Snack of corn and roselle calyx residues: process optimization

Víctor Manuel Rivera-Castro[1]

Roberto Gutiérrez-Dorado[2]

Erick Paul Gutiérrez-Grijalva[1]

José Basilio Heredia[1]

María Dolores Muy-Rangel[1,§]

1 Centro de Investigación en Alimentación y Desarrollo AC-Coordinación Culiacán. Culiacán, Sinaloa, México. CP. 80015. (vrivera2019@gmail.com; erick.gutierrez@ciad.mx; jbheredia@ciad.mx).

2 Programa de Posgrado Integral en Biotecnología-Facultad de Ciencias Químico-Biológicas-Universidad Autónoma de Sinaloa. Culiacán, Sinaloa, México. CP. 80015. (rgutierrez@uas.edu.mx).

Autora para correspondencia: mdmuy@ciad.mx.

Abstract

Rich in fiber and bioactive compounds, roselle calyx decoction residues offer opportunities in food processing. This study sought to develop a directly expanded snack with a mixture of corn and roselle calyx decoction residues, optimizing its physical qualities. It was conducted in 2023, using the response surface methodology with three factors and three responses. The directly expanded snack showed expansion index values between 1.75 to 2.64, bulk density of 0.15 to 0.38 g cm^{-3} and firmness of 6.8 to 19.6 N, with attractive purple-lilac shades. The regression models were adequate and using the desirability method, optimal conditions (OT= 132.3 °C, SS= 240 rpm) and roselle inclusion level (J= 12.4%) were determined for an optimized directly expanded snack. It was concluded that the inclusion of up to 12.4% of roselle calyx allowed obtaining snacks with excellent physical characteristics.

Keywords:

Hibiscus sabdariffa L., Zea mays L., physical parameters, response surface methodology, snack.

Introduction

Mexico leads the production of roselle in the Americas, with the dried calyx of the fruit standing out in the production of concentrates and decoctions (SIAP, 2023). Roselle decoction residues (DRs), derived from extraction processes, contain up to 80% dietary fiber, offering significant potential (Sáyago-Ayerdi *et al.*, 2013).

An alternative to use roselle DRs is the production of directly expanded snacks (DESs), consumed as appetizers. These are gaining popularity due to their ease of consumption, produced through an expansion process that involves low moisture, heat, pressure, and protein-carbohydrate interactions; extrusion is the most widely used method (Félix-Medina *et al.*, 2020).

The snack market is constantly growing, reaching a global value of 538 billion dollars in 2022, with Mexico contributing 11.96 billion dollars and per capita consumption of more than 13 kg (Statista, 2023); therefore, it is attractive to produce products with these characteristics.

The consumption of DESs is associated with products rich in carbohydrates and fats, with low nutritional density and digestive problems due to the lack of fiber and a high glycemic index (>70) (Calvo-López and Martínez-Bustos, 2017). The growth in production and demand for healthy options has driven the search for ingredients that increase nutritional value, with fiber standing out (Gopirajah and Muthukumarappan, 2017).

Key parameters for DES acceptance include expansion index, bulk density, and firmness, all of which determine sensory quality. Expansion is crucial because of its effects on bubble structure and rupture; density influences texture, seeking a lower density, and firmness, related to shear force, decreases with increasing bubbles (Moraru and Kokini, 2003; Meng *et al.*, 2010).

In commercial DES production, corn starch is common but lacks nutritional input. For this reason, the use of whole meal cereal flours, such as purple corn, rich in polyphenols, proteins, and fiber, is being studied (Urias-Lugo *et al.*, 2014). Although DESs with whole meal flours show less expansion, adjusting extrusion conditions is essential (Han *et al.*, 2018). Optimization using the response surface methodology presents itself as the most effective strategy.

This study seeks to evaluate the incorporation of roselle DR flour into purple corn flour. Using the response surface methodology, it is sought to determine the optimal temperature and screw speed conditions of the extruder, as well as the optimal level of roselle inclusion to achieve DES with ideal physical characteristics.

Materials and methods

Plant material

Purple corn kernels of the olotillo variety (17° 07' 49.3" north latitude, 99° 07' 18.15" west longitude) and roselle calyces of the landrace variety (16° 58' 46.2" north latitude, 99° 06' 53.9" west longitude), collected in the municipality of Ayutla de los Libres, Guerrero, were used. The roselle calyces were prepared as mentioned by Mayo-Mayo *et al.* (2020), the decoction calyces were dehydrated at 40 °C per 48 h, the dried calyces were ground in a model F20342 coffee grinder (KRUPS North America Inc.) to a particle size ≤ 0.38 mm.

In the case of the corn kernels, they were cleaned manually by removing grains in poor condition and garbage, then they were ground in a model 1000A electric seed mill (LEJIEYIN, China) to a particle size ≤0.38 mm. Both samples were stored in refrigeration (4 °C) until use. Prior to the extrusion process, the corn-roselle mixtures were adjusted to a moisture of 15%.

Extrusion conditions

The directly expanded snacks (DESs) were prepared by using a model 20DN laboratory single screw extruder (CW Brabender Instruments, Inc. New Jersey, USA), which was equipped with a

19 mm diameter screw, 20:1 length, 3:1 screw compression ratio, 3 mm outlet die and a feed flow of 30 kg h.

The equipment has three heating zones, adjusted with a gradient of 20 °C between each section. Table 1 shows the different outlet temperatures (OT) of the extruder, as well as the screw speeds (SS) and roselle calyx inclusion content (J), which were used to obtain the different DESs according to the experimental design. Subsequently, the DESs were cut into 5 cm pieces and placed in a forced-air oven at 60 °C per 30 min.

Table 1. Experimental design and results of the response variables analyzed to the directly expanded extrusion snacks.

Run[*]	Process variables						Response variables		
	Coded values			Uncoded values					
	X1	X2	X3	OT	SS	J	EI	BD	F
1	-1	-1	-1	130	104	3.43	2.21	0.33	19.62
2	1	-1	-1	160	104	3.43	2.28	0.17	11.56
3	-1	1	-1	130	206	3.43	2.64	0.15	10.35
4	1	1	-1	160	206	3.43	2.49	0.21	13.27
5	-1	-1	1	130	104	12.06	2.04	0.38	17.35
6	1	-1	1	160	104	12.06	1.75	0.23	6.81
7	-1	1	1	130	206	12.06	2.08	0.21	8.99
8	1	1	1	160	206	12.06	2.02	0.22	7.29
9	-1.682	0	0	120	155	7.75	2.2	0.25	16.73
10	1.682	0	0	170	155	7.75	1.89	0.18	7.45
11	0	-1.682	0	145	70	7.75	1.99	0.3	16.27
12	0	1.682	0	145	240	7.75	2.27	0.21	11.38
13	0	0	-1.682	145	155	0.5	2.52	0.18	12.7
14	0	0	1.682	145	155	15	1.83	0.24	8.66
15	0	0	0	145	155	7.75	2.25	0.23	14.68
16	0	0	0	145	155	7.75	2.41	0.22	14.04
17	0	0	0	145	155	7.75	2.33	0.23	14.6
18	0	0	0	145	155	7.75	2.33	0.19	13.48
19	0	0	0	145	155	7.75	2.33	0.21	14.6
20	0	0	0	145	155	7.75	2.32	0.24	14.77

[*] = Does not correspond to the order of the run; OT= outlet temperature (°C); SS= screw speed (rpm); J= level of inclusion of roselle in snacks (%, w/w); EI= expansion index; BD= bulk density (g cm^{-3}); F= firmness (N).

Expansion index (EI) and bulk density (BD)

The DESs (10 pieces) had the length (cm), the average diameter (cm) of three sections (points and center) and the weight (g) measured. The EI was calculated with equation 1 and the BD with equation 2 (Espinoza-Moreno *et al.*, 2016):

$$EI = \frac{\text{Average diameter of the DES}}{\text{outlet diameter}}$$

equation 1.

$$BD\left(g\,cm^{-3}\right) = \frac{\text{DES weight}}{\pi\left(\text{DES radius}\right)^2 \text{DES length}}$$

equation 2.

Firmness

Firmness (Newtons) was quantified by using 50 DESs, a Model TA1 texture meter (AMETEK, Knightdale, North Carolina, USA), a 500 N cell and a cutting tip of 2 mm in diameter, penetration speed of 10 mm min^{-1} up to 60% of the diameter of the snack (Anton and Luciano, 2007).

Color

Three measurements were made on the DESs (10 per treatment) on a white background, using a model CM700d colorimeter (Konica Minolta, USA). Luminosity (L), chromaticity (C) and hue or true color angle (°H) values were reported.

Response surface methodology and optimization

A response surface experimental design was employed, with three factors: outlet temperature (OT), screw speed (SS), and percentage of inclusion of roselle in the mixture (J). The Design Expert 13 program was used, which generated 20 experimental runs (Table 1). Process variables included OT ranging from 120 to 170 °C and SS from 70 to 240 rpm, while J ranged from 0.5 to 15%. The responses evaluated were the expansion index (EI), bulk density (BD), and firmness (F) of the DESs.

A composite central rotatable experimental design was used, with 20 experiments performed in random order. Treatments were performed only once, excluding mean and standard deviation (Espinoza-Moreno *et al.*, 2016). Statistical analysis included least squares regression and analysis of variance to fit second-order regression models to the experimental data of the response variables.

For the optimization process, the numerical method of desirability of the RSM was used, trying to find a desirability (D) greater than 0.7, it was sought to maximize the variables J and EI, and minimize BD and F. Additionally, surface and contour graphs were generated to analyze the effect of the extrusion process variables and the level of inclusion of roselle on the physical properties (EI, BD, F) of the DESs.

Results and discussion

Response variable modeling

Table 1 shows the experimental results of the response variables studied. The DESs showed values of EI between 1.75 and 2.64, BD between 0.15 and 0.38 g cm^{-3}, and F between 6.81 and 19.62 N. Despite the challenges noted by Han *et al.* (2018) on the use of fiber-rich sources, such as roselle calyces DRs, in the making of DES, our results showed high experimental values of expansion and low density and firmness. In contrast to concerns about competition between fiber and starch for water, our DESs demonstrated positive results in bubble formation and final properties.

The statistical results of the analyses of regression and variance of the data of the response variables BD, EI, and F, are presented as predictive models in coded factors (equations 3-5). All regression models were significant with p-values ≤ 0.0001. In general, it was observed that these regression models presented an R^2_{adj} of 82 to 97%.

EI=-4.068+0.093(OT)+0.002(SS)-0.049(J)-0.0003(OT)^2 equation 3, (p-value of the model <0.0001; R^2_{adj}= 0.822). BD=1.976-0.012(OT)-0.010(SS)+0.005(J)+0.00006(OT*SS) equation 4, (p-value of the model <0.0001; R^2_{adj}= 0.859).

F=22.615+0.504(OT)-0.513(SS)+2.743(J)+0.003(OT*SS)-0.014(OT*J)-0.004(OT)^2-0.071(J)2 equation 5, (p-value of the model <0.0001; R^2_{adj}= 0.9633).

The regression models did not show a lack of fit (p> 0.05), and in all the responses of the DESs, the linear terms were significant (p> 0.1). EI and F showed significance in the quadratic terms (OT*OTand J*J). In addition, significant interactions were observed in BD and F (OT*SS and OT*J only for F).

Response variable analysis

Expansion index (EI)

As EI is one of the key attributes, these values are expected to be greater than 1.5 (Huber, 2001). Figure 1A shows the behavior of EI. Where the values are higher at OT between 130 and 145 °C, with a J close to 0.5%. This phenomenon was attributed to the content of roselle, which provides more fiber, inhibiting expansion due to ruptures in cell walls and lower starch content (Han *et al.*, 2018).

Figure 1. 3D response surface graphs of the different response variables studied on the DESs as a function of the variables OT and J, at a constant SS of 155 rpm.
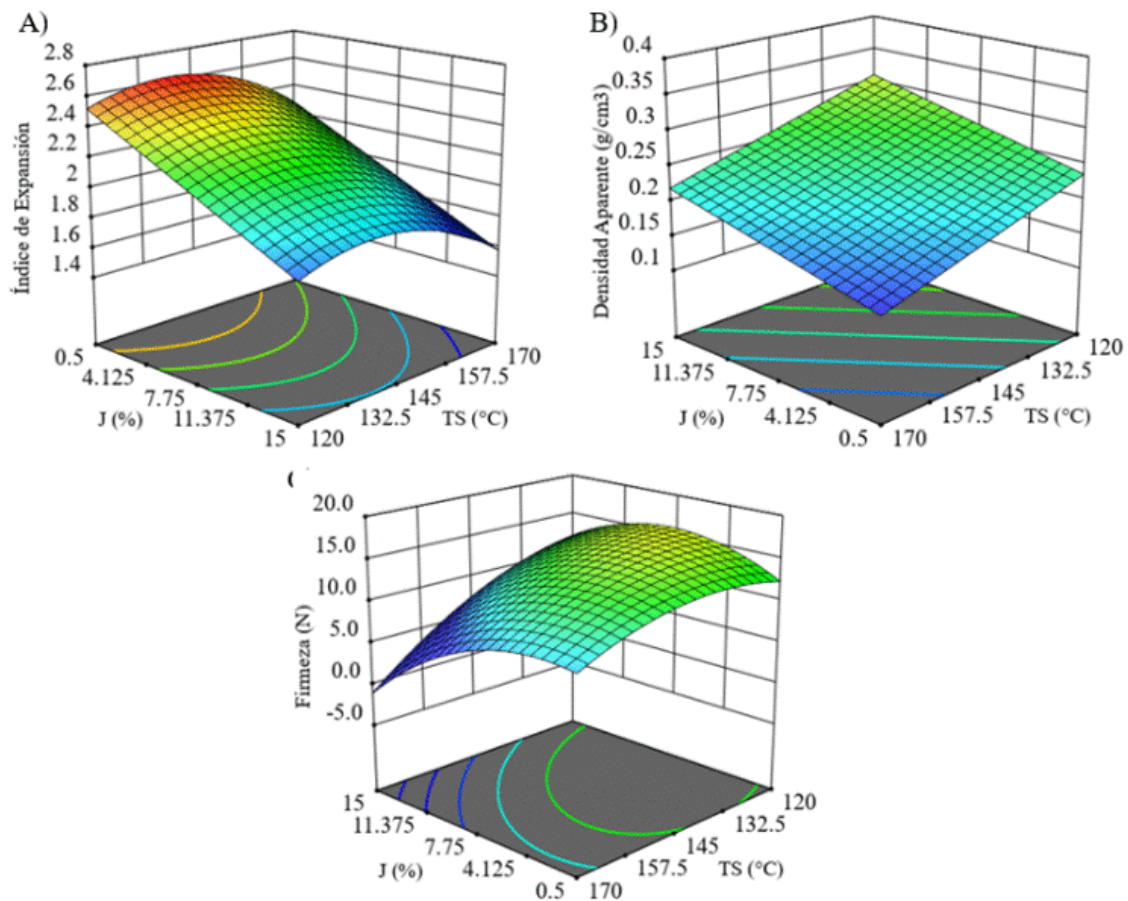
The study by Huber (2001) points out that with a higher SS, greater pressure is generated in the barrel and die, which creates a pressure differential that favors the expansion of the snack, regardless of the composition of the food matrix. The low moisture in the corn-roselle mixture favored moisture distribution in the starch granules and improved the viscoelastic and shear properties of the mixture (Anderson, 1969).

Results similar to this study are supported by Rivera-Mirón *et al.* (2020), who evaluated the inclusion of pineapple pulp in a corn DES, where they observed that, the higher the pineapple pulp content, the lower the EI due to the fiber content. With conditions of OT of 150 °C, SS of 200 rpm and 15% of pineapple pulp, they obtained the highest expansion (1.86), lower than the maximum reported in this study (2.64).

Bulk density (BD)

BD is inversely proportional to EI, it reflects the degree of expansion of the snacks. During expansion, the rapid evaporation of water generates large, stable bubbles, thus reducing the bulk density in the products (Huber, 2001).

The graphical representation of the model for BD, Figure 1B, shows the effect of OT and J, with high values of BD at SS of 155 rpm, with low OT and high content of roselle (J) standing out. O'Shea *et al.* (2013) mention that the high temperatures and pressures in the barrel of the equipment facilitate rapid expansion as the product exits the die, this was corroborated by the results of the model and (Figure 2).

Figure 2. Graph showing the overall desirability (D), optimal conditions of the extrusion process, and the optimal content of roselle in the DES.
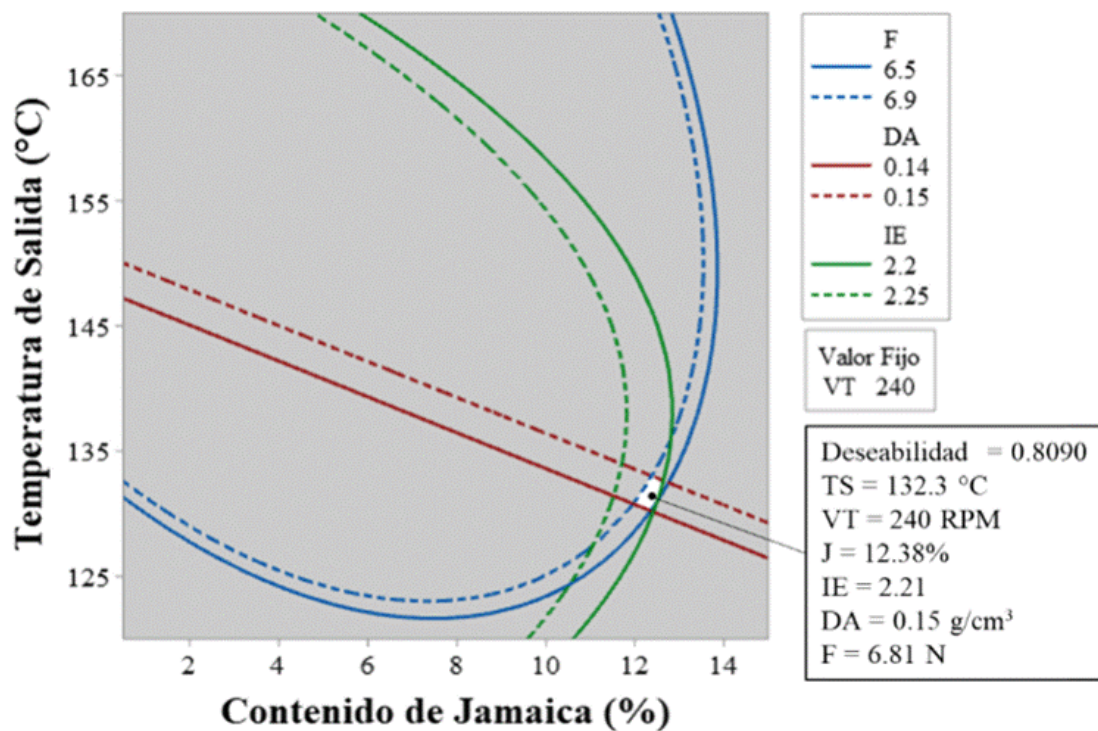
Firmness (shear force)
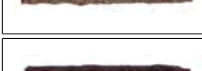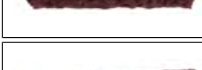
Figure 1C, at SS of 155 rpm, shows an interaction between the process variables OT and J. Low values of OT and J result in high values of F, while high levels of both indicate low firmness in the DESs. This indicates that higher percentages of roselle favor the decrease in firmness. This phenomenon may be due to the fiber content provided by roselle, allowing greater moisture retention, and improving the interactions between the components (Han *et al*., 2018). Chinellato *et*

al. (2016) indicated that the addition of roselle calyx powder reduced the hardness of tapioca DESs. In this study, an inverse relationship between F and J was observed (Table 1).

Color of the snacks

Table 2 presents the results of snack color. In general, snacks exhibited an L between 31.9-52.5, variable C between 9.7-16.9 and °H ranging from 13.0-47.9. According to the color circle of Minolta (1994), the color values of snacks are in the red-blue range, characteristic of their natural pigments. The lack of an overall pattern in the color variables may be due to the phenol richness of both mixtures, which could undergo similar changes, especially due to the extruder outlet temperature. The variability of color between snacks can be attributed to the variable effect of the light beam, influenced by their morphological characteristics due to expansion (Cid-Ortega and Guerrero-Beltrán, 2015).

Table 2. Color coordinates, diameter, and pictures of directly expanded snacks (DESs) prepared according to the runs of the RSM experimental design.

Run	Coordinates			Diameter (mm)	DES
	Luminosity (L)	Chromaticity (C)	°Hue (°H)		
1	37.3 ±4.8	12.8 ±2.2	26.3 ±5.5	5.76	
2	46.1 ±3.5	11.1 ±1	33.3 ±5.7	6.83	
3	43.1 ±4.4	11.2 ±0.9	28 ± .3	7.91	
4	41.9 ±3.5	12.2 ±1.7	24.9 ±9.5	7.47	
5	31.4 ±4.9	10.2 ±2.1	22.4 ±6.3	6.13	
6	48.3 ±4.7	12.3 ±2.1	32.8 ±8.2	5.25	
7	38.6 ±2.2	17 ±1.2	13±1	6.23	
8	47.2 ±2.6	13.2 ±0.6	19.3 ±4.8	6.06	
9	40.4 ±4.3	13.8 ±1.5	18.1 ±2.4	6.6	
10	50.7 ±1.9	12.2 ±0.9	40.6 ±5.7	5.67	
11	42.0 ± 2.9	13.4 ±1.2	26.4 ±3.7	5.96	
12	39.4 ±2.9	13.2 ±0.8	24.5 ±2.5	6.8	
13	52.5 ±2.9	9.7 ±1	44.6 ±7.8	7.57	

Run	Coordinates			Diameter (mm)	DES
	Luminosity (L)	Chromaticity (C)	°Hue (°H)		
14	37.2 ±2.5	15.3 ±1.5	18.5 ±1.3	5.5	
15	39.8 ±3.6	13.3 ±1.2	24.8 ±3.6	6.74	
16	45.4 ±2.6	13.3 ±0.7	20.2 ±2.2	7.24	
17	45.3 ±2.6	13.5 ±0.7	20.3 ±2.6	6.4	
18	41.4 ±2	14.2 ±1.4	19 ±2.5	7	
19	40 ±3.3	12.7 ±1	27 ±2.6	6.98	
20	38.6 ±4.4	13.9 ±1.4	21.3 ±5.8	9.65	

The findings of Mayo-Mayo *et al*. (2020) incorporated roselle calyx into corn chips, obtaining a hue angle of 10, indicating colors close to purple, highlighting their sensory attributes and general acceptance thanks to the distinctive color of roselle, these authors mention that the current demand for natural foods and vibrant colors has increased, with fruits and vegetables rich in intense pigments, such as roselle, standing out.

Optimization

In this research, optimization focused on maximizing EI and J, and minimizing BD and F of DESs. The overall desirability (D) reached 0.809 (Figure 2), considered good for food, since values above 0.7 are classified as acceptable by consumers (Fabila-Carrera, 1998). In Figure 2, a white area shows the optimal process conditions (OT= 132.3 °C, SS= 240 rpm, and J= 12.38%) to obtain a DES with an EI= 2.21, BD= 0.15 g cm^{-3} and firmness= 6.81 N.

The study by Rivera-Mirón *et al.* (2020) reported a D of 0.739, for OT conditions of 165 °C, 15% pineapple pulp in corn flour DESs adjusted to 23.04% moisture and a SS of 200 rpm, the DESs presented an EI of 1.53 and BD of 4.01 g cm^{-3}.

On the other hand, Pensamiento-Niño *et al.* (2018) evaluated the addition of mango pulp in a cassava starch snack, obtaining optimal values for OT= 135.81 °C, 7.97% mango pulp, moisture of 18.84%, and SS of 100 rpm. With a D of 0.772, they achieved a DES with an EI of 1.52 and BD of 0.66 g cm^{-3}, it can be observed that in both cases and in this study, it was feasible to achieve DESs with at least the minimum EI values required for expanded foods (Huber, 2001).

Conclusions

This study focused on finding the optimal conditions for the production of directly expanded snacks (DESs) of purple corn with roselle calyx DR flour. Ideal conditions were OT of 132.3 °C, SS of 240 rpm and inclusion level of roselle calyx DR of 12.4%, with an overall desirability greater than 0.8. EI, BD, and F values of the DESs of 2.21, 0.15 g cm^{-3} and 6.81 N, respectively, were predicted, presenting an attractive purple-lilac color.

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