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Optimization of Extrusion Conditions for an Extruded Food Enriched with Mango By-Products (*Mangifera indica* var. Tommy Atkins) via Response Surface Methodology

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Abstract: Today, mango by-products are produced in significant amounts, posing an environmental problem. Their incorporation into food products offers a solution to this problem. An extruded food product formulated with the flours of white corn, mango peel, and mango kernel was developed. To obtain the desired quality regarding the physical and chemical characteristics of the extruded food product, an optimization study was carried out. Response surface methodology was used to evaluate the effect of the following extrusion process variables on the physical (expansion index (EI), hardness, water absorption index (WAI), water solubility index (WSI)) and chemical properties (total polyphenol content and antioxidant capacity): the die temperature (DT, 100–130 °C), the feed moisture content (FMC, 17–21%), and the screw speed (SS, 80–120 rpm). Response surface and regression models were performed to determine the responses as a function of the process variables. Model optimization was carried out with an R^2 of >0.60, maximizing the WAI and minimizing the hardness and the WSI. The optimal conditions were a DT of 120.66 °C, an FMC of 21.88%, and an SS of 66.36 rpm. The extruded product's characteristics were an EI of 1.10, a hardness of 63.66 N, a WAI of 5.41 g/g, a WSI of 16.20%, a TPC of 3402 mg GAE/100 g sample, and an antioxidant capacity of 90.09 mg Etrolox/g (measured by DPPH) and 79.38 mg Etrolox/g (measured by ABTS); the overall desirability value was 0.870.

Keywords: mango by-products; extrusion cooking; physical properties; chemical properties; optimization with response surface methodology; mango peel and kernel

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1. Introduction

Mango is a tropical fruit that belongs to the Anacardiaceae family. Its functionality is attributed to its high content of antioxidants (phenolic compounds, carotenoids, and ascorbic acid), nutritional composition (fiber, minerals, and vitamins), and sensory attributes (sweet flavor and attractive color) [1]. In Mexico, excess mango production at certain times of the year causes post-harvest losses of almost 40%; this is due to poor agricultural practices, damage to crops, sensory aspects that are related to organoleptic characteristics, as well as the lack of marketing by small producers [2–4]. The economic value of this

fruit is reduced because it has a short maturation period [5], which is why it is generally consumed fresh, or the pulp is utilized for processed products (juices, concentrates, and jams). Depending on the variety of the fruit and the process used, the amount of mango by-products generated varies and can be as much as to up to 60% of the total weight of the fruit, of which 5–10% is the pulp bagasse, 15–20% is the kernel seed, and 15–20% is the peel [1,6]. Throughout the world, the mango industry produces more than 20 million tons of by-products each year—this creates a harmful effect on the environment due to the lack of places to dispose of them [7]. Currently, there is no legislation for the use of mango by-products in food. However, Regulation (EU) No. 68/2013 of the European Union allows the use of by-products obtained from processing fresh fruits and vegetables as feed materials [8]. The valorization of new raw materials obtained from by-products is a way to achieve proper management. This way, the ecological impact is reduced, and production is as profitable as possible, promoting a circular economy [1].

Food processing at high temperatures causes nutrient losses [9]. To avoid this, the extrusion process can be used as an alternative, which allows mango by-products to be transformed into value-added products since the processing time is shorter compared with conventional cooking times, resulting in lower nutrient losses [5]. Due to the versatility of the process, unconventional and perishable raw materials can be incorporated. Extrusion is a continuous cooking and molding process involving short durations, high temperatures, and the combination of moisture, pressure, screw speed, residence time, temperature, and mechanical shearing, among others, to cook foods rich in starch [10]. These factors cause the fed raw material to undergo molecular transformations and chemical reactions, such as the formation of complexes between amylose and lipids, starch gelatinization, degradation reactions of vitamins and pigments, and protein denaturation [11]. Extrusion processing is used for the manufacturing of breakfast cereals, pasta, textured vegetable protein, expanded snacks, and baby food [12]. Currently, there is a need to create foods with better sensory and nutritional properties, such as maintaining color, enriching nutrients, conserving phenolic compounds, and increasing dietary fiber [13,14].

Mango peel and kernels have been employed to develop new nutritionally balanced and healthier mango products (noodles, bread, biscuits, cookies, and other bakery products). The addition of these by-products increases the carotenoid content and antioxidant capacity and improves the amount of dietary fiber, providing added value [1,6]. Mazlan et al. [6] evaluated the effect of the expansion index and hardness on an extrudate developed with corn grits and mango peel flour (MPF) (8.34% to 25%), while Korked et al. [15] developed an extrudate with the addition of 5.45% mango peel fiber. Both studied how to optimize extrusion conditions (DT, FMC, and SS); however, to our knowledge, mango kernels have not been used in extrusion processes. Gupta et al. [16] indicated that more research is required to increase the acceptance and quality of mango-kernel-derived products. Thus, the development of an extruded food product containing mango peel and mango kernel flours is an option to valorize the mango by-products that are currently discarded and to reduce that quantity. It could also be an opportunity to develop the economy of some mango-producing regions.

In a previous investigation, our research group [17] found that the best formulation for an extrudate contained white corn flour and the addition of mango by-products. However, this work was carried out under constant extrusion conditions, so it was not possible to assess how the temperature, moisture, and screw speed affect the extrudates. Because it is essential to identify the optimal processing conditions to obtain a high-quality and marketable product [12,18], the aim of this research was to (a) evaluate the effect of the extrusion process variables (screw speed, die temperature, and feed moisture content) on the physical and chemical properties of the extrudates and (b) to optimize the extrusion conditions through the use of response surface methodology.

2. Materials and Methods

2.1. Raw Materials

Mangoes *(Mangifera indica)* of the Tommy Atkins variety and white corn *(Zea mays)* were acquired from local markets in Guadalajara, Jalisco (Mexico). The chemical reagents used in this research were of analytical grade.

2.2. Preparation of Flour

Mango peel and kernels were dried at 60 °C in a RedLINE convection oven (Binder, Tuttlingen, BW, Germany) [9] until a moisture content lower than 10% was reached. Then, the mango kernel and peel and the corn were ground to obtain powders with average particle sizes of 0.40 mm, 0.50 mm, and 0.60 mm, respectively. An optimized mixture of 500 g of flour was prepared with 8.34%, 33.33%, and 58.33% mango kernel flour (MKF), mango peel flour (MPF), and white corn flour (WCF), respectively [17]. The flours were hydrated to different humidity levels (17–21%) through spraying them with water using a spray bottle and mixing them for ten minutes at medium speed in a KP26M1XER commercial mixer (KitchenAid, MI, USA). Finally, they were stored overnight (12–14 h) to ensure homogeneity before processing. The following bromatological flour analyses were conducted according to Mexican regulations: crude protein (NMX-F-608-NORMEX-2011) [19]; moisture (NMX-F-083-1986) [20]; fats (ethereal extract) (NOM-086-SSA1-1994) [21]; crude fiber (NMX-F-613-NORMEX-2003) [22]; ash (NMX-F-607-NORMEX-2013) [23]; carbohydrates (determination by difference (Method 986.25) A.O.A.C. Volume l. 1990) [24].

2.3. Extrusion Conditions

A Brabender single-screw Intelli-Torque extruder (South Hackensack, NJ, USA) was used that had a compression ratio of 3:1, a barrel diameter of 19.05 mm, and a 20:1 L/D ratio. A circular die of 3 mm and 20 mm in diameter and length, respectively, was used. The extruder was conditioned with a pressure and temperature sensor with three heating zones (feed, melting, and die zone). The temperature profile was established with a change in temperature between zones of 10 °C, with the die temperature (DT) being the highest temperature along the barrel. Extrusion process variables (screw speed (SS), moisture content (FMC), and DT) were established based on 17 experimental runs (Table 1). Then, extrudate products were dried at 60 °C in a RedLINE convection oven (Binder, Tuttlingen, BW, Germany) until their moisture content was 5% and stored for further analysis [13].

2.4. Physical Properties

2.4.1. Expansion Index (EI)

The diameter of the extrudate was measured with a Vernier Caliper (Mitutoyo Co., Kawasaki, JPN) and divided by the internal diameter of the extruder die. Ten measurements were taken per extruded sample [6].

2.4.2. Hardness

Based on adjustment assays and according to the equipment specifications, extrudates were punctured with a compression cylinder probe (2 mm radius) at a penetration of 20%, a speed of 5 mm/s, and a distance of 10 mm using a Stable Micro System TA.XT plus texture analyzer (Godalming, SY, UK). The hardness was expressed as the maximum force used to break the sample. Ten measurements were conducted per sample [25].

2.4.3. Water Solubility Index (WSI) and Water Absorption Index (WAI)

As mentioned in Raleng et al. [26], the WSI and WAI were determined in triplicate, with slight modifications. In a 50 mL tube, a mixture of 25 mL distilled water and 2.5 g of the sample was shaken using a Genie Scientific Industries SI-1100 Roto-shake (Bohemia, NY, USA) at room temperature for 30 min. In order to separate the suspension, a Luzeren Universal TDL-40B centrifuge (Tlajomulco de Zúñiga, JAL, Mexico) was used for 10 min at 2770× *g*. Finally, the supernatant was dried to constant weight at a temperature of 105 °C in

a RedLINE convection oven (Binder, Tuttlingen, BW, Germany). The WSI (as a percentage) was expressed as grams of dissolved solid in the supernatant per gram of dry solid, and the WAI was expressed as grams of gel gained per gram of dry solid.

Table 1. Experimental design for the extrusion process.

Run	Coded Level			Actual Level		
	DT (°C)	FMC (%)	SS (rpm)	DT (°C)	FMC (%)	SS (rpm)
1	0	0	0	115	19	100
2	−1	−1	−1	100	17	80
3	1	−1	−1	130	17	80
4	−1	1	−1	100	21	80
5	1	1	−1	130	21	80
6	−1	−1	1	100	17	120
7	1	−1	1	130	17	120
8	−1	1	1	100	21	120
9	0	0	0	115	19	100
10	1	1	1	130	21	120
11	−1.68179	0	0	90 [a]	19	100
12	1.68179	0	0	140 [a]	19	100
13	0	−1.68179	0	115	15.5 [a]	100
14	0	1.68179	0	115	22.5 [a]	100
15	0	0	−1.68179	115	19	66 [a]
16	0	0	1.68179	115	19	134 [a]
17	0	0	0	115	19	100

DT: die temperature; FMC: feed moisture content; SS: screw speed. [a] Values were rounded up to the nearest whole number to set extrusion conditions.

2.5. Chemical Properties

2.5.1. Extraction Method

Extracts from each extrudate were prepared via a method adapted from Bandyopadhyay et al. [27]. One gram of ground sample (<250 µm) was extracted for the first time with 20 mL of methanol/water (50:50 v/v). The mixture was shaken in a Genie Scientific Industries SI-1100 Roto-shake (Bohemia, NY, USA) at room temperature for 1 h. Then, it was centrifuged (Labogene, Lynge, ALL, Denmark) at 2770× g for 15 min. The supernatant was collected in an amber container. After that, a second extraction was performed as described above, adding 20 mL of acetone/water (70:30 v/v) to the residue. Finally, both supernatants were mixed and stored at 4 °C. This extract was used to determine the total phenolic and antioxidant capacity using ABTS and DPPH.

2.5.2. Total Polyphenol Content (TPC)

According to the method developed by Basilio-Atencio [28], with slight modifications, the total polyphenol content was measured using 2N Folin–Ciocalteu reagent (diluted with water, 1:10 v/v). First, 150 µL of Folin–Ciocalteu reagent and 30 µL of extract were added to a 96-well microplate and then mixed with 120 µL of Na_2CO_3 (20% w/v). The samples were kept in the dark for 1 h. Finally, the absorbance was measured with a ThermoFisher Scientific spectrophotometer (Multiskan GO, Waltham, MA, USA) at 760 nm. The results were reported as mg gallic acid equivalents (GAE) per 100 g sample.

2.5.3. Antioxidant Capacity Assay

DPPH Radical Scavenging Capacity

DPPH (2,2-Diphenyl-1-picrylhydrazyl) was measured with the method developed by Polat et al. [29], with slight modifications. Amounts of 200 µL of DPPH (500 µmol, with methanol) and 20 µL of sample extract were placed in a 96-well microplate. Then, the samples were kept in the dark for 30 min. The absorbance of the sample was read at 515 nm

using a spectrophotometer (Multiskan GO, Waltham, MA, USA). The results were reported as mg Trolox equivalents (Etrolox) per g sample. Methanol was used as a blank.

ABTS Radical Scavenging Capacity

An ABTS radical cation assay was performed according to Lizárraga-Velázquez et al. [30], with slight modifications. A prepared solution of 7 mM ABTS was reacted with 10 mL of distilled water and 2.5 mM of $K_2S_2O_8$ at room temperature and in darkness for 16 h under constant stirring. Methanol was used to adjust the absorbance of the ABTS solution to 0.7 ± 0.02 with a spectrophotometer (Multiskan GO, Waltham, MA, USA) at 734 nm. A 20 µL volume of the sample extract and 200 µL ABTS solution were added to a 96-well microplate and incubated for 6 min at room temperature. The absorbance of the sample was read using a spectrophotometer at 734 nm. The results were reported as mg Trolox equivalents (Etrolox) per g sample. Methanol was used as a blank.

2.6. Experimental Design and Statistical Analysis

A central composite rotatable 2^2 design was used with three central points and 17 experiments (Table 1). An additional mixture consisting of non-extruded flour (NEM) was prepared. The independent variable levels were FMC, 17–21%; DT, 100–130 °C; and SS, 80–120 rpm. The dependent variables were TPC, antioxidant capacity (ABTS and DPPH), WSI, WAI, hardness, and EI as response variables. Data were reported as means ± SD of three repetitions.

Experimental data for each variable were used to fit into a second-order polynomial (Equation (1)), using response surface methodology (RSM).

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_{ii}^2 + \sum_{\substack{i=1 \\ i<j}}^{3} \sum_{j=1}^{3} \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

where Y is the predicted response (dependent variable); X_i and X_j are the coded process variable values (FMC, DT, and SS); β_0 is the interception value (constant), β_i is the coefficient of the first-order term; β_{ii} is coefficient of the quadratic term; β_{ij} is the coefficient of the interaction term; and ε is the error term [31]. An analysis of variance (ANOVA) and a Tukey's test ($p < 0.05$) were performed to find the difference between means using Statgraphics Centurion XV (Version 15.2.06, The Plains, VA, USA). The model fit was obtained using p-values, the lack of fit, the F-value model, the coefficient of determination (R^2), and the fitted coefficient of determination ($R_{adj}{}^2$). The desirability function method was used to obtain the individual optimum value and for the simultaneous optimization of extrusion conditions [32].

3. Results and Discussion

The experimental results are shown in Table 2. The regression coefficients were obtained by adjusting the experimental data to Equation (1). The coefficients of determination, R^2, and adjusted R^2 were determined to obtain the quality of the fitted model. The effects of independent parameters on the chemical and physical properties of extrudates during extrusion were studied using RSM. The independent parameters DT, FMC, and SS had a significant effect ($p < 0.05$) on the dependent parameters. Table 3 presents the estimated coefficients. No model was found to fit TPC and DPPH because they did not have significant coefficients.

Table 2. Response variable results via response surface methodology.

Run	EI [1] (-)	Hardness [1] N	WAI [1] g Wet/g Sample	WSI [1] %	Total Polyphenol [1] mg GAE/100 g Sample	DPPH [1] mg Etrolox/g Sample	ABTS [1] mg Etrolox/g Sample
1	1.17 ± 0.02 ^{a,b}	61.88 ± 12.30 ^{a,b,c}	4.65 ± 0.07 ****	17.75 ± 0.39 ^{ns}	4445 ± 49.27 ***	119.84 ± 6.10 ****	83.29 ± 4.48 ^{ns}
2	1.05 ± 0.02 ^{h,i}	91.79 ± 15.56 ^{f,g}	3.77 ± 0.17 ^{ns}	18.41 ± 0.19 ^{ns}	4938 ± 43.02 **	115.39 ± 4.36 ****	79.76 ± 4.72 ^{ns}
3	1.02 ± 0.04 ^{i}	65.12 ± 16.44 ^{b,c,d}	4.70 ± 0.25 ****	18.61 ± 0.21 ^{ns}	3627 ± 23.35 *	81.46 ± 2.79 ^{ns}	84.87 ± 4.84 ^{ns}
4	1.03 ± 0.02 ^{h,i}	79.04 ± 21.96 ^{d,e,f}	4.26 ± 0.09 ^{ns}	16.28 ± 1.30 ^{ns}	3238 ± 32.87 ^{ns}	69.53 ± 6.08 **	83.72 ± 4.24 ^{ns}
5	1.12 ± 0.07 ^{b,c,d,e,f}	48.84 ± 13.54 ^{a,h}	5.34 ± 0.21 ****	18.23 ± 1.74 ^{ns}	4534 ± 51.42 **	65.89 ± 2.05 ****	76.76 ± 4.06 **
6	1.06 ± 0.02 ^{f,g,h,i}	98.65 ± 21.37 ^{g}	4.45 ± 0.10 ***	16.58 ± 1.50 ^{ns}	2747 ± 20.26 **	62.05 ± 2.10 ****	79.61 ± 4.09 ^{ns}
7	1.14 ± 0.08 ^{b,c,d}	48.25 ± 14.40 ^{a,h}	5.02 ±0.29 ****	17.97 ± 1.13 ^{ns}	3305 ± 40.01 ^{ns}	98.76 ± 7.27 ^{ns}	81.49 ± 3.48 ^{ns}
8	1.04 ± 0.02 ^{h,i}	86.40 ± 19.83 ^{e,f,g}	4.45 ± 0.09 ***	16.20 ± 0.78 ^{ns}	4619 ± 52.60 ****	64.64 ± 1.40 ****	71.87 ± 3.79 ****
9	1.15 ± 0.03 ^{b,c}	72.37 ± 12.90 ^{c,d,e}	4.94 ± 0.15 ****	17.17 ± 0.51 ^{ns}	3136 ± 37.63 ^{ns}	63.30 ± 2.62 ****	84.25 ± 4.60 ^{ns}
10	1.21 ± 0.13 ^{a}	45.60 ± 14.53 ^{h}	5.08 ± 0.09 ****	19.66 ± 0.32 *	4272 ± 44.50 *	74.37 ± 3.37 *	80.38 ± 3.79 ^{ns}
11	1.06 ± 0.02 ^{g,h,i}	86.49 ± 15.62 ^{e,f,g}	4.09 ± 0.04 ^{ns}	17.36 ± 0.68 ^{ns}	2210 ± 19.98 ***	120.34 ± 8.01 ****	76.91 ± 2.67 **
12	1.13 ± 0.10 ^{b,c,d,e}	52.96 ± 17.17 ^{a,b,h}	4.91 ± 0.04 ****	21.88 ± 0.42 ****	3024 ± 18.57 *	100.15 ± 7.13 ^{ns}	80.70 ± 2.33 ^{ns}
13	1.11 ± 0.02 ^{c,d,e,f,g}	82.87 ± 21.85 ^{e,f,g}	4.52 ± 0.07 ****	18.45 ± 0.97 ^{ns}	2889 ± 12.18 *	100.37 ± 4.21 ^{ns}	89.28 ± 4.21 ^{ns}
14	1.09 ± 0.04 ^{d,e,f,g,h}	74.14 ± 15.29 ^{c,d,e}	5.28 ± 0.10 ****	16.32 ± 0.60 ^{ns}	2487 ± 24.45 ****	129.04 ± 12.30 ****	67.86 ± 1.01 ****
15	1.13 ± 0.02 ^{b,c,d}	60.11 ± 15.37 ^{a,b,c,h}	4.83 ± 0.10 ****	17.31 ± 0.67 ^{ns}	3857 ± 28.32 *	81.57 ± 5.93 ^{ns}	77.49 ± 2.73 **
16	1.17 ± 0.03 ^{a,b}	73.16 ± 15.48 ^{c,d,e}	5.13 ± 0.16 ****	17.70 ± 0.62 ^{ns}	1707 ± 12.64 ****	95.28 ± 7.39 ^{ns}	81.00 ± 2.38 ^{ns}
17	1.07 ± 0.03 ^{e,f,g,h,i}	78.26 ± 11.79 ^{d,e,f}	4.79 ± 0.23 ****	17.28 ± 0.69 ^{ns}	2803 ± 20.43 *	89.61 ± 6.18 ^{ns}	70.15 ± 3.16 ****
NEM	N.A.	N.A.	3.92 ± 0.12	17.27 ± 0.80	3475 ± 32.96	88.82 ± 5.69	88.08 ± 4.33

Identical letters in the same columns are statistically equal (HSD Tukey, $p < 0.05$); significant differences between the NEM and the design were * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$; **** $p < 0.0001$; ns—non-significant; NEM—non-extruded flour mixture (before extrusion process); N.A.— not applicable. [1] Mean ± SD; EI—expansion index; WAI—water absorption index; WSI—water solubility index; GAE—gallic acid equivalents; Etrolox—Trolox equivalents.

Table 3. Estimated coefficients of the response variables via response surface methodology.

Parameters	EI (-)	Hardness (N)	WAI (g of Gel/g Sample)	WSI (%)	Total Polyphenol (mg EAG/100 g Sample)	DPPH (mg Etrolox/g Sample)	ABTS (mg Etrolox/g Sample)
Intercept	1.10	68.53	4.84	17.25	3380.07	92.61	79.38
Linear							
X_1	0.0313 *	−14.97 ***	0.3360 ***	1.07 ***	114.59	−1.84	1.09
X_2	0.0071	−4.29 *	0.1807 **	−0.3502 *	100.31	−2.56	−3.59 *
X_3	0.0218	1.18	0.1050 *	−0.0340	−366.84	−0.6878	−0.4289
Interactions							
X_1X_2	0.0262	0.7600	0.0262	0.4775 *	212.75	0.4138	−0.6800
X_1X_3	0.0237	−4.29	−0.1013	0.3375	28.25	10.50	1.53
X_2X_3	−0.0038	1.77	−0.1338 *	0.4775 *	454.00	4.95	−0.5875
Quadratic							
X_1^2	−0.0191	−0.6611	−0.1466 **	0.7204 **	−18.62	1.00	0.0619
X_2^2	−0.0173	2.99	0.0023	−0.1106	6.48	2.58	−0.0212
X_3^2	0.0003	−1.75	0.0306	−0.0682	39.72	−6.71	0.2175
Model (*p*-value)	0.0282	0.0002	<0.0001	0.0004	N.S.	N.S.	0.0107

Significant at * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$; N.S.— non-significant; X_1—die temperature; X_2—feed moisture content; X_3—screw speed; EI—expansion index, WAI—water absorption index, WSI—water solubility index.

3.1. Effects of Extrusion Conditions on Physical Properties

3.1.1. Expansion Index

The EI measures a phenomenon in which structural transformations occur due to the production of air bubbles within the samples. The expansion index in extruded foods is an important characteristic to study due to its importance in consumer acceptability [13]. The EI of extrudates varied between 1.02 ± 0.04 and 1.21 ± 0.13 (Table 2). The quadratic model was significant, thus inferring that only the linear effect of DT was significant in the model ($p = 0.0282$) (Table 3). Figure 1a,b show that the EI value has a tendency to linearly increase with an increasing DT and SS. The FMC had no significant effect. Mazlan et al. [6] observed that the EI of corn grit and mango peel powder extrudates tended to increase with increasing SS and DT; this agrees with the results reported in the present work. This effect occurs when cutting the extrudates with a high SS, as the extrudate leaves the die under high-pressure conditions. In the same way, Pensamiento-Niño et al. [14] indicated that increasing the DT favors an increase in the EI of extrudates enriched with up to 10% mango pulp, based on taro flour. A high DT and a low FMC has the effect of causing structural and phase changes in the extrudates. This is contrary to what has previously been determined by Altaf et al. [13], who stated that using an SS between 250 and 380 rpm reduces the EI of

chickpea and rice flour extrudates. These contrasting results may be because the SS values were lower in this work. Increasing the SS causes further structural degradations due to high shear and low viscosity [13].

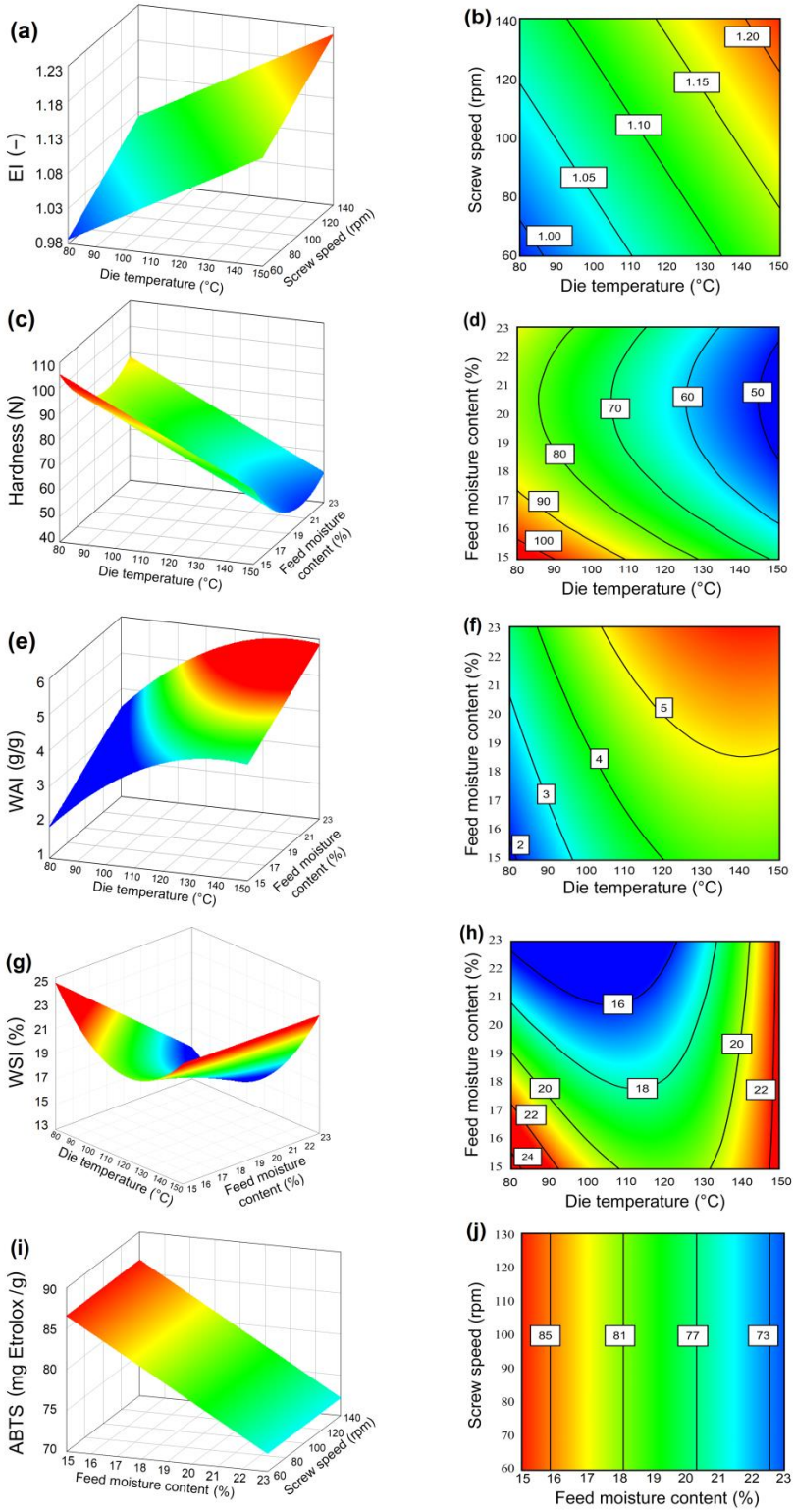

Figure 1. Response surface graphs for the effect of the extrusion conditions on the (**a**,**b**) expansion index (EI); (**c**,**d**) hardness; (**e**,**f**) water absorption index (WAI); (**g**,**h**) water solubility index (WSI); and (**i**,**j**) antioxidant capacity of the extrudates by ABTS.

3.1.2. Hardness

The texture, measured by hardness, is defined as the peak force required to penetrate the sample. Texture reflects structure and is associated with the EI. Hardness affects consumer acceptability [6,19]. The values obtained for hardness were between 45.60 ± 14.53 and 98.65 ± 21.37 N (Table 2). The quadratic model was significant (p = 0.0002), implying that the DT and FMC independent variables had a linear effect on the model (Table 3). The model data were fitted with an R^2 of 0.8635, an adjusted R^2 of 0.8014, and a lack-of-fit of 0.7129. Figure 1c,d show the effect of DT and FMC on the hardness of the extrudates, showing that the hardness was lower under the conditions of a high FMC and DT, while the highest hardness values were found under conditions of a low FMC and DT. A saddle point was present at intermediate FMC and DT values. This trend is similar to that found by Mazlan et al. [6], who developed an extrudate with corn grits and up to 25% mango peel and obtained hardness values between 34.55 N and 379.83 N that decreased with increasing DTs. These hardness values are higher than those reported in this work, where the values were less than 100 N, although the percentages of mango peel flour were higher in some mixtures. This implies that, contrary to common belief, when interacting with starch, fiber can form an expanded matrix as long as its quantity is low [33]. In an extrudate developed with taro flour and enriched with up to 10% mango pulp, the hardness values were between 0.80 and 96.54 N; these values are more in line with the present work [14]. An increase in DT leads to an increase in the gelatinization degree and a decrease in viscosity, which promotes an increase in the EI [33]. Bubble formation and a less dense product cause a reduction in hardness [10]. These data are contrary to those presented by Calderón-Castro et al. [34], who found that the degradation of material with starch increases the hardness value due to the severity of the process. This could be due to the release of OH- functional groups and their interactions with each other, causing stiffer and less elastic materials. Higher DT and SS values are related to softer extrudates, while increasing the FMC and SS values facilitates starch gelatinization, producing extrudates with lower hardness values [33].

3.1.3. Water Absorption Index

The WAI is a parameter that indicates the amount of water absorbed by the starch in excess water [11]. It represents starch digestibility in vitro and indicates the gelatinization degree [13]. The determined values for WAI were between 3.77 ± 0.17 and 5.34 ± 0.21 g wet/g sample (Table 2). When comparing the WAI value of non-extruded flour (NEM) with the values of the extrudates, it was noted that the extrudates tended to have an equivalent or higher WAI value. Consequently, the extrusion process increases the absorption of the extruded products. The quadratic model was significant ($p < 0.0001$), implying that the DT, FMC, and SS variables had a linear effect on the model. In addition, there was an interaction between DT and SS variables, and DT had a quadratic effect (Table 3). The model data were fitted with an R^2 of 0.9151, an adjusted R^2 of 0.8642, and a lack-of-fit of 0.529. Figure 1e,f show the effect of FMC and DT on the WAI of the extrudates, where the WAI was found to increase with increasing DT and FMC values. This trend is similar to that found by Sandhu et al. [10] and Karun et al. [35], who found that the WAI increases because the FMC causes an improvement in the extrudate's water retention, which causes a plasticizing effect because it decreases starch degradation. If the SS increases, the mixture's residence time is lower, and the polymeric chain length is reduced as a result of shearing; therefore, the WAI of the extrudate decreases [6,7]. Although other authors have reported the opposite, they mentioned that as the DT increases, the WAI decreases, because a high DT favors starch degradation, and the dextrinization phenomenon prevails over the gelatinization phenomenon [6,8].

3.1.4. Water Solubility Index

The WSI measures the released and solubilized polysaccharides after the addition of excess water to starch granules. It is used as a marker for the degradation of molecular components such as fibers, proteins, and starch (dextrinization) [13]. The WSI values were

between 16.20 ± 0.78 and 21.88 ± 0.42 g/100 g (Table 2). By comparing the NEM value (17.27 ± 0.80%) to those determined in the extruded samples, it was found that the extruded samples had a similar or decreased WSI. For this reason, extrusion decreases the solubility percentage in water. The quadratic model was significant (p = 0.0004), implying that the DT and FMC variables had a linear effect on the model. In addition, there was an interaction between the DT and SS variables, an interaction between the FMC and SS variables, and a square effect of DT (Table 3). The model data were fitted with an R^2 of 0.9151, an adjusted R^2 of 0.8642, and a lack-of-fit of 0.529. Figure 1g,h show how the extrudates' WSIs change due to effect of DT and FMC; one can observe that the WSI decreases as DT decreases and FMC increases. This trend is similar to that found by Pardhi et al. [11], who found that an increase in DT leads to a lower WSI. This phenomenon is caused by the formation of starch and fiber complexes and a decrease in starch content, producing a lower WSI [33]. At low FMC and high DT values, the WSI increased; this is consistent with the reported results. The severity of the process (a high DT) causes starch gelatinization because of the high levels of solubility and absorption that cause the granular structure to rupture and open [34].

3.2. Effects of Extrusion Conditions on Chemical Properties

3.2.1. Total Polyphenol Content

Polyphenols are natural compounds found in almost all vegetables and fruits. Their structure is distinguished by the presence of one or more phenolic rings, and they have the ability to scavenge free radicals [33]. This has beneficial implications in diabetes, obesity, dyslipidemia, oxidative stress, and inflammation [36]. The TPC was between 1707 ± 12.64 and 4938 ± 43.02 mg GAE/100 g of sample (Table 2). It was observed that the obtained values were higher than those obtained by Bandyopadhyay et al. [27], who developed a cookie containing 30% mango peel flour (MPF) and another cookie with 30% mango kernel flour (MKF) that presented values of 1530 ± 51 and 2240 ± 26 mg GAE/100 g of sample, respectively. The total polyphenol data did not fit any model. This means that the extrusion parameters (DT, SS, and FMC) had no effect on the polyphenol content in the studied intervals.

An ANOVA was performed to compare the results between the NEM and the 17 different extrusion conditions. It was found that there were significant differences between the NEM and the different conditions (Table 2), except for conditions 4, 7, and 9, showing that most conditions tended to maintain or increase the total phenolic content. This agrees with the reported results of Şensoy et al. [37] and Leonard et al. [33], who expressed that extrusion does not change the TPC because it activates phenolic acid polymerization. On the other hand, Samyor et al. [12] indicated that the TPC decreases due to its thermolabile nature [15]. The analysis revealed two trends: In the first case, the value tends to increase when DT, FMC, and SS decrease, with the highest TPC found below 90 °C at 16% FMC and 75 rpm. In the second case, high values were found when increasing the DT, FMC, and SS values, with the highest TPC obtained above 135 °C at 22% FMC and 125 rpm. These values are similar to those found by Salgado et al. [5], who developed an extrudate with mango and found that the TPC can be increased in two different ways at the minimum and maximum DT levels studied. This is contrary to the finding of Rani et al. [38], who showed that simultaneous increases in DT and SS have a greater negative effect on the phenolic content.

3.2.2. Antioxidant Capacity Measured by ABTS

Antioxidants are compounds capable of retarding or preventing oxidation. The antioxidant capacity is evaluated by measuring a substance's ability to reduce the radical anion ABTS to its non-radical form [30]. An ANOVA was performed to compare the results between the NEM and the 17 runs. It was found that there were no significant differences between the NEM and the extrudates (Table 2), except for conditions 5, 8, 11, 14, 15, and 17, showing that most of the runs maintained or increased the antioxidant capacity. This tendency was similar to the effect of the extrusion process on the TPC [33]. The quadratic

model was significant (p = 0.0107), thus implying that only the linear effect of FMC was significant (Table 3). Figure 1i,j show the effect of DT and FMC on the antioxidant capacity of the extrudates. When the antioxidant capacity increased, the FMC decreased. Similarly, Basilio-Atencio et al. [28] reported that the ABTS value increases because the extent of antioxidant degradation is lower than that of antioxidant release. Leonard et al. [33] stated that there are contradictory findings in the literature regarding the effect of increasing the SS, as some authors identified a decrease in the antioxidant capacity. This could be because, in addition to the bromatological composition of the raw material and the extrusion conditions, the antioxidant capacity also depends on the balance between degradation and the formation of new compounds and products that are derived from Maillard reactions and changes in phenolic compounds.

3.2.3. Antioxidant Capacity Measured by DPPH

Results obtained for DPPH were between 62.05 ± 2.10 and 129.04 ± 12.30 mg Etrolox/g sample (Table 2). It was not possible to represent the antioxidant capacity data measured by DPPH in a model because the extrusion process parameters were not statistically significant. This could be due to the fact that the response variable intervals were small, so significant changes were not observed. An ANOVA was performed to compare the results between the NEM and the experimental runs. It was found that there were significant differences between the NEM and the different extrudates (Table 2), except under conditions 3, 7, 12, 13, 15, 16, and 17, showing that most of the runs tended to maintain or increase the antioxidant capacity as measured by DPPH. The antioxidant capacity hinges on the composition and quantity of bioactive compounds. Therefore, TPC reduction might not affect the extruded food [33]. The DPPH value tended to increase when the DT, FMC, and SS decrease, as higher values were found below 100 °C at 17% FMC and 80 rpm. This trend is similar to that reported by Rani et al. [38], who suggested that, like the TPC, the effect of extrusion on the antioxidant capacity was similar; therefore, there was a decrease in antioxidant capacity due to the increase in the DT and SS. However, other authors have not found similar trends, finding that increases in the DT, SS, and FMC cause the DPPH content of the sample to increase and, thus, increase its antioxidant capacity [12].

3.3. Optimization and Validation

The optimal extrusion conditions were determined using numerical optimization according to the concept of desirability. The EI and antioxidant capacity measured by ABTS had significant models; however, the R^2 was less than 0.60, so they were not used for design optimization. In the same way, the TPC and antioxidant capacity measured by DPPH did not have a significant model and were also not considered. Design optimization was performed by maximizing the WAI and minimizing the hardness and the WSI. The rest of the variables remained in the same range. For each response variable, the experimentation intervals and importance values used can be found in Table 4. The optimal conditions were an FMC of 21.88%, a DT of 120.66 °C, and an SS of 66.36 rpm. The product obtained had the following characteristics: TPC = 3402 mg GAE/100 g sample; antioxidant capacity measured by ABTS = 79.38 mg Etrolox/g and DPPH = 90.09 mg Etrolox/g; EI = 1.10; hardness = 63.66 N; WAI = 5.41 g/g; WSI = 16.20%; and a desirability value of 0.870. The extrudates obtained under the optimal extrusion conditions were analyzed in triplicate to validate the models. No significant differences were found between the predicted values ($p > 0.05$) and the experimental results. The identified relationship between the responses and the extrusion process variables confirms the adequacy of the model [31]. Figure 2 shows the optimized extruded food (photography taken with a Realme Gt 2 Pro).

Table 4. Optimum extrusion process parameter values and responses.

	Importance	Target	Experimental Value		Optimum Value	Experimental Value [a]	Desirability
			Min	Max			
Experimental Factor							
DT (°C)	3	Optimum	89.7731	140.227	120.66		0.870
FMC (%)	3	Optimum	15.6364	22.3636	21.8844		
SS (rpm)	3	Optimum	66.3641	133.636	66.3642		
Response Variable					Predicted values		
EI (-) [NS]	3	Range	1.02	1.21	1.10	1.16 ± 0.06	
Hardness (N)	3	Minimize	45.60	98.65	63.66	66.92 ± 5.51	
WAI (g/g)	3	Maximize	3.77	5.34	5.41	5.00 ± 0.43	
WSI (%)	3	Minimize	16.20	21.88	16.20	15.93 ±0.55	
Total Polyphenol (mg GAE/100 g sample) [NS]	3	Range	1707	4938	3402	3317 ± 308	
DPPH (mg Etrolox/g) [NS]	3	Range	62.05	129.04	90.09	85.19 ± 10.96	
ABTS (mg Etrolox/g) [NS]	3	Range	67.86	89.28	79.38	76.70 ± 4.00	

NS: non-significant; DT: die temperature; FMC: feed moisture content; SS: screw speed; EI: expansion index; WAI: water absorption index; WSI: water solubility index. [a] Values are the means of three measurements ± standard deviation (SD).

Figure 2. Optimized extruded food.

3.4. Bromatological Composition of the Optimized Extruded Food

The results of the bromatological analyses of the NEM and the extrudate under optimal conditions are presented in Table 5. As a result of moisture loss, the protein, fat, ash, and carbohydrate values increased in the optimized extrudate compared with the NEM.

Table 5. Bromatological composition of the raw material and optimized extruded food.

Sample	Moisture (%)	Protein, Nx6.25 (%)	Fat (%)	Carbohydrate (%)	Ash (%)	Crude Fiber (%)
Non-extruded flour mixture (NEM) [1]	9.95	5.90	2.63	75.18	1.47	4.87
Optimized extruded food	3.10	6.50	3.84	81.46	1.70	3.40

NEM—non-extruded flour mixture (before extrusion process). [1] Theoretically calculated.

The extrudate's chemical and physical properties are affected by the bromatological composition of the raw materials [15]. A high protein content affects the viscoelastic properties, increasing the extrudate's resistance to breakage because the starch granules retain water in the form of steam and form thermoplastic complexes [13]. Protein denaturation in the raw material results in a higher protein digestibility after extrusion. Overall, the addition of by-products with a high protein content produces less expanded extrudates with a higher hardness and density. During extrusion, lipids function as plasticizers and give the extrudates a sticky texture [33]. They are usually present in low quantities because they cause a reduction in the thermal and mechanical energy produced by friction. The decrease in fat content contributes to improving the hardness; this can be attributed to the loss of lipids in the form of oil through the die extruder, as well as to the formation of lipid structures with starch and proteins [13].

3.5. Comparison of Techno-Functional Properties

Samples of 100% WCF, 100% MPF, and 100% MKF were extruded, and their techno-functional properties were compared with that of the optimized extrudate (Table 6). When comparing the EI of the extrudates, it was observed that 100% WCF and 100% MKF extrudates had a similar expansion; this could be due to the fact that the starch amounts in both flours were similar. Although the optimized extrudate contained 58.33% WCF and 8.34% MKF, the EI was closer to the values obtained for the extrudate with 100% MPF; this variation could be due to the interaction of starch with the high dietary fiber content [39]. Similar optimized extrudate hardness values were obtained in the 100% WCF and 100% MKF extrudates. However, the value for the extrudate containing 100% MPF was almost double; this could be due to the fact that the fiber quantity in the mango peel tends to increase the extrudate hardness [24]. Generally, mango peel increases the hardness of extrudates because their expansion is decreased. The hardness of corn–mango peel extrudates developed by Dey et al. [40] increased as the mango peel content exceeded 8%; a similar tendency was found in the extrudates studied in this work. The WAI values were different for the four extrudates evaluated; however, the WAI of the optimized extrudate was similar to that of the 100% MPF extrudate, corresponding to the highest values, because the dietary fiber content is linked to the WAI value [41]. The WSIs were also different for the four analyzed extrudates; nevertheless, the WSI of the optimized extrudate was between that of the 100% WCF extrudate and the 100% MKF extrudate. This effect could be due to the chemical composition of the extrudates, as they had higher starch contents than the 100% MPF extrudate. The highest WSI value (100% MPF extrudate) could be attributed to the fact that during the extrusion process, the degradation of food starch, fibers, and proteins was higher [13]. The TPCs of the extrudates developed with 100% MPF and 100% MKF were similar; however, the value for the 100% WCF extrudate was almost half that of the other extrudates. This was due to the flour's chemical composition, since it has been reported that mango by-products have a high number of phenolic compounds [7]. The optimized extrudate had a similar TPC to that of the 100% WCF extrudate. This could be due to the fact that the optimized extrudate contained mainly WCF (58.33%) and that the TPC can also be affected by the extrusion conditions [37]. The 100% MKF extrudate had the highest antioxidant capacity as measured by DPPH and ABTS, followed by the 100% MPF extrudate and, finally, the 100% WCF extrudate. The antioxidant capacity of the optimized extrudate was similar to that of the 100% WCF extrudate, probably due to the chemical composition of the optimized extrudate, as it contains mostly WCF. The optimized extrudate's TPC and antioxidant capacity measured by DPPH and ABTS were similar to those of the 100% WCF extrudate [5].

Table 6. Techno-functional properties of extrudates.

Sample	EI [1] (-)	Hardness [1] N	WAI [1] g Wet/g Sample	WSI [1] %	Total Polyphenol [1] mg GAE/100 g Sample	DPPH [1] mg Etrolox/g Sample	ABTS [1] mg Etrolox/g Sample
Extruded 100% white corn flour (WCF) [1]	1.42 ± 0.07	50.85 ± 5.66	3.85 ± 0.26	11.49 ± 0.78	3966 ± 356	82.06 ± 9.86	110.92 ± 12.40
Extruded 100% mango peel flour (MPF) [1]	0.93 ± 0.04	102.86 ± 11.16	5.08 ± 0.27	27.65 ± 1.06	6357 ± 270	118.71 ± 6.32	187.11 ± 15.34
Extruded 100% mango kernel flour (MKF) [1]	1.42 ± 0.08	52.88 ± 5.51	2.82 ± 0.17	17.29 ± 0.81	6163 ± 292	175.17 ± 15.59	318.38 ± 25.39
Optimized extruded food (58.33% WCF, 33.33% MPF, 8.34% MKF)	1.16 ± 0.06	66.92 ± 5.51	5.00 ± 0.43	15.93 ± 0.55	3317 ± 308	85.19 ± 10.96	76.70 ± 4.00

[1] Mean ± SD; EI: expansion index; WAI: water absorption index; WSI: water solubility index; GAE: gallic acid equivalents; Etrolox: Trolox equivalents.

4. Conclusions

RSM was used to determine the optimal extrusion process variables in order to obtain a new extruded food containing mango by-products. The statistical models developed exhibited a good fit, with R^2 values of >0.86. The extrusion conditions, DT, and FMC had a significant effect on the physical and chemical properties of the extrudates. An increase in the DT led to extrudates with a higher IE, IAA, and ISA and a lower hardness, while increasing the FMC increased the IAA and decreased the hardness, ISA, and antioxidant capacity (as measured through ABTS). On the other hand, an increase in the screw speed increased the expansion, hardness, and IAA and decreased the ISA of the extrudates. The optimal conditions were an FMC of 21.88%, a DT of 120.66 °C, and an SS of 66.36 rpm. The development of new foods containing mango by-products could represent an opportunity to reduce the ecological impacts and take advantage of the beneficial properties of mango by-products and to enhance the nutritional content of new foods. In future studies, it will be necessary to study the functional properties of foods developed using by-products.

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