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Design of gluten-free instant whole acorn-maize blended flour by extrusion process**

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A b s t r a c t. Gluten-free products have become popular due to the increasing number of people with gluten-related disorders. In this study, the effect of whole acorn flour and whole maize flour levels of 25, 62.5, and 100% and extrusion process variables, including screw speed 120, 160, and 200 rpm and feed moisture content of 12, 15, and 18%, on the production of gluten-free products were investigated. The results demonstrated that increasing the whole acorn flour had a positive effect on water absorption index, angle of repose, carr index, and Haussner ratio, but negatively impacted expansion index and water solubility index. Increasing feed moisture had a notable impact on increasing bulk density and hardness and bowl life hardness while decreasing water solubility index. The optimal conditions for the fabrication of the blended meal were whole acorn flour 63.81%, 120 rpm screw speed, and 18% feed moisture content. The samples produced in these conditions had an 18.23 expansion index, 6.34 humidity, 4.93 g g^{-1} water absorption index, 0.16 g g^{-1} water solubility index, 5.70 N hardness, 2.85 N bowl-life, L^* 60.29, 26.14 angle of repose, 1.15 Haussner ratio, and 14.31 carr index. Our findings suggest an innovative approach for applying whole acorn flour to prepare nutritious gluten-free instant products by extrusion processing.

K e y w o r d s: gluten free, extrusion, instant product, acorn flour, functional properties

1. INTRODUCTION

Gastrointestinal diseases are a significant concern in human societies, particularly in developing countries. The high glycemic index and gluten content of most foods restrict the diets of people with celiac disease. Additionally, a lack of fiber is associated with gluten-free diets due to the use of refined flour or starch. This situation has created a need to develop alternative flours that are non-allergenic and widely available for the production of gluten-free foods (Sotelo-Díaz *et al.*, 2023). Whole grains are a good source of phytochemicals and contain greater amounts of fiber and minerals than their refined counterparts (Singh *et al.*, 1997). Multigrain products can also provide enhanced functionality. Whole maize flour (WMF), the most widely grown cereal in the world, is primarily starchy and contains iron and zinc, making it a cost-effective option (Sotelo-Díaz *et al.*, 2023). Whole acorn flour (WAF) is introduced as a rich source of carbohydrates (84%), especially starch (31-51%), proteins (8%), and unsaturated fatty acids (0.7-9%), particularly linoleic acid, as well as microelements, such as calcium, magnesium, potassium, and other minerals (copper, iron, manganese, and zinc), which exceed the content in cereal by an average of four to five times (Korus *et al.*, 2017; Rakić *et al.*, 2006). Food products made from WAF are also known to be high in fiber and phenolic compounds (Beltrão Martins *et al.*, 2020). An increasing number of scientific reports have explored the potential of WAF in manufacture of biscuits, bread, pastries, porridge, and coffee-like beverages and have acknowledged its significant impact on the emulsifying, stabilizing, and texturizing properties of these products (Pasqualone *et al.*,

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2019; Beltro Martins *et al.*, 2020; Korus *et al.*, 2017). In the past decade, a variety of ready-to-eat products have been successfully produced to provide quick preparation, convenience, and high nutritional value in the food market. The extrusion technology has been applied to the processing of instant flour on an industrial scale (Hashemi *et al.*, 2017). Thermoplastic extrusion involves the application of mechanical shear, heat, and pressure to transform raw materials into a final product with specific functional properties (Lotfi *et al.*, 2020). Many recent studies on expanded products have utilized whole grain flours, which are considered to be healthy (Oliveira, 2017). Sotelo-Díaz *et al.* (2023) used extrusion cooking to enhance the instant properties of corn-cowpea powder, while Obilana *et al.* (2014) studied the influence of extrusion treatment on the physical and functional properties of millet-based instant powder. Wang *et al.* (2020) reported the improvement of instant properties of kudzu powder through extrusion. However, limited information is available on optimization of the effects of extrusion on the functional properties of instant gluten-free products. Given the increasing demand for nutritious and ready-to-eat products, particularly for vulnerable populations, the aim of this study was to develop an optimized protocol for the production of whole grain extrudates and to analyze their physicochemical, functional, and reproducible properties.

2. MATERIALS AND METHODS

2.1. Materials

2.1.1. Raw materials

To prepare the whole flour, commercial whole maize grain was obtained from Golden Maize in Mashhad, Iran, and whole acorn meal (Cultivar BRS 310) was supplied from Lorestan province. The whole grains were ground and milled using a laboratory scale disk hammer mill (Toos shekan) equipped with a 0.8 mm sieve opening to obtain fine particles. The resulting sample was packed in a polyethylene bag at 25°C for further use.

Protein, fat, ash, and moisture analysis of the raw materials were performed according to standard AACC (2000) procedures. All results are presented as means (SD) of triplicate analyses.

2.2.2. Formulation extrusion

WAF was used as a substitute for WMF at various levels (25, 62.5, 100%). Treatments were formulated by spraying a calculated amount of distilled water to achieve the desired feed moisture content (12, 15, and 18% db), vacuum sealed in polyethylene bags, and stored overnight at 4°C. Extrusion was carried out in a co-rotating twin screw extruder (DS56, Jinan Saxin, China) with a feed rate of 40 kg h^{-1}, a length-to-diameter ratio of 20:1, a nominal compression ratio of 1:1, and a 3 mm round die. The cylinder temperature in the transition zone was 150°C. In order to reach the final humidity below 5%, the extrudates were dried in an oven at 60°C for 5-6 h. To prepare the final instant maize-acorn blend, the expanded products were ground with a laboratory scale hammer mill (Toos shekan) and sieved through a 250-μm laboratory sieve before analysis (Hashemi *et al.*, 2017).

2.2.3. Determinations

2.2.3.1. Cross-sectional expansion index (EI)

Ten pieces from each experimental run were selected at random, and the diameter was measured at two different locations on the extrudate with a digital caliper. The EI was calculated according to the following Eq. (1) (Lotfi *et al.*, 2020):

$$\text{EI} = \text{Extrudate Diameter}/\ \text{Die diameter}. \tag{1}$$

2.2.3.2. Water absorption index (WAI) and water solubility index (WSI)

The WAI and WSI of the extrudates were analyzed using the formula developed by Atukuri *et al.* (2019). The expanded sample (3 g) was ground and mixed with 30 ml of distilled water and stored at room temperature for 30 min, stirring gently. It was then centrifuged at 3000 rpm for 15 min (Eppendorf). After centrifugation, a supernatant and a gel were obtained. The supernatant was poured into an evaporating dish of known weight and dried in an oven at 110°C until the weight was constant. The gel was weighed and the WAI (g g^{-1}) was calculated as the gram of gel obtained per gram of sample collected according to Eq. (2). The WSI (%) was the weight of dry solids in the supernatant after evaporation and was expressed as a percentage of the original sample as shown in Eq. (3):

$$\text{WAI} = \frac{Weight\ of\ gel(g)}{Weight\ of\ sample(g)}, \tag{2}$$

$$\text{WSI} = \frac{Weight\ of\ dissolved\ solids\ in\ supernatant}{Weight\ of\ sample} 100. \tag{3}$$

2.2.3.3. Texture analysis

The hardness (N) of the dry expanded products was determined using a Texture Analyzer WITH a 50 kg load cell (TA-XT2i Plus, Stable Micro Systems Ltd.). From each treatment, 2 samples were randomly selected and the test consisted in measuring the maximum force required to penetrate a 10 mm diameter cylindrical pin into the sample at a constant rate of 1 mm per second (Oliveira *et al.*, 2017).

2.2.3.4. Texture analysis after immersion in milk (Bowl-life)

The extrudates were soaked in milk with 3% fat for 3 min and then drained for 10 s. The sample volume was the same as for the dry grain texture analysis, and the milk volume was four times greater than the sample weight. The analysis conditions were the same as for the instrumental texture analysis of the dry grain. Six measurements were made for each treatment (Oliveira *et al.*, 2017).

2.2.3.5. Color

The color of the extrudates was measured with ColorQuest XE (HunterLab, USA) in terms of L^*, a^*, and b^*: L^* (brightness; 0 = black, 100 = white), a^* (+60 = reddish, -60 = green), and b^* (+60 = yellowish, -60 = bluish) (Bisharat *et al.*, 2015).

2.2.3.6. Bulk density, Haussner ratio (HR), and Carr index (CI)

The flow behavior of textured powder is calculated using CI and HR. Both HR and CI represent a numerical value that depends on both the bulk density and the tapped density of the powder (Bodhmage, 2006; Carr, 1965). These two parameters were calculated as shown below:

$$\text{Haussner ratio} = \frac{\rho T}{\rho B}, \tag{4}$$

$$\text{Carr index} = \frac{\rho T - \rho B}{\rho T} 100. \tag{5}$$

The tap density was measured by tapping the graduated cylinder 100 times on a bench so that the powder settled and no settlement was visible. The tapping process consisted of an average of two tapping processes per second. Density was calculated as demonstrated below:

$$\rho T = \frac{Ws}{Vt}, \tag{6}$$

where: Vt is the volume occupied after tapping (m^3). The particle density was measured using a helium multipycnometer (Quantachrome MVP 4AC232). The procedure consists in passing helium under pressure from the reference cell into a cell containing a sample of material. The particle density was determined by the ratio between the mass of the sample (Ws, kg) and the average particle volume (Vp, m^3):

$$\rho p = \frac{Ws}{Vp}. \tag{7}$$

2.2.3.7. Angle of repose

The angle of repose corresponds to the flow properties of the material and is a direct indication of the possible flowability (Carr, 1965). The lower the value, the easier the material flows. This property is mainly used to construct conveyor belts for material handling. The angle of repose was measured according to standard method ASTM C1444 (ASTM, 2000) by pouring the sample into a funnel held at a fixed height above a flat support as described by Bodhmage (2006). According to ISO 3435/1, the funnel outlet was located at a height of 6 cm above the pad. The sample was poured into the funnel by hand at a constant rate until the tip of the powder cone touched the funnel nozzle. The diameter of the cone formed was measured at the base at four points to determine the angle of the cone using the following formula:

$$A = \tan^{-1}\left(\frac{H}{R}\right). \tag{8}$$

where: H is the height of the cone (m) and R is the mean radius of the base of the cone (m). The extrudate was collected on the flat pad and the angle between the horizontal and the slope of the sample on the left and right sides was averaged. This test was repeated three times for each treatment.

2.2.3.8. Dispersibility

Dispersibility of a powder in water refers to its ability to break down into particles that can pass through a sieve with a specified pore diameter. This property was evaluated using the method described by Shin *et al.* (2003). Specifically, 15 g of the sample was placed in a 100 ml graduated cylinder filled with water, stirred for 90 s, and allowed to settle for 15 min. The volume of settled particles was then subtracted from 100 and the resulting difference was reported as the dispersibility percentage.

2.2.3.9. Statistical analysis

Table 1 demonstrates the coded levels and the actual values of the independent variables. To study the effects of the independent variables (maize:acorn flour ratio: x_1, screw speed: x_2, humidity: x_3) on the dependent characteristics, the central composite design method is used. The data analysis was carried out using Design Expert 7.0.0 software. Six central points were specified in the test plan to calculate the repeatability and error of the test. The following equation was used to describe the models:

$Y = b_0+b_1x_1+b_2x_2+b_3x_3+b_1x_1^2+b_2x_2^2+b_3x_3^2+b_1x_1x_2+b_1x_1x_3+b_2x_2x_3$.

The coefficient model is represented by b (constant coefficient), b_1, b_2, and b_3 (linear effect), b_{11}, b_{22}, and b_{33} (quadratic effect), b_{12}, b_{13}, and b_{23} (interaction effect). The significance of the model was determined by analysis of variance (ANOVA) for each response. R^2 (explanatory coefficient), Adj-R^2 (corrected explanatory coefficient), and CV (coefficient of variation) were examined for adequacy of the predicted model. The optimal conditions were selected using the optimization technique for the different variables, while either less or one range was considered for the responses (Hashemi *et al.*, 2017).

Table 1. Independent numerical variables and their levels

Numerical variables	Symbol	Coded variable levels		
		-1	0	+1
WAF (%)	A	25	62.5	100
Moisture (%)	B	12	15	18
SS (rpm)	C	120	160	200

3. RESULT AND DISCUSSION

3.1. Physicochemical characteristics of raw materials

The physicochemical characteristics of whole maize grain included the following: 11.78±0.34% moisture (db), 6.37±0.13% proteins (db), 1.63±0.20% fat (db), 0.56± 0.01% ash (db), and 2.84±0.02% total fiber (db), and the whole acorn meal was reported to contain 7.96±0.14% moisture (db), 5.01±0.06% proteins (db), 7.79±0.05% fat (db), 1.35±0.06% ash (db), and 5.91±0.11% total dietary fiber (db).

Multiple regression and analysis of variance (ANOVA) provided information that helped to select the appropriate model and to examine the statistical significance of the response variables. The adequacy of the model was assessed using the coefficient of determination (R^2) and the coefficient of variation (CV). No significant lack of fit was found for all response variables (Table 2), indicating that these models accurately predict responses.

3.2. Expansion index (EI)

The EI of the extrudates varied from 13.21 to 19.38 g cm^{-3}. As shown in Table 2, both the linear and quadratic terms for WAF and linear terms for moisture showed a significant effect on the EI ($p \leq 0.005$) as shown in Fig. 1a. Increasing the percentage of WAF added independently led to a decrease in the expansion of the product. The lowest level of EI was observed in the sample containing 100% WAF. This treatment was the richest in dietary fiber and fat. It has been observed that feeds with increasing dietary fiber content decrease the EI of extrudates (Bisharat *et al.*, 2015).

However, the simultaneous increase in both parameters, *i.e.* screw speed and WAF, led to an increase in the EI of the product. According to in Fig. 1b the EI elevates significantly with increasing moisture content. The influence of feed moisture on the density and EI of extrudates has been well documented in previous studies (Ding *et al.*, 2006; Piyasiri *et al.*, 2020). Higher feed moisture content during extrusion can improve dough elasticity through melt plasticization, leading to increased gelatinization and increased EI of final products (Akande *et al.*, 2017).

The role of water as a plasticizer in amorphous regions of starch granules is a well-known phenomenon. Water plays a crucial role in the process of starch gelatinization and contributes significantly to the rheological properties of the melt and the formation of gas bubbles (Hagenimana *et al.*, 2006). It was observed that increasing the screw speed up to 170 RPM rpm resulted in an extrudate with a higher EI. It can be assumed that higher screw speeds with increasing temperature are probably due to the increase in the degree of improving superheating of the water in the extruder. The phenomenon of water superheating has been observed to cause the formation of bubbles and a reduction in melt viscosity, which ultimately leads to an increase in dough elasticity and an improvement in the EI of products. This effect has already been demonstrated by Fletcher *et al.* (1985).

The application of the screw speed higher than 170 rpm resulted in increased degradation and dextrinization of the starch polymer, which was reflected in a reduced EI of the extruded product. This phenomenon is due to the higher mechanical shear at a high screw speed, which increases starch fragmentation during extrusion cooking at low feed moisture (Hagenimana *et al.*, 2006).

For the EI prediction model, the equation can be constructed in terms of the coded values as follows:

$$EI = 19.25-1.40\times A+0.3860\times B-0.0140\times C+0.4025\times AB-0.0950\times AC+0.1300\times BC-1.75\times A^2-0.1873\times B^2-1.07\times C^2.$$

Table 2. Coefficient of variables in the suggested model for response variables

Parameter	ER	Dry hardness (N)	Bowl-life hardness (N)	WAI	WSI
A	0.0026**	0.0046	0.0193	0.012	0.0113
B	0.013	0.0113	0.0436	0.109	0.0277
C	0.97*	0.676	0.2847	0.880	0.3348
AB	0.0106	0.0421	0.0313	0.0002	-
AC	0.8963	-	0.2995	0.0501	-
BC	0.8781	-	0.803	0. 478	-
A^2	0.0405	0.0362	0.0024	0.0232	-
B^2	0.693***	0.0351	0.1155	0.3158	-
C^2	0.1427	-	0.0076	0.6758	-
R^2	0.799	0.863	0.892	0.947	0.881
Adjust R^2	0.637	0.807	0.795	0.901	0.839

A – WAF (%), B – moisture (%), C – SS (rpm). Significant at: *P < 0.05; **P < 0.01; ***P < 0.001.

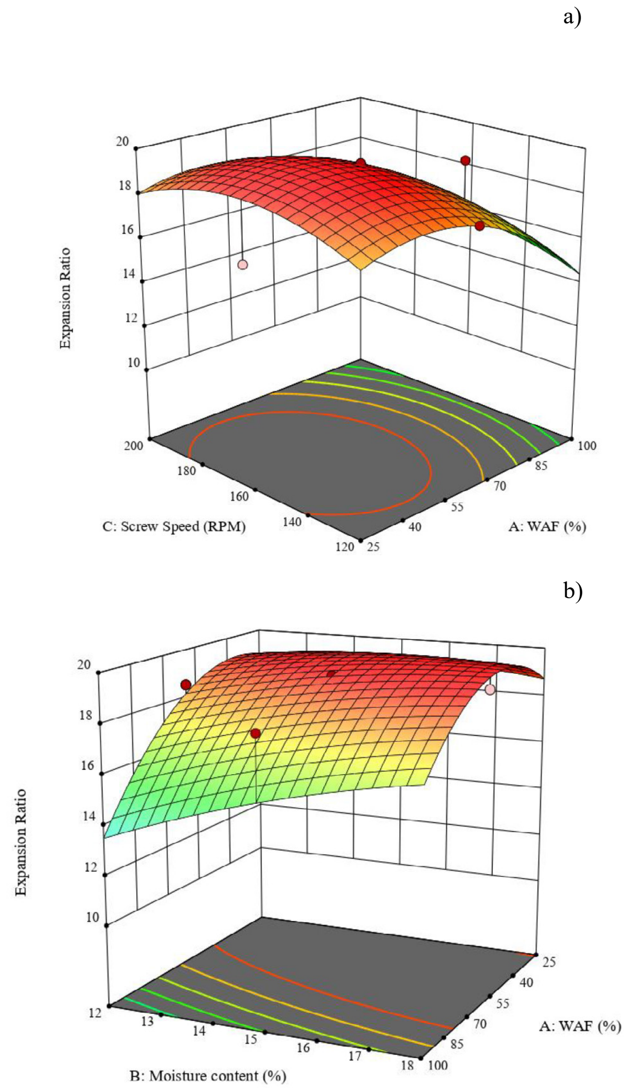

Fig. 1. Response surface plot for the influence of the screw speed-moisture content (a) and the WAF-moisture content (b) on the EI of the final products.

3.3. Dry texture analysis

The consumer's perception of the hardness and softness of samples is linked to the extent of expansion and the structure of the cells of the extruded material (Ding *et al.*, 2006). Hardness is the maximum force required to break a sample (Lotfi *et al.*, 2020). The minimum and maximum hardness of the textured material were found to be 1.534 and 8.140 (N), respectively. According to Table 2, the linear effect of AF and moisture on hardness was significant ($p \leq 0.005$).

As the feed moisture increased, the hardness of the expanded mixtures also increased (Fig. 2). Generally, products with a low EI have fewer holes, thicker cell walls, greater breaking strength, and a harder texture. Moisture acts as a lubricant and reduces the shear force and process temperature, slowing the melting rate of the molten material and resulting in a low EI, high hardness product (Atukuri *et*

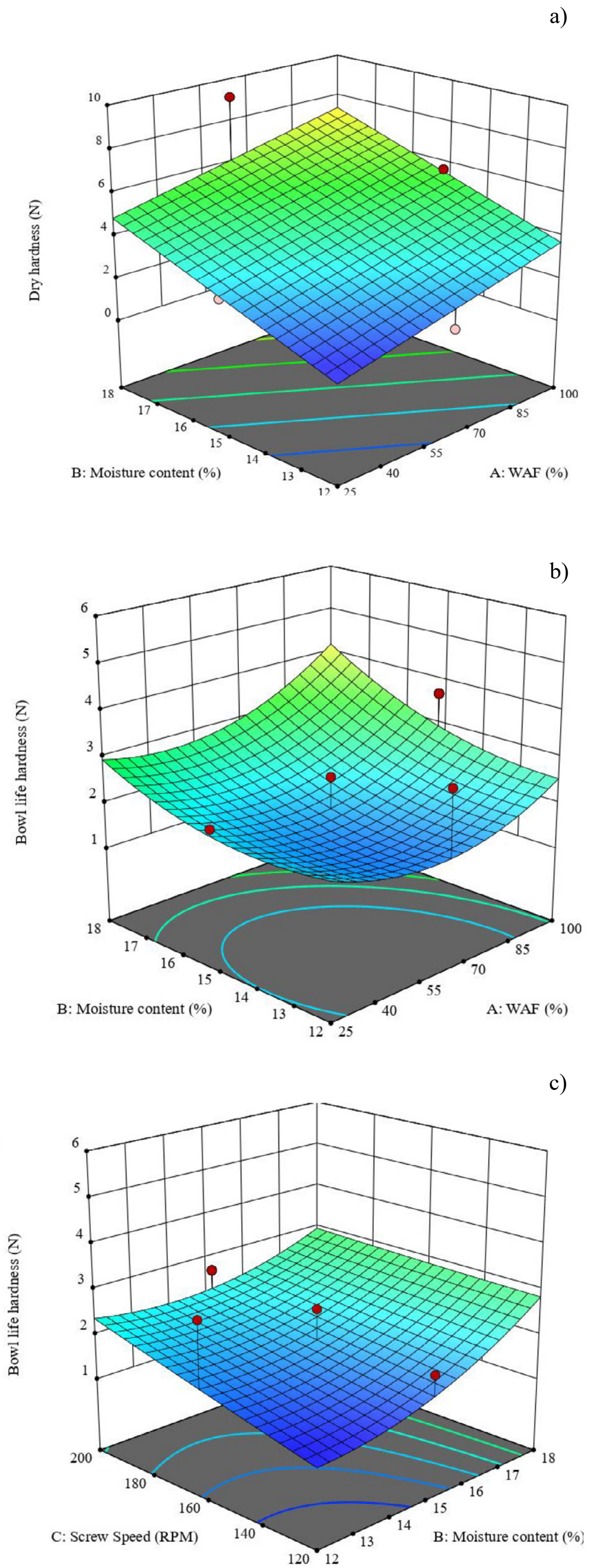

Fig. 2. Response surface plot for the influence of the screw speed-moisture content (a) and the WAF-moisture content (b) on the hardness and the influence of the screw speed-WAF (c) and the WAF-moisture content (d) on the bowl life of the final products.

al., 2019). Moreover, high moisture levels limit the growth of bubbles and air cells in the product, causing compression of bubbles and reducing the amount of cooked starch, resulting in a denser and harder texture (Sotelo-Díaz *et al.*, 2023). The findings reported by Lotfi *et al.* (2020) are consistent with the recent research.

3.4. Texture analysis after soaking in milk (bowl life)

Bowl life refers to the length of time that extrudates can maintain their texture crispness after soaking in milk. The index is related to the structure integrity, water absorption capacity, solubility, and density of the expanded product. Texture crispness is a crucial factor influencing the acceptability and consumption of expanded products (Oliveira *et al.*, 2017). Changes in microstructure characteristics that occur after immersing the products in milk are due to the plasticizer role of amorphous compounds, such as starch and proteins. As a result, the force required to soften the product is reduced and the crispness of the product decreases. The index range was between 1.18 and 5.23 (N). According to the analysis of variance, the linear and quadratic effects of the amount of WAF, moisture, and their interaction on the texture hardness after the immersion were significant. Increasing the level of WAF up to 70% led to decreased texture hardness and increased crispiness of the samples (after immersion in milk) due to the increase in the fiber amount and protein reduction. The hardening of the flow path due to the increased amount of fiber in the extrudates led to reduced moisture absorption, resulting in expanded products with a firm texture after soaking in milk, and increased texture (Robin *et al.*, 2015). After the immersion in milk, the formation of a layer of lipids and micelles on the surface of the product limited mass transfer and moisture absorption. As a result, the absorption of moisture by the product was lower compared to the state of immersion in water, leading to reduced crispiness and hardness of the products. Thus, expanded products that retain their texture after being immersed in milk are representative of their proper texture (Oliveira *et al.*, 2017). Cheewapramong *et al.* (2002) investigated the characteristics of extruded cereal with the aim of evaluating the partial substitution effect of defatted peanut flour with maize. The results demonstrated that the addition of peanuts decreased the bowl life. The potential shelf life of the samples can be inferred from their water absorption capacity. Samples with high moisture absorption tend to have a shorter bowl life due to quicker texture softening. Therefore, reducing moisture absorption could extend the bowl life of the products. The difference in WAI can be attributed to the nature of starch or dietary fiber in the compounds (Brennan *et al.*, 2013). Increasing the screw speed reduces the molecular weight of amylose and amylopectin, leading to increased water absorption capacity and greater product crushing in milk. This results in a softer texture and increased stickiness of the product (Ravindran, 2011).

The equation of the fitted model for the hardness of the extrudates is as follows:

$$\text{Hardness} = 3.28-0.5200\times A+0.3300\times B+0.0600\times C-0.6250\times AB+0.4500\times AC-0.1750\times BC+0.3318\times A^2-0.8182\times B^2-0.1682\times C^2.$$

The equation of the obtained model for the bowl life of the extrudates is as follows:

$$\text{Bowl life hardness} =1.88+0.3894\times A+0.5952\times B+0.3554\times C+0.2237\times AB+0.4085\times AC-0.2963\times BC+0.6753\times A^2+0.4167\times B^2+0.0156\times C^2.$$

3.5. Water absorption index (WAI)

The WAI indicates the water holding capacity of starch after swelling in excess water and determines the integrity of starch in an aqueous dispersion (Ding *et al.*, 2006). The range of changes in the WAI values for the extruded blends was from 3.99 to 5.29. As shown in Table 3, the effects

Table 3. Coefficient of variables in the suggested model for response variables

Parameter	L^*	a^*	b^*	Angle of repose	(HR)	(CI)	Dispersibility
A	0.0001	0.0001	0.0001	0.0307	0.0282	0.0030	0.0173
B	0.0301	0.0068	0.4377	0.0017	0.0280	0.0401	0.01660
C	0.295	0.391	0.0470	0.0029	0.1982	0.6200	0.1680
AB	-	-	0.3115	0.0108	-	-	-
AC	-	-	0.0500	0.190	-	-	-
BC	-	-	0.2100	0.0050	-	-	-
A^2	-	-	0.3620	0.0020	-	-	-
B^2	-	-	0.0690	0.5201	-	-	-
C^2	-	-	0.0781	0.0032	-	-	-
R^2	0.819	0.868	0.893	0.898	0.854	0.824	0.738
Adjust R^2	0.785	0.844	0.896	0.810	0.722	0.665	0.688

Explanations as in Table 2.

of the feed moisture, screw speed, and barrel temperature and the interaction effect of the feed moisture and barrel temperature on WAI were significant (P≤0.01). The WAI of the samples was higher than that for textured plantain/corn/soybean weaning diets, but lower than that for expanded barley meal and similar to that for extruded millet meal (Gulati *et al.*, 2016). The WAI value tended to improve with increasing feed moisture and acorn meal, possibly due to the increase in swollen crude fiber, which plays an important role in increasing WAI (Hashemi *et al.*, 2017). The WAI improved with the increasing screw speed, while a decreasing trend was observed for samples processed at 200 rpm (Fig. 3).

Boosting the screw speed due to the simultaneous increase in heat and shear force causes the starch to become more dextrinized and degraded. Water as a plasticizer reduces the breakdown of starch granules and leads to an increased WAI (Oliveira *et al.*, 2017). It is noteworthy that starch granules must reach a certain degree of conversion in order to achieve a significant WAI. A lower screw speed and higher feed moisture during the extrusion process can reduce polymer damage and increase the availability of hydrophilic groups that can bind more water, leading to an increase in WAI values (Li *et al.*, 2023). It is likely that the WAI increased due to changes in the cell wall structure and its opening through extrusion. Additionally, the higher degree of porosity leads to greater water uptake during rehydration.

The fitted model for WAI based on the coded values of the variables is shown in the following equation:

$$WAI=4.92+0.0867\times A-0.0014\times B-0.0048\times C-0.3235\times AB+0.0110\times AC-0.0818\times BC-0.3081\times A^2+0.0634\times B^2-0.1226\times C^2.$$

3.6. Water solubility index (WSI)

The WSI is often regarded as an indicator of starch conversion and molecular component breakdown through extrusion. It indicates the quantity of soluble polysaccharide liberated from the starch polymer of extruded products, providing insights into the degree of starch modification (Lotfi *et al.*, 2020; Singh *et al.*, 1997). The range of changes in the WSI of the samples was between 0.149 and 0.350 g g^{-1} (Table 2). According to Table 3, the linear effects of the feed moisture, screw speed, and barrel temperature, the interaction effect between feed moisture and screw speed, and the quadratic effect of feed moisture on WSI were significant (P ≤ 0.01).

The current solubility results were similar to the previous report for extruded rice flour, but lower than values reported for extruded millet-barley weaning mix and higher than for potato-based snack extrudates (Ding *et al.*, 2005; Singh *et al.*, 2014). The WSI decreased with the increasing forage moisture and acorn meal as well as auger speed and acorn meal simultaneously (Fig. 3a, b). Similar effects of

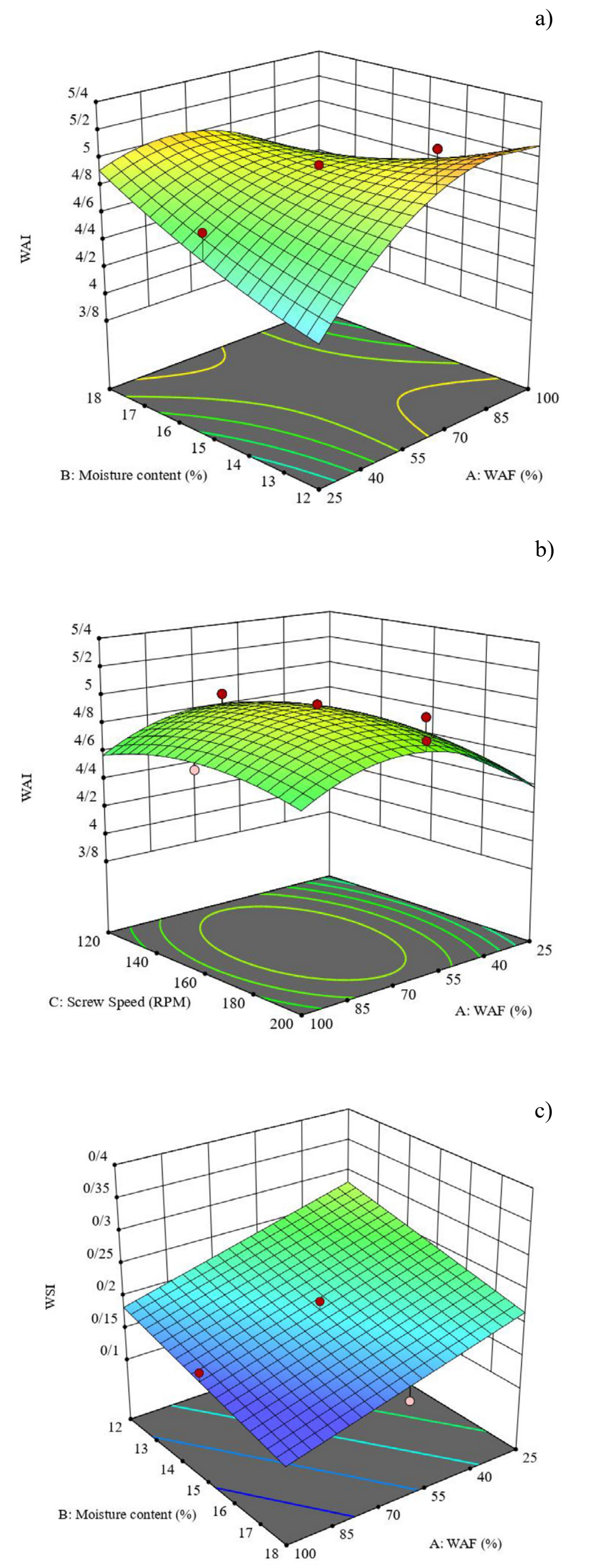

Fig. 3. Response surface plot for the influence of the WAF-moisture content (a) and the WAF-crew speed (b) on the WAI and the influence of the WAF-moisture content (c) on the WSI of the final products.

feed moisture and screw speed on WSI have been previously reported for rice-, corn-, and sorghum-based extrudates (Obilana *et al.*, 2014; Hagenimana *et al.*, 2006). Higher feed moisture was expected to decrease viscosity, reduce shear, and reduce the degree of dextrinization of starch polymers, resulting in a lower WSI. Higher feed moisture also reduced the friction between the melted dough and the inner surface of the barrel, leading to minimal damage to the starch granules and, consequently, a lower WSI of the extrudate.

The equation of the obtained model is as follows:

$$WSI = 0.2007-0.0495\times A-0.0324\times B+0.0020\times C.$$

3.7. Color

Color changes during the extrusion process can serve as a visual indicator to evaluate the intensity of browning reactions, degree of curing, and destruction of pigments (Olivera *et al.*, 2018). In the results of the variance analysis, the linear model and independent effect of the acorn flour parameter, humidity for lightness and red color component and for yellowness component in the quadratic polynomial model, and the independent effect of the acorn flour percentage, screw speed, and the interaction effect of acorn flour and screw speed were all statistically significant ($p<0.05$). As shown in Fig. 3, increasing the WAF and screw speed simultaneously caused a reduction in brightness and an improvement in the redness and yellowness of the product. This is related to the higher protein and crude fiber content in the feed as well as lysine and other amino acids that can react with regenerating sugars to cause the extruded products to darken in the Maillard reaction (Bisharat, 2015). The presence of brown pigments in the WAF also contributed to the observed darkness of the product. Increasing the screw speed accelerates the reaction between protein and sugars, leading to a darker color of the product. In contrast, reducing the amount of humidity reduces the lightness due to various competitive effects. High temperature along with low humidity intensifies the Maillard reaction, which causes the color to darken. With an increase in humidity, the amount of heat generated by friction decreases, causing the Maillard reaction to occur less intensively and color intensity to decrease. In these conditions, the destruction of pigments decreases, and the yellowness factor increases (Ravindran *et al.*, 2011). Improvement of the WAF percentage results in a reduction in starch gelatinization and a reduction in longitudinal and transverse expansion. As a result, the dispersion of pigments on the surface of the product decreases, resulting in a darker final color. Boosting moisture content decreases the melt temperature and viscosity, reducing the destruction of pigments in the extruder and increasing the brightness indicators while decreasing the redness and yellowness (Boluk *et al.*, 2023). Rhee *et al.* (2004) reported similar results regarding the reduction in the yellowness index with increasing humidity for corn-based products, while Gulati *et al.* (2016) reported a reduction in the redness index in millet flour with increasing humidity. Increasing the screw speed due to the effect of shearing force along with high temperature accelerates the caramelization reactions, resulting in a darker color of the product (Atukuri *et al.*, 2019). Brennan *et al.* (2013) demonstrated that increasing the amount of green and yellow peas up to 10% resulted in an increase in the green, redness, and browning index of extrudates.

The equations of the obtained models for color changes are as follows:

$$L^* = 59.35-5.15\times A+1.41\times B+0.2770\times C$$

$$a^*=13.17+1.31\times A-0.4150\times B+0.1180\times C$$

$$b^*=29.93+2.94\times A-0.3010\times B+0.7530\times C+0.4438\times AB-0.7812\times AC+0.5563\times BC-0.9800\times A^2-0.6800\times B^2-0.2400\times C^2.$$

3.8. Powder flow tests

Powder flowability is a critical property that determines the ability of powders to flow and fluidize freely without clumping, sticking, or compacting. The flowability of powders depends on several factors, including moisture content, particle shape, size, density, and surface charges (Bodhmage *et al.*, 2006; Sotelo-Díaz *et al.*, 2023). Among these factors, humidity is the most significant factor affecting the flowability of powders. As humidity increases, the likelihood of liquid bridges forming between solid particles increases, resulting in lumpy powder and reduced flowability (Bodhmage *et al.*, 2006). Adhesion and consistency of powders are also factors that contribute to the reduction in flowability. The HR and CI values given in Table 4 were used to determine the flow behavior of the samples.

3.8.1. Angle of repose

The angle of repose is generally considered a key indicator of powder flowability, with lower angles indicating better flow properties. This property is strongly influenced by several factors, including moisture content, surface characteristics (such as smoothness and sphericity), particle size, and storage time, as well as chemical nature, liquid bridge formation, electric charges, Van der Waals bonds, and magnetic dipole-dipole reactions (Akande *et al.*, 2017). The regression analysis of the present study indicated that the linear effects of feed moisture and screw

Table 4. Flowability of powders in terms of Carr's index (CI), and Hausner's ratio (HR)

Flow type	(HR)	(CI)
Excellent flow	$HR < 1.11$	$CI < 10$
Good flow	$1.12 < HR<1.18$	$11 < CI < 15$
Fair flow	$1.19 < HR < 1.25$	$16 < CI < 20$
Passable flow	$1.26 < HR < 1.34$	$21 < CI < 25$
Poor flow	$1.35 < HR < 1.45$	$26 < CI < 31$
Very poor flow	$1.46 < HR < 1.59$	$32 < CI < 37$
Extremely poor flow	$HR > 1.60$	$CI > 38$

speed as well as the quadratic effect of moisture on the angle of repose were all significant (P≤0.05) (Table 3). Furthermore, the interaction effect between feed moisture and screw speed was also significant (P≤0.05). The mixed extrudates exhibited angles of repose ranging from 25.24 to 35.17, placing them in the category of very free-flowing powders according to the classification of the flow type, which includes very flow-flowing powders<30°, free-flowing 30-38°, fair to passable flow 38-45°, cohesive 45-55°, and very cohesive >55° (Shah *et al.*, 2008). As shown in Fig. 4a, by improving the WAF content in the extrudates, the angle of repose increased. This finding is attributed to the reduction in particle size resulting from the increased screw speed and shear force application, as the angle of repose tends to increase as particle size decreases due to the increased friction between particles and the decreased tendency of larger particles to stick together (Obilana *et al.*, 2014). These issues agree with previous studies conducted by Wang *et al.* (2020) and Sotelo-Díaz *et al.* (2023). Rennie (1999) concluded that powders with particle sizes greater than 200 microns exhibit free-flowing behavior, while powders with smaller particle sizes have lower and poor flowability due to the increased adhesion and contact area between particles. Increased humidity was observed to decrease the angle of repose due to the increased moisture absorption by the particles, which weakens their internal bonds and facilitates faster water penetration (Fig. 5b). Powders with higher angles of repose exhibit stronger internal connectivity and are less likely to sink after being poured into liquids. Different production conditions can significantly affect the flowability and angle of repose of powders. Reduction in surface moisture, adhesion, and hygroscopicity can decrease the angle of repose (Carr *et al.*, 1965). However, increased moisture content can lead to the generation of solidified bridges and increased adhesion between fragments, resulting in higher angles of repose.

The equations of the fitted models after neglecting the effect of non-significant factors for the non-coded form of the process variables were as follows:

Angle of repose = $29.93+0.8010\times A-1.35\times B+1.25\times C+1.11\times AB-0.498\times AC-1.28\times BC+2.52\times A^2+0.4014\times B^2-2.34\times C^2$.

3.8.2. Haussner ratio (HR)

The handling and processing of powders are greatly influenced by their ability to flow, which is determined by various physical properties *e.g.* size, shape, surface structure, particle density, bulk density, moisture content, temperature, pressure, and fat (Hashemi *et al.*, 2017). While the angle of repose is a commonly used measure of powder flowability, Haussner ratio (HR) has been proposed as a more comprehensive indicator that accounts for both the dynamic and static properties of powder movement and transport (Rennie *et al.*, 1999). In this study, a binomial statistical model was developed for HR, and the effect of formulation components was evaluated. According

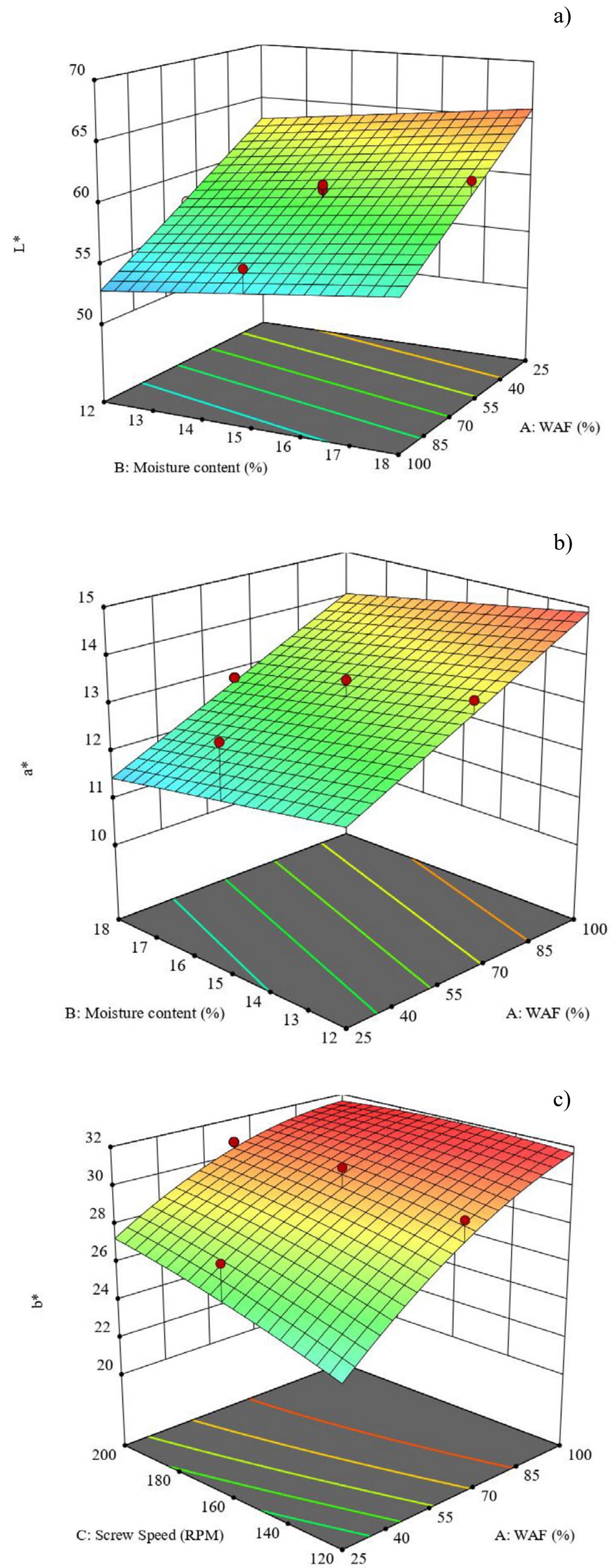

Fig. 4. Response surface plot for the influence of the WAF-moisture content (a), the moisture content-screw speed (b), and the WAF-screw speed (c) on the color parameters (L^*, a^* and b^*) of the final products.

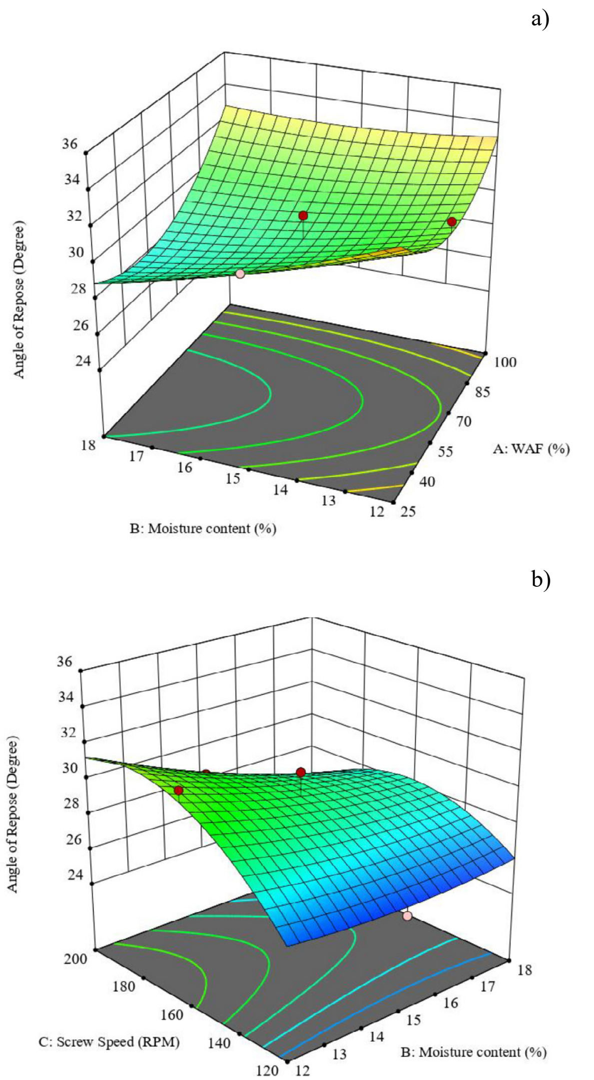

Fig. 5. Response surface plot for the influence of the WAF-moisture content (a) and the moisture content-screw speed (b) on the angle of repose.

to the research result, the independent effect of the WAF and humidity on the HR of the extruded powder was significant (P≤0.05). The range of HR was between 1.13 and 1.64, which is classified according to the standard table as excellent to flowable powders. Figure 6a reveals that the simultaneous increase in the acorn powder and screw speed resulted in an elevation of HR. This finding can be attributed to the higher shear force and increased porosity of the product resulting from the more expanded and porous structure. Porosity is a critical parameter that impacts on the agglomerate strength of extruded powder, as higher porosity facilitates greater air volume distribution among particles, making water more accessible (Sharma *et al.*, 2012). Meanwhile, humidity can alter the degree of cohesion due to its effect on the glass transition temperature.

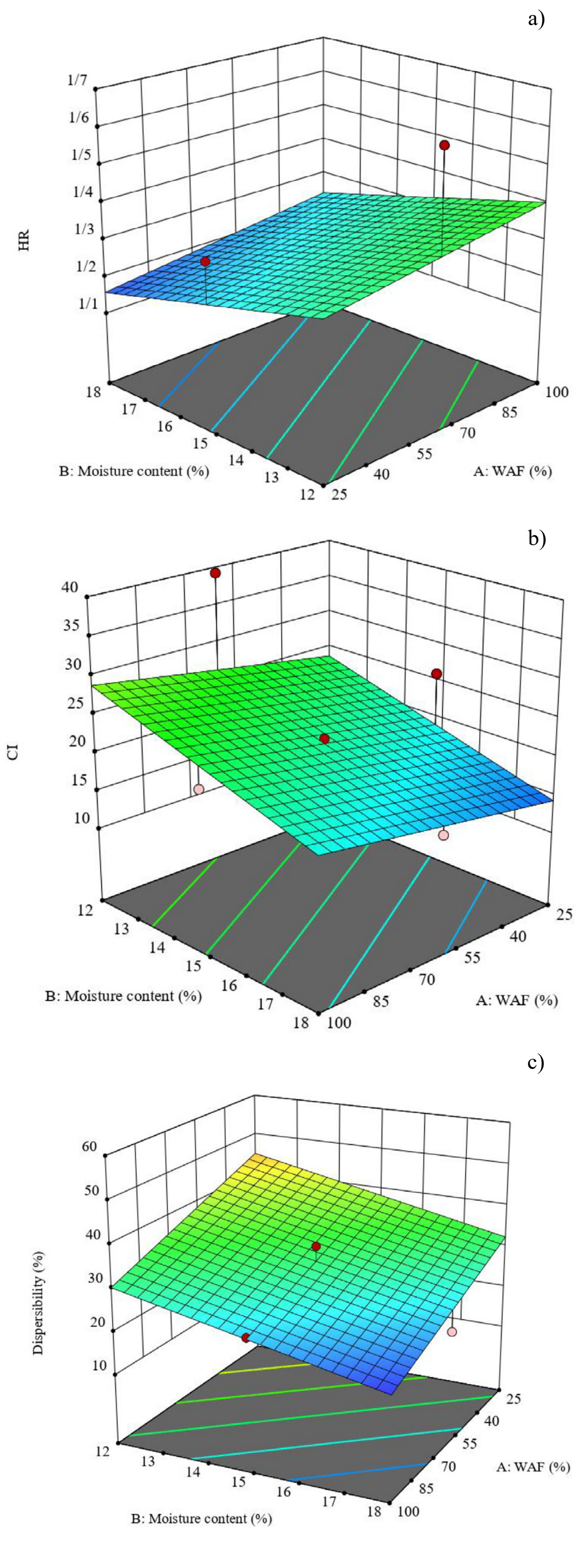

Fig. 6. Response surface plot for the influence of the variables on the Haussner ratio (a), Carr index (b), and dispersibility (c) of the final products.

Increasing humidity can lead to the formation of liquid bridges between particles, resulting in higher consistency and lumpiness of the powder. In contrast, reducing the moisture content of the powder can reduce the formation of liquid bridges between particles, leading to decreased cohesion of the powder (Wang *et al.*, 2020). Our finding is consistent with the report of other investigators who have studied extruded corn-cowpea mixes (Sotelo-Díaz *et al.*, 2023).

The equation of the fitted model is as follows:

HR = +1.28+0.0420×A-0.0810×B+0.0450×C.

3.8.3. Carr index

The flowability of powders is often quantified by the Carr index (Cl%), which has an inverse relationship with flowability, such that higher values indicate weaker flowability. In this study, a two-term statistical model was proposed for the Carr coefficient, and the effect of formulation components on this parameter was investigated. According to the result, the independent effects of the WAF amount, screw speed, and humidity on the Carr index of the powder were significant ($P \leq 0.05$). The Carr ratio, calculated to be between 11.76 and 27.86, classified the powder behavior as having good flowability according to the standard table (Table 3). As shown in Fig. 6b, the Carr index increased and flowability decreased with the increasing acorn powder amount and rotation speed. The difference in mass, particle density, and moisture content of the formulation components can explain this finding. Powders with smaller particle sizes tend to have poor flowability due to their larger surface area per unit mass (Rennie *et al.*, 1999). The fineness of particles increases the contact surface and elevates the Carr index and Haussner coefficient, ultimately leading to decreased powder flowability (Bodhmage, 2006). Conversely, powders with rough surfaces and larger sizes tend to have better flowability, as confirmed by the results of other studies (Obilana *et al.*, 2014). Additionally, high amounts of fat in powders can reduce their flowability (Wang *et al.*, 2020). Cohesion is an internal property of powders and is related to the intermolecular forces that keep particles together. When particles collide, they may stick together and form a mass, unless they are broken by a force greater than the cohesive force. Humidity and contact surface are effective factors in determining the degree of cohesion (Sharma *et al.*, 2012). In fact, as the temperature increases with the increasing screw speed, the humidity decreases, leading to a reduction in the generation of liquid bridges and decreasing the cohesion of powders. Related findings have been reported by Sotelo-Díaz *et al.* (2023) and Wang *et al.* (2020).

The equations of the fitted models are as follows:

CI = +21.57+2.53×A-4.75×B+2.59×C,

HR = +1.28+0.0420×A-0.0810×B+0.0450×C.

3.8.4. Dispersibility

Whole grain extrudates are precooked products that can be easily reconstituted by adding water or milk, which makes them a convenient option for consumers. The dispersibility of extrudates in water is determined as an indicator of their ability for immediate reconstitution. In other words, when the surface of each particle is easily wetted, the material is rapidly dispersed through the solvent. This ability is crucial for instant reconstitution and consumer acceptability (Sharma *et al.*, 2012; Akande *et al.*, 2017). The dispersibility changes were in the range from 17.53 to 51.07, showing appropriate reconstitution ability (Fig. 6c). For a product to be considered instant, it must complete these stages within a few seconds. Therefore, the final products in this study can be considered as instant porridge. The high dispersibility of the extrudates is an important functional quality that enhances their convenience and consumer acceptability. The ability of the extrudates to be instantly reconstituted upon the addition of water or milk is a desirable characteristic that can save time and effort during preparation. The findings of this study highlight the importance of evaluating the dispersibility of food products as an indicator of their instant reconstitution ability. These results can be useful for optimizing the production of whole grain extrudates and other food products with desired functional properties.

The equation in terms of coded factors that can be used to make predictions about dispersibility is as follows:

Dispersibility = 31.42-7.37×A-6.38×B+3.57×C.

4. CONCLUSIONS

Optimal conditions considered for the production of expanded whole grain with minimum hardness, HR, CI, angle of repose, maximum ER, WAI, crispness (mainly after immersion in milk), and intermediate dispersibility were obtained. Lighter products were also preferred. They were determined to be 63.81% WAF, 18% moisture, and 120 rpm screw speed. The experimental values for producing textured mixtures were an EI of 18.23%, dry hardness of 5.70 (N), bowl-life crispness of 2.85 (N), HR of 1.15, CI of 14.31, angle of repose of 26.14, and L^* of 60.29. Samples produced in these optimal conditions exhibited no significant difference from the optimal values. The addition of the WAF improved the water absorption index and dispersibility in water, while decreasing expansion, L^*, and b^* in the mixture. The developed model was statistically valid and adequately predicted extrudate properties for different processing variables.

This finding has demonstrated that WAF can be successfully used as a substitute for WMF in gluten-free products. Additionally, the extrusion process was found to be effective in the production of instant ingredients that can be prepared quickly with nutritional benefits and desired flow properties, which is important for their handling, processing, and end use. The optimal conditions for producing blended flour with desired properties were determined, and the developed model was determined to be statistically valid and adequately predicted extrudate properties for different processing variables. Future work is recommended to evaluate sensory analysis, nutrient bioavailability, and shelf stability to improve the general acceptance of these blends.

Conflicts of Interest: The Authors do not declare any conflict of interest.

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