02	556.66 ± 7.95	4.96 ± 0.09
12	110	145	286.85 ± 3.67	15.93 ± 0.12	565.87 ± 7.19	6.30 ± 0.09
13	110	145	289.72 ± 4.65	14.34 ± 0.28	571.78 ± 5.54	4.82 ± 0.10

Tr: treatment; T: extrusion temperature; SS: screw speed; TPC: total phenolic compounds; TA: total anthocyanins; AOX: antioxidant activity; WSI: water solubility index.

Germany) at 3000 × *g* for 10 min at 10 °C, and the supernatant was collected and stored at -20 °C until use. The extraction was carried out in quadruplicate under dark conditions to avoid anthocyanin degradation.

2.7 Determination of Total Phenolic Compounds (TPC)

Determination of total phenolic content samples was conducted according to the previous method (Singleton & Rossi, 1965). An aliquot (0.5 mL) of sample dissolved in 50% of methanol was mixed with 2.5 mL of Folin-Ciocalteu reagent and 2 mL of 7.5% Na_2CO_3, followed by incubation for 120 minutes at room temperature. After incubation the absorbance was recorded at 725 nm. The content of phenolic compounds was calculated using standard curve for gallic acid. Results were expressed as milligrams equivalents of gallic acid (EGA) per 100 g of sample (db). Total phenolic content in each sample was determined by the sum of the free and bound fractions. The determination was carried out in quadruplicate.

2.8 Determination of Total Anthocyanins (TA)

The TA was determined by using the method described by Abdel-Aal & Hucl (1999). The absorbance of the samples was measured immediately at 520 nm in a microplatereader (Model xMark TM; Bio-Rad, CA, USA). The anthocyanin content of samples was calculated using the following Equation 1:

$$Anthocyanins\ (mg/100\ g) = \left(\left(A * MW * DF * 100\right) / \varepsilon\right) * \left(V/m\right) * 1000 \quad (1)$$

Where A is the absorbance at a wavelength of 520 nm, MW is the molecular weight of cyanidin-3-glucoside (449.2 gmol-1), ε is the molar extinction coefficient (26,900 L mol-1 cm-1), V is the volume of the extract (L), DF is the dilution factor, and m is the weight of the sample (g). The results were expressed as mg equivalents of cyanidin-3-glucoside (EC3G) per 100 g of dry weight. The determination was carried out in quadruplicate.

2.9 Antioxidant activity (AOX)

The antiradical DPPH (2,2-Diphenyl-1-picrylhydrazyl) activity of the flour extracts was determined according to the procedure described by Shimamura et al. (2014) using the stable radical DPPH. The absorbance of the samples was measured at 517 nm. Trolox was used as a standard and the results expressed as µmol ET/100 g, db. The determination was carried out in quadruplicate.

2.10 Water Solubility Index (WSI)

The method described by Anderson et al. (1969) was used for the evaluations. 2.5 g of sample was suspended in 30 mL of water at 30 °C. The suspension was shaken on an orbital shaker at moderate speed (250 rpm/30 min) and subsequently centrifuged (3000 x g/30 °C/10 min). The supernatant was carefully decanted into a tared container for the determination of solids. WSI, expressed as percentage, was calculated from the weight of dry solids recovered by evaporation of the supernatant (110 °C/12 h). The WSI was determined in triplicate.

2.11 Experimental design and statistical analysis

To determine the effect of the extrusion process conditions and its optimization, the response surface methodology (RSM) was used, through the statistical package Design Expert, version 07. The ranges of the process conditions were selected according to the literature and adjusted by preliminary analysis. The scheme of the experimental design with the levels and process variables used are presented in Table 1. A rotating central composite experimental design was used with two factors [Extrusion temperature (T, 50-170 °C), screw speed (SS, 50-240 rpm)] in five levels, the design generated 13 treatments. The experiments were randomized to minimize systematic bias from external factors on the response variables. Regression analysis and ANOVA were performed for each of the response variables.

The response variables that were optimized in the extruded flours were TPC, TA, AOX and WSI, all of them in search of their maximum values. To find a solution that maximizes multiple responses, the objectives were combined into a general composite function called the desirability function. Then the numerical optimization found a point that maximized the desirability function. The validation of the nixtamalization extrusion process conditions estimated by the model was experimentally evaluated, for which the pigmented corn flour extrusion process was carried out under the optimized treatment conditions, subsequently the parameters were determined again TPC, TA, AOX, and WSI, and the values obtained were compared with the values predicted by the model.

3 Results and discussion

3.1 Checking the fitted model

Table 1 shows the different combinations of extrusion process factors (treatments) used to obtain the extruded flours and the average experimental values of the three repetitions carried out in the analysis of each of the response variables obtained for each combination. Table 2 showed the regression coefficients and the ANOVA results of the second order prediction model, showing the relationships between the response variables and the process factors (T and SS). The predictive models in terms of coded factors were presented in the Equations 2-5:

$$AOX = +564.63 + 20.21^{*} A^{*} B + 29.83^{*} A^{2} - 12.31^{*} B^{2} \quad (2)$$

(P − value of model < 0.0001; adjusted $R^2 = 0.8885$)

$$TPC = +280.32 + 5.61^{*} A^{*} B + 20.02^{*} A^{2} \quad (3)$$

(P − value of model < 0.0001; adjusted $R^2 = 0.8101$)

$$TA = +14.84 - 3.98^{*} A - 0.45^{*} B + 4.39^{*} A^{*} B + 1.63^{*} B^{2} \quad (4)$$

(P − value of model = 0.0004 adjusted $R^2 = 0.\ 0.8541$)

$$WSI = +4.84 + 1.90^{*} A + 0.29^{*} A^{*} B + 0.69^{*} A^{2} \quad (5)$$

(P − value of model < 0.0001; adjusted $R^2 = 0.9155$)

3.2 Effect of T and SS on Total Phenolic Compounds (TPC)

The values obtained for phenolic compounds were found in a range of 268.06-330.53 mg EAG/100 g, db (Table 1), the highest value (330.53 mg EAG/100 g, db) was at of T: 50 °C and SS: 145 rpm while the lowest values (268.06 mg EAG/100 g, db) were found in T: 152 °C and SS: 78 rpm. López & Baeza (2010) reported that the content of phenolic compounds in raw pigmented creole corn, found values of 465 mg acid gallic/100 g db., close with the results obtained in this investigation. In Figure 1a, it can be seen that, maintaining a low temperature of 50 °C and increasing the screw speed until reaching 240 rpm, a linear decreasing effect in the amount of phenolic compounds is observed. On the other hand, if a low screw speed (50 rpm) is maintained and the temperature is increased, it can be observed that the phenolic compounds decrease when they reach a temperature of approximately 110 °C. As they continue advancing and reach a temperature of 170 °C it is observed that there is an increase in phenolic compounds. Is observed of combining low temperatures with low screw speed, and high temperatures with high screw speed, higher values of phenolic compounds are obtained. This can be attributed to the release of phenolic compounds that are bound to the cell wall matrix. On the other hand, when using

Table 2. Values of calculated regression coefficients, ANOVA results of the second-order polynomial models, and the effects of the process factors on response variables.

Process factors	Response variables			
	TPC mg EGA/100 g	TA mg EC3G/100 g	AOX µmol ET/100 g	WSI %
Intercept				
β_0	280.32	14.84	564.63	4.84
Lineal				
$\beta_1(T)$	0	-3.98 (P = 0.0002)	0	1.90 (P < 0.0001)
$\beta_2(VT)$	0	-0.4464 (P = 0.4883)	0	0
Quadratic				
$\beta_{11}(T)^2$	20.02 (P < 0.0001)	1.63 (P = 0.0369)	29.83 (P < 0.0001)	0.6938 (P = 0.0012)
$\beta_{22}(VT)^2$	0	0	-12.32 (P = 0.0096)	0
Interaction				
$\beta_{12}(T*VT)$	5.61 (P = 0.1639)	4.39 (P = 0.0010)	20.21 (P = 0.0028)	0.2877 (P = 0.1654)
Model (F value)	26.59	18.56	32.86	33.51
Model (P-value)	P < 0.0001	P = 0.0004	P < 0.0001	P < 0.0001
R^2	0.8417	0.9027	0.9163	0.9437
Adjusted R^2	0.8101	0.8541	0.8885	0.9155
Lack of fit (P-value)	0.3593	0.0772	0.0878	0.5998
CV (%)	2.55	10.97	1.72	7.16

Note: P values in the parentheses denote the statistical significance to the terms of the quadratic regression models; model terms with P values ≤ 0.1 are significant, whereas model and lack of fit with P values ≤ 0.05 are significant. β_1: temperature; β_2: screw speed; TPC: total phenolic compounds; TA: total anthocyanins; AOX: antioxidant activity; WSI: water solubility index.

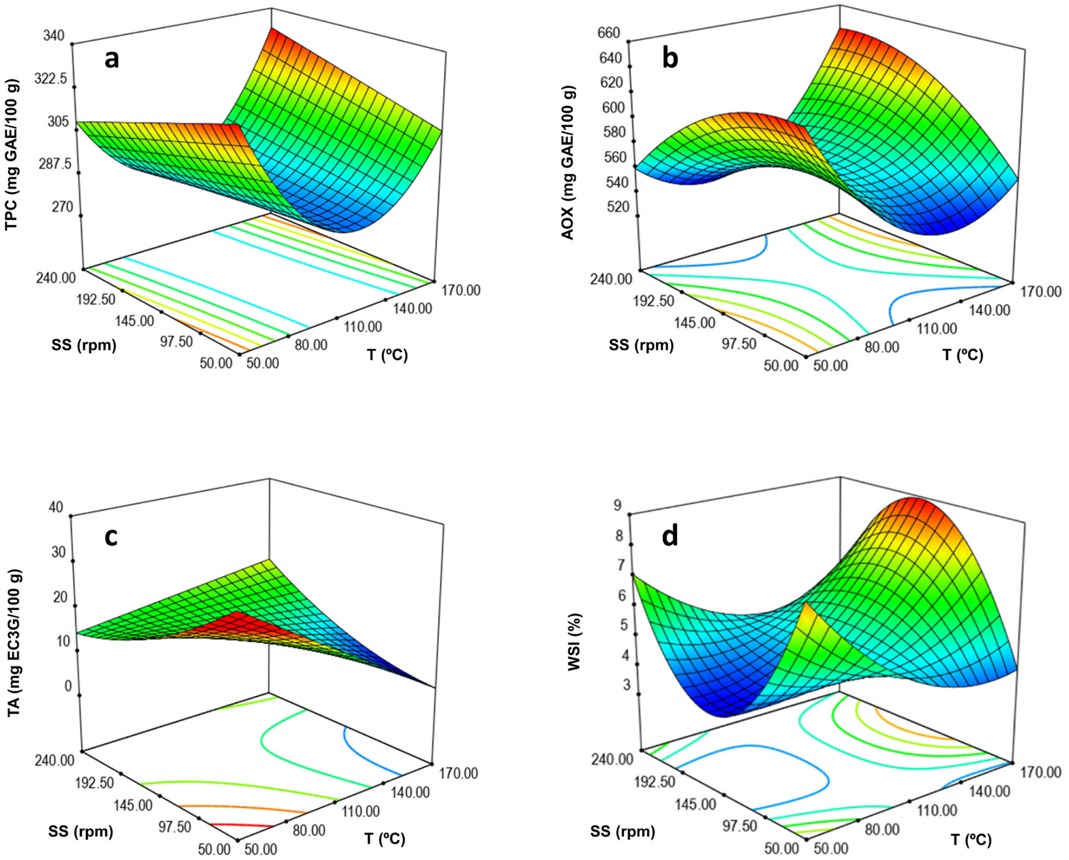

Figure 1. Effect of temperature (T) and screw speed (SS) on (a) TPC: total phenolic compounds, (b) TA: total anthocyanins, (c) AOX: antioxidant activity and (d) WSI: water solubility index.

high temperatures combined with a low screw speed, there would be a degradation of phenolic compounds since the corn would be exposed to a high temperature for a longer time.

Various studies have shown that the extrusion process causes a decrease in total phenolic compounds, total flavonoid compounds, total tannin content, in barley, whole and decorticated sorghum and shelled buckwheat seeds, but this occurs by a process of decomposition and polymerization of these compounds (Wang et al., 2013). The different opinions are due to the fact that the extrusion process behaves differently according to the matrices that the food has. In this case the state of the non-extractable polyphenols changed to extractable due to the hydrolysis of the fiber or protein residues linked to polyphenols. When performing the extrusion process of purple corn flour, it was observed that the extrusion process does not negatively affect phenolic compounds. This can be attributed to the correct combination of temperatures and screw speed, as well as the characteristics of the food matrix.

3.3 Effect of T and SS on Total Anthocyanins (TA)

The values obtained from TA were found in a range of 6.37-26.04 mg EC3G/100 g, db (Table 1), the highest value (26.04 mg EC3G/100 g, db) was found under the conditions of T: 68 °C and SS: 78 rpm while the lowest values (6.37 mg EC3G/100 g, db) were found at T: 170 °C and SS: 145 rpm. Our results are into the range reported by Menchaca-Armenta et al. (2020) that found a range of TA in extruded nixtamalized blue corn flours of 18.81 to 23.2 mg EC3G/100 g. Aguayo-Rojas et al. (2012), reported that raw blue corn contains 27.52 mg EC3G/100 g, db. Our results indicate that the extrusion method has a good antioxidant retention capacity since the maximum value found is about the range they obtained. In Figure 1b, it can be seen that, at T 50 °C and increasing SS until reaching 240 rpm, a linear effect is observed, decreasing TA to values of 15 mg EC3G/100 g, db. On the other hand, if a SS of 50 rpm is maintained and T is increased, it can be observed that the TA decreases to the point of greater degradation, this is mainly due to the sensitivity of this component to temperature where the heat causes them to degrade with the loss of glycosylating sugars with the consequent opening of the ring and production of colorless chalcones (Ruiz-Gutiérrez et al., 2015).

Another characteristic that influences the anthocyanin content in a food is the moisture. According with the literature, in a range of percentages of 18-27% moisture it is beneficial since it favors the lubrication of the process, but when the moisture exceeds the percentage of 27% this causes the starch to gelatinize and form a paste that causes the process to be slower and the product is exposed to temperature and heat for a longer time, mechanical shear and consequently degrading the anthocyanin content (Camacho-Hernández et al., 2014). A low moisture combined with a high screw speed is counter productive as this leads to a higher cutting speed and heat during the extrusion process and the anthocyanins are heat sensitive and this causes their degradation (Charunuch et al., 2011). It was observed that the extrusion process of purple corn conditioned to a moisture of 15%, can be adequate to preserve the TA content in the flour.

3.4 Effect of T and SS on antioxidant activity (AOX)

The values of antioxidant activity measured by DPPH method were found in a range of 536.39-661.62 μmol ET/100 g (Table 1), the highest value (661.62 μmol ET/100 g db) was found under the conditions of T: 50 °C and SS: 145 rpm while the lowest values (536.39 μmol ET/100 g db) were found in T: 110 °C and SS: 240 rpm. In Figure 1c, it can be seen that, maintaining a low temperature of 50 °C and increasing the screw speed until reaching 240 rpm shows a quadratic effect that decreases the AOX as the temperature increases. On the other hand, if a screw speed of 50 rpm is maintained and the temperature is increased until reaching 170 °C, a decrease in antioxidant capacity is observed with a quadratic effect. It is observed that the best AOX results are found in the regions of lower T and low SS and high T and high SS. In this case, the increase in antioxidant capacity is not so noticeable, but the advantage of this raw material is that we have a good retention capacity and, as several authors have mentioned, the response of the antioxidant capacity depends mainly on the composition of the food to be processed. Altan et al. (2009) point out that AOX of barley-based extrudates from fruit and vegetable by-products is related to the availability and complexity of phenolic compounds, so the extrusion process could cause changes that promote the availability of TPCs. In the extrusion process, all the parameters have an impact on the results of a final product, for which it is recommended that the moisture of the samples be kept low, since according with the literature, the greater presence of water can cause the shear effect to be more destructive of compounds with AOX. In this same sense, it is also important to avoid high screw speed because that causes a greater input of mechanical energy and in turn increases the shear effect (Ahmad & Kumar, 2018). Jan et al. (2017), observed in gluten-free extrudate from germinated chenopodium flour, that a high percentage of moisture also has a negative effect on the antioxidant capacity due to the polymerization effect, while high temperatures and screw speed provide a significant increase in antioxidant capacity and should be mainly to the process of destruction of cell walls in the hydrothermal process and the release of compounds with antiradical potential, such as ferulic acid. As results of this research, a good retention capacity of antioxidant capacity was obtained in Creole pigmented corn using moderately high temperatures, and low moisture (15%).

3.5 Effect of T and SS on the Water Solubility Index (WSI)

The WSI values were found in a range of 4.15-8.96% (Table 1), the highest value (8.96%) was found under the conditions of T: 50 °C and SS: 145 rpm while the lowest values (4.15%) were found in T: 110 °C and SS: 50 rm. In Figure 1d, it can be seen that, at a T of 50 °C and as SS increases, a quadratic effect is observed, where the WSI decreases to 3% until reaching 145 rpm, when continuing to increase the SS up to 240 rpm the WSI increases to 7%. At SS: 50 rpm, increasing T shows a quadratic effect where the WSI decreases up to 4%. At low T and SS, WSI can be observed around 6%. Valenzuela-Lagarda et al. (2017), when preparing snacks expanded by extrusion based on mixtures made up of squid, potato and corn, reported WSI values of 13.1-27.1%, indicating that structural changes occur during the extrusion process that increase capacity solubility of the food matrix. Becker et al. (2014), observed that when there is a degradation of macromolecules of amylase, it promotes the formation of molecules with lower molecular weight and converts it into amylopectin by breaking bonds, and consequently the product is more soluble in water. This indicates that corn flour is less soluble in water compared to other types of flours, such as extruded rice flour that has a percentage of water solubility that ranges from 20.79-34.81. WSI is also considered an indicator of the degradation of compound molecules and starch, it is highly related to the extrusion cooking that a food has. The water solubility index also allows to verify the severity caused by the extrusion process, which can cause degradation and solubilization of starch, granules, proteins and fibers present in the food (Trombini et al., 2016)

Kothakota et al. (2013), in a study on evaluation and characterization of extruded product by using various by-products point out that as there is a considerable increase in SS there is an increase in solubility, due to the high shear mechanics that is exerted on the product, also that high moisture can harm solubility since at the time of processing it causes gelatinization and plasticization of the product. Dlamini et al. (2007) point out that solubility is related to soluble molecules that are associated with the conversion of starch, if there is low degradation of starch there is less solubility. Subjecting a food to the extrusion process with the appropriate conditions of temperature, moisture and screw speed, we can obtain a nutritionally beneficial product since starch becomes easier to process to digest (Njoki & Faller, 2001).

3.6 Model optimization and verification

The response variables that were optimized in the extruded corn flour were TPC, TA, AOX, and WSI, all of them in search of their maximum values. A numerical optimization was carried out to select the optimal area with the best combination of processing conditions (T and SS) to obtain optimized extruded corn flours, where the maximum value of desirability (D) was obtained. The desirability value obtained during the optimization of the extruded corn flour was D = 0.9598 (Figure 2). Desirability values in the range of 0.7 to 1.0 provide a good and acceptable product according to a subjective scale reported by Carrera (1998). In Figure 2, the optimal region can be observed, finding values for the process variables of T (50 °C) and SS (50 rpm). In order to validate the efficiency of the prediction model, pigmented corn flour was extruded using these process conditions and the TPC, TA, AOX, and WSI parameters were again evaluated to contrast with those predicted (Table 3). The optimal extrusion conditions estimated TPC: 331.58 mg EGA/100 g, TA: 33.14 mg EC3G/100 g, AOX: 640.07 μmol ET/100 g, and WSI: 8.21%,

Table 3. Values predicted by the model versus experimental values obtained.

Response variable	Predicted value	Experimental value*	% adjusted
Total phenolic content (TPC) mg EGA/100 g	331.58	299.61 ± 3.69	90.35
Total anthocyanins (TA) mg EC3G/100 g	33.14	29.9 ± 1.25	90.22
Antioxidant activity (AOX) µmol ET/100 g	640.07	676.34 ± 17.02	105
Water solubility index (WSI) %	8.21	6.31 ± 0.17	76.85

*Mean ± standard deviation, n = 3.

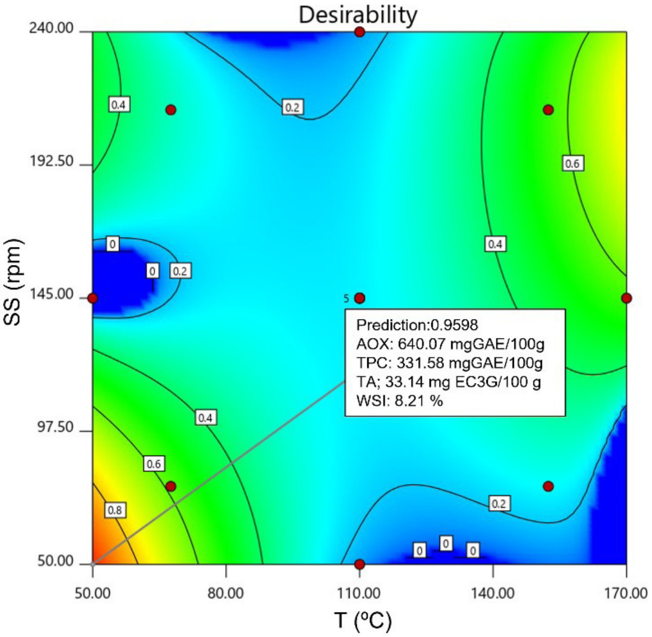

Figure 2. Contour plot of optimum conditions to produce extruded purple cornmeal.

while the optimized experimental conditions (50 ºC and 50 rpm) showed TPC: 299.61 ± 3.69 mg EGA/100 g, TA: 29.9 ± 1.25 mg EC3G/100 g, AOX: 676.34 ± 17.02 µmol ET/100 g, and WSI: 6.31 ± 0.17%. Indicating a 90.35, 90.22, 105, and 76.85% adjusted value from that of the model for TPC, TA, AOX, and WSI, respectively (Table 3). These results show that the optimization prediction model is adequate, thus allowing the prediction of the behavior of the response variables under different process conditions.

4 Conclusion

The adjusted of the values of TPC, TA, AOX and WSI obtained experimentally in corn flour with the predicted values for the model, show the response surface methodology was adequate for the evaluation and modeling of the process factors (T and SS). The process factors had a significant effect on all the evaluated parameters, T was the main factor that had a great effect on these parameters, mainly in their linear and quadratic term. The optimized nixtamalized extrusion processing conditions (temperature of the fourth zone of the extruder: 50 °C and screw speed: 50 rpm) allowed to generate a flour with the use of pigmented corn native to the state of Guerrero, with suitable nutraceutical and technological properties for developed new corn purple-based products. This could contribute to promote the protection, preservation and use of the corn biodiversity of this state, preserving its tradition and culture but also improving its commercial level.

Abbreviations

TPC: total phenolic compounds. TA: total anthocyanins. AOX: antioxidant activity. WSI: water solubility index. T: temperature. SS: screw speed. A: absorbance of anthocyanins at a wavelength of 520 nm. MW: molecular weight of cyanidin-3-glucoside. ε: molar extinction coefficient. V: volume of the extract. DF: dilution factor. m: weight of the sample. DPPH: 2,2-Diphenyl-1-picrylhydrazyl. RSM: response surface methodology. EAG: equivalents of gallic acid. EC3G: equivalents of cyanidin-3-glucoside. ET: equivalents of Trolox. D: desirability.

Conflict of interest

None.

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Author contributions

Leonor Bonilla-Vega: Investigation. Elias Hernández-Castro: Writing - Original Draft preparation. Roberto Gutiérrez-Dorado: Methodology-Formal analysis. Mirna Villamar-Vázquez: Review & Editing. Gregorio Sarabia-Ruiz: Validation. Jose Luis Valenzuela-Lagarda: Conceptualization, Supervision, Resources.

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