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SUBPRODUTOS DE MANDIOCA (*Manihot esculenta*) E PALMITO PUPUNHA (*Bactris gasipaes Kunth*) COMO POTENCIAIS MATÉRIAS-PRIMAS PARA O ENRIQUECIMENTO NUTRICIONAL DE MASSAS ISENTAS DE GLÚTEN À BASE DE SORGO (*Sorghum bicolor*)

CASSAVA AND PEACH PALM BY-PRODUCTS AS POTENTIAL RAW MATERIALS FOR NUTRITIONAL ENRICHMENT OF SORGHUM-BASED GLUTEN-FREE PASTA

SUBPRODUCTOS DE YUCA Y PALMA COMO POTENCIALES MATERIAS PRIMAS PARA EL ENRIQUECIMIENTO NUTRICIONAL DE PASTA SIN GLUTEN A BASE DE SORGO

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Resumo

A maioria das massas isentas de glúten disponíveis no mercado apresenta baixo valor nutricional. Farinha de sorgo e de subprodutos do processamento de palmito pupunha e de mandioca foram utilizadas para desenvolver massas isentas de glúten, visando conferir enriquecimento nutricional e sustentabilidade às formulações. Em um delineamento fatorial completo 2^3 com dois pontos centrais, avaliaram-se os efeitos das proporções de farinha de palmito pupunha (PF = x_1), de sorgo (SF = x_2) e de mandioca (CF = x_3) sobre a composição proximal, os compostos fenólicos totais (CFT), a capacidade antioxidante *in vitro*, o cozimento, a textura e a cor das massas desenvolvidas. Modelos de primeira ordem foram significativos ($p < 0,05$) para fibra alimentar e DPPH, limítrofes para CFT e perda de sólidos no cozimento (PSC) e não significativos para dureza. A formulação 8 (15% PF, 60% SF, 15% CF) apresentou o maior enriquecimento nutricional entre as avaliadas, com 24,32 g/100 g de fibra alimentar, 5,24 g/100 g de lipídios, 2,91 g/100 g de cinzas, 211,56 mg de equivalentes de ácido gálico (GAE)/100 g de fenólicos e 3,42 mg de equivalentes de Trolox (TE)/g (ABTS), mas a maior PSC (15,39%), indicando compromisso entre enriquecimento nutricional e qualidade de cozimento. PF e CF aumentaram a luminosidade das massas, e SF a reduziu. Os resultados indicam viabilidade tecnológica da proposta, com obtenção de massas isentas de glúten com elevado teor de fibras a partir de subprodutos; sendo que uma avaliação sensorial se faz necessária para verificar a aceitabilidade.
Palavras-chave: fibras alimentares; atividade antioxidante; dureza; propriedades de cozimento.

Abstract

Most commercial gluten-free pasta has low nutritional value. In this study, sorghum flour and by-products from peach palm and cassava processing were used to develop gluten-free pasta, aiming to improve its nutritional profile and enhance formulation sustainability. Using a 2^3 full factorial design with two central points, we evaluated the effects of the proportions of peach palm flour (PF = x_1), sorghum flour (SF = x_2), and cassava flour (CF = x_3) on proximate composition, total phenolic content (TPC), in vitro antioxidant capacity, cooking quality, texture, and color of the developed pasta. First-order models were significant ($p < 0.05$) for dietary fiber and DPPH, borderline significant for TPC and cooking loss (CL), and non-significant for hardness. Formulation 8 (15% PF, 60% SF, and 15% CF) showed the highest nutritional enrichment among all formulations, containing 24.32 g/100 g of dietary fiber, 5.24 g/100 g of lipids, 2.91 g/100 g of ash, 211.56 mg GAE/100 g of phenolics, and 3.42 mg TE/g (ABTS). However, it also presented the highest CL (15.39%), indicating a trade-off between nutritional enrichment and cooking quality. PF and CF increased the luminosity (L*) of the pasta, whereas SF reduced it. These results demonstrate the technological viability of using agro-industrial by-products to produce gluten-free pasta with high fiber content; nonetheless, sensory evaluation is needed to confirm consumer acceptability.

Keywords: dietary fiber; antioxidant activity; hardness; cooking properties.

Resumen

La mayoría de las pastas libres de gluten disponibles en el mercado presentan un bajo valor nutricional. Harina de sorgo y de subproductos del procesamiento de palmito pupuña (*peach palm*) y de yuca se utilizaron para desarrollar pastas libres de gluten, con el fin de aportar enriquecimiento nutricional y sostenibilidad a las formulaciones. En un diseño factorial completo 2^3 con dos puntos centrales, se evaluaron los efectos de las proporciones de harina de palmito pupuña (PF = x_1), de sorgo (SF = x_2) y de yuca (CF = x_3) sobre la composición proximal, los compuestos fenólicos totales (CFT), la capacidad antioxidante *in vitro*, la calidad de cocción, la textura y el color de las pastas desarrolladas. Los modelos de primer orden fueron significativos ($p < 0,05$) para la fibra dietética y el DPPH, limítrofes para los CFT y la pérdida de sólidos por cocción (PSC), y no significativos para la dureza. La formulación 8 (15% PF, 60% SF, 15% CF) presentó el mayor enriquecimiento nutricional entre las evaluadas, con 24,32 g/100 g de fibra dietética, 5,24 g/100 g de lípidos, 2,91 g/100 g de cenizas, 211,56 mg de equivalentes de ácido gálico (GAE)/100 g de fenólicos y 3,42 mg de equivalentes de Trolox (TE)/g (ABTS), pero la mayor PSC (15,39%), lo que indica no compromiso

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entre el enriquecimiento nutricional y la calidad de cocción. La PF y la CF aumentaron la luminosidad de las pastas, mientras que la SF la redujo. Los resultados indican la viabilidad técnica de la propuesta, obteniendo pastas libres de gluten con un alto contenido de fibra a partir de subproductos; no obstante, es necesaria una evaluación sensorial para verificar su aceptabilidad.

Palabras clave: Fibra dietética; actividad antioxidante; dureza; propiedades de cocción.

1. Introduction

Pasta is among the most widely consumed food products globally. Its popularity underscores the need for alternative formulations that address the growing demand for healthier, more sustainable, and convenient foods, particularly for individuals with specific dietary restrictions. Gluten-free pasta, formulated for those with gluten intolerance, is often characterized by high caloric content and limited nutritional value. It is estimated that approximately 1% of the global population is affected by celiac disease, while an additional 10% may experience adverse reactions to gluten (Papagianni et al., 2024).

Sorghum (*Sorghum bicolor*), a nutrient-dense cereal from the Poaceae family, is gaining agricultural importance due to its adaptability to diverse environments, drought tolerance, and resistance to fungi and mycotoxins. Although primarily used as animal feed in Brazil, sorghum presents promising potential for human consumption due to its rich nutritional profile, including phenolic compounds, essential minerals, and a starch content of up to 87% - a key attribute for gluten-free baked goods (Paiva et al., 2019).

The production of heart of palm (*Bactris gasipaes* Kunth – “palmito pupunha”) is considered advantageous because of the plant’s early maturation, prolific shoot production, and resistance to enzymatic browning. Brazil remains the world’s leading producer and exporter of canned heart of palm, accounting for 95% of global output. However, up to 84% of harvested stems are discarded during processing. The basal portion, unsuitable for canning due to its fibrous texture, can be milled into flour, which has shown potential for nutritional enhancement of baked products while maintaining acceptable sensory characteristics (Danesi et al., 2018).

Cassava (*Manihot esculenta*) is a dietary staple food in many developing countries, valued for its high energy content and capacity to grow in poor soil conditions. The fibrous by-product generated during cassava starch extraction, known

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as cassava bran, contains residual starch and dietary fiber and may be utilized in food applications. Its composition, however, depends on the processing methods employed (Ronko et al., 2020).

The incorporation of dietary fiber into gluten-free pasta formulations offers both nutritional and technological benefits. Fiber contributes to improved nutritional value and may help preserve the microstructure of pasta by maintaining starch granule integrity (Mercier et al., 2016). Furthermore, the use of food industry by-products as raw materials provides not only nutritional benefits, through the inclusion of fiber and bioactive compounds, but also economic and environmental advantages.

This study aimed to develop gluten-free pasta using rice flour, psyllium, and xanthan gum, commonly employed ingredients in gluten-free formulations, while evaluating the effects of incorporating varying proportions of sorghum flour and flours derived from peach palm and cassava by-products on the technological, nutritional and in vitro antioxidant properties of the product.

2. Material and Methods

2.1 Preparation of Flours and Pasta Formulation

The raw materials used for the preparation of sorghum flour (SF), cassava by-product flour (CF), and peach palm by-product flour (PF) were donated by local companies in Paraná, Brazil.

Sorghum flour was obtained from AGR-8 Soluções em Agronegócio (Londrina, Paraná, Brazil). The grains were washed to remove impurities resulting from the harvesting process, then dehydrated in a forced-air oven (MA035; Marconi, Piracicaba, Brazil) at 40 °C for 24 hours, following the method described by Queiroz et al. (2020). The dried grains were subsequently milled using a STAR-FT51® mill.

Cassava by-product flour was derived from cassava bran, the solid residue from starch extraction, donated by Amifec (Maria Helena, Paraná, Brazil), which supplied the material already dehydrated and obtained from raw material declared safe for consumption. The material was ground using a STAR-FT51® mill according to the methodology reported by Navacchi et al. (2012).

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Peach palm by-product flour was produced from residues generated during the processing of canned and minimally processed hearts of palm, donated by Frateli (Guaraqueçaba, Paraná, Brazil). The basal portion of the stem, which is unsuitable for canning due to its fibrous texture, was chopped, washed, sanitized, and then dehydrated in a forced-air oven (MA035; Marconi) at 40 °C for 24 hours. The dehydrated material was milled using a STAR-FT51® mill, as described by Danesi et al. (2018).

Other ingredients, including rice flour (RF), soybean oil, salt, xanthan gum, and psyllium, were purchased from local markets in Ponta Grossa, Paraná, Brazil. Rice flour was the base ingredient and was replaced by SF, PF and CF according to the experimental design (Table 1); it was therefore absent from formulation 8. Preliminary tests were conducted to define the amount of water required to obtain a workable dough. Water was added according to the needs of each formulation, adjusted to the composition of the flour blend, remaining close to a 1:1 water:flour ratio. The concentrations of soybean oil (3%), salt (1%), xanthan gum (1%), and psyllium (5%) were fixed across all formulations. A 2^3 full factorial design with two central point replicates was employed (Table 1), varying the proportions of SF, CF, and PF. Pasta preparation involved mixing and homogenizing the ingredients in a planetary mixer (KitchenAid K45SS), followed by shaping into fettuccine-style nests using a pasta-cutting machine (Le Cuisine).

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Table 1. Full factorial design 2^3 with two central points (coded and real values) for the development of gluten-free pasta

Sample	PF (%) x_1	SF (%) x_2	CF (%) x_3	Rice flour (%)
1	(-1) 5	(-1) 30	(-1) 5	50
2	(+1) 15	(-1) 30	(-1) 5	40
3	(-1) 5	(+1) 60	(-1) 5	20
4	(+1) 15	(+1) 60	(-1) 5	10
5	(-1) 5	(-1) 30	(+1) 15	40
6	(+1) 15	(-1) 30	(+1) 15	30
7	(-1) 5	(+1) 60	(+1) 15	10
8	(+1) 15	(+1) 60	(+1) 15	0
9	(0) 10	(0) 45	(0) 10	25
10	(0) 10	(0) 45	(0) 10	25

(PF) = peach palm by-products flour, (SF) = Sorghum flour, (CF) = Cassava by-products flour

The nests were dehydrated in a forced-air oven (MA035; Marconi) at 40 °C for 12 hours and then hermetically sealed for storage. The responses of the experimental design were evaluated based on the physical, nutritional and antioxidant characteristics of the pasta (Section 2.9). A schematic diagram of the production process is presented in Figure 1.

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Figure 1 Pastas production process

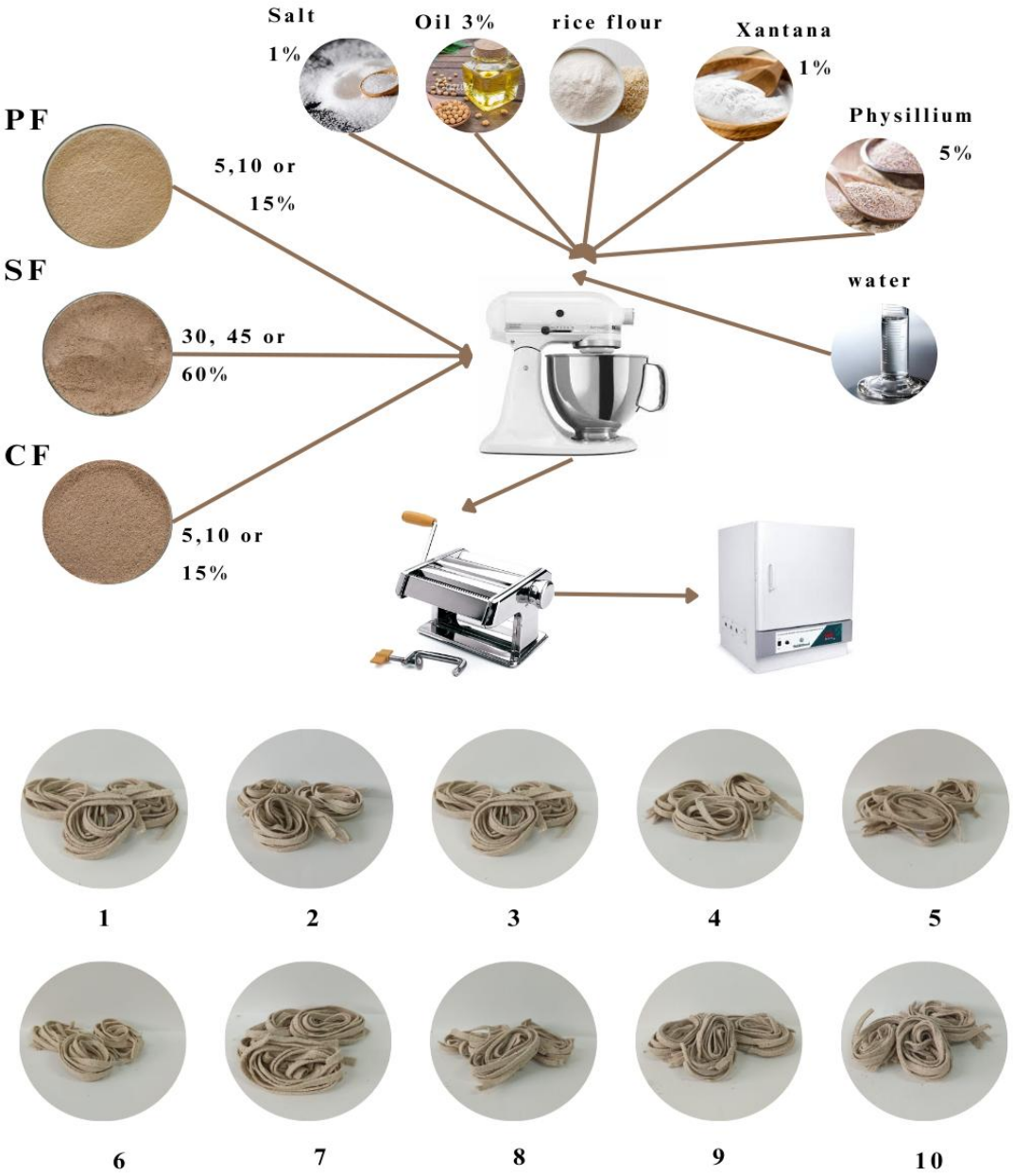

Figure caption: PF - peach palm flour; SF - sorghum flour; CF - cassava flour

2.2 Characterization of the flours

The flours (SF, PF and CF) were characterized for proximate composition (Section 2.4), total phenolic content and antioxidant activity by the DPPH and FRAP

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assays (Section 2.5). ABTS was not determined in the flours.

2.3 Characterization of the pasta formulations

The pasta formulations (Table 1) were characterized for proximate composition (Section 2.4), total phenolic content and antioxidant activity by the ABTS, DPPH and FRAP assays (Section 2.5), and for cooking properties (Section 2.6), texture (Section 2.7) and color (Section 2.8). The results for the flours and for the pasta formulations are presented and discussed separately (Sections 3.1 and 3.3–3.4).

2.4 Proximate composition of the flours and pasta formulations

The proximate composition of the flours and pasta samples was determined using official AOAC methods (Horowitz et al., 2006). Moisture content was assessed according to method no. 925.09, ash content by method no. 923.03, and lipid content by method no. 920.85. Protein content was calculated using a nitrogen-to-protein conversion factor of 6.25 (method no. 920.87). Dietary fiber content was determined using the enzymatic gravimetric method (method no. 985.29) with a commercial kit (Merck, Sigma-Aldrich, Germany). Total carbohydrate content was estimated by difference, 100 minus the sum of moisture, ash, protein, lipid and dietary fiber contents (g/100 g); therefore, the carbohydrate values reported exclude dietary fiber (available carbohydrates).

2.5 Total phenolic compounds and antioxidant activity of the flours and pasta formulations

For the analysis of total phenolic compounds and antioxidant activity, extracts were prepared by mixing 1 g of sample with 20 mL of 80% ethanol, followed by agitation in an ultrasonic bath (Unique USC 1400) at room temperature for 3 hours. The mixture was then centrifuged (CELM, Combate, Barueri, São Paulo, Brazil) at 1,000 ×g for 20 minutes, and the supernatant was collected. This extraction procedure was repeated twice by adding an additional 5 mL of 80% ethanol each time, following the protocol of Bolanho et al. (2014), with minor modifications.

Total phenolic content (TPC) was quantified using the Folin-Ciocalteu reagent

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(1N). After 60 min of reaction in the dark at room temperature, absorbance was measured at 725 nm using a spectrophotometer (UVmini-1240, Shimadzu, Japan). A standard curve was constructed using gallic acid, and results were expressed as mg of gallic acid equivalents per 100 g of sample (mg GAE/100 g), within a calibration range of 0 – 100 µg/mL (Singleton et al., 1999).

Antioxidant activity was evaluated using the DPPH (2,2-diphenyl-1-picrylhydrazyl) radical scavenging assay, according to Brand-Williams et al. (1995). The extracts were mixed with a 1.0 mM ethanolic DPPH solution at a 1:1 (v/v) ratio and kept for 60 min at room temperature in the dark, and absorbance was recorded at 517 nm. Results were expressed as mg of Trolox equivalents per gram of sample (mg TE/g), based on a standard curve constructed with Trolox (160 – 1,600 µmol/L).

The ferric reducing antioxidant power (FRAP) assay was conducted following the method described by Benzie and Strain (1996). The FRAP reagent was prepared using 10 mM TPTZ (2,4,6-tris(2-pyridyl)-s-triazine), 20 mM $FeCl_3$, and 0.3 M acetate buffer at pH 3.6. A reaction mixture containing 90 µL of extract or Trolox standard solution (160 – 1,600 µmol/L) or blank, 2.7 mL of FRAP reagent, and 270 µL of distilled water was kept for 60 min at room temperature in the dark, and absorbance was measured at 595 nm. Results were expressed as mg TE/g of sample.

The ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid)) radical scavenging activity was assessed following the protocol of Re et al. (1999). The ABTS radical was generated by combining 10 mL of 7 mM ABTS stock solution with 0.176 mL of 140 mM potassium persulfate and allowing the mixture to react for 12 h in the dark at room temperature. The working solution was adjusted to an absorbance of 0.660 at 734 nm. A 30 µL aliquot of extract was mixed with 3 mL of the working solution in the dark. Absorbance was measured at 734 nm using a spectrophotometer (UVmini 1240, Shimadzu). Results were expressed as mg of Trolox equivalents per gram of sample (mg TE/g), using a Trolox calibration curve.

2.6 Cooking properties of the pasta

Optimal cooking time (OCT), water absorption (WA), and cooking loss (CL) were determined by immersing 10 g of pasta in 400 mL of boiling water. At 30-second

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intervals, a portion of the pasta was removed to check for the disappearance of the white core, which indicates complete starch gelatinization and thus the optimal cooking point (AACC, 2000).

Water absorption was calculated as the percentage increase in weight after cooking, based on the difference between the cooked and uncooked sample weights, relative to the initial uncooked weight. For cooking loss, the residual cooking water was collected and dried at 105 °C in a Petri dish until a constant weight was achieved. The mass of the dried solids was then expressed as a percentage of the initial uncooked pasta weight (Palavecino et al., 2017).

2.7 Texture analysis of the pasta

The texture profile of the cooked pasta was assessed using a texture analyzer (Model TA-XTplus, Stable Micro Systems, UK) equipped with a P36/R cylindrical probe. The device was calibrated prior to use. The analysis was performed in compression mode with a pre-set force, test and post-test speeds of 2 mm/s, and a deformation of 75%. Pasta samples were cooked to their optimal cooking time and allowed to cool at room temperature for one minute before analysis. Texture parameters (hardness, N; adhesiveness, N·s; elasticity (springiness), dimensionless; chewiness, N) were recorded directly by the texture analyzer software (Arribas et al., 2020).

2.8 Color analysis of the pasta

Colorimetric properties of the pasta were measured using a digital colorimeter (Miniscan EZ, Hunterlab®, USA). The following parameters were evaluated: L* (lightness, ranging from 0 = black to 100 = white), a* (red-green axis; positive values indicate redness, negative values indicate greenness), and b* (yellow-blue axis; positive values indicate yellowness, negative values indicate blueness).

2.9 Data analysis

Formulations 1 to 8 were analyzed in three analytical replicates of the same sample, whereas formulations 9 and 10 are replicates of the same dough formulation

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(central point). Results are expressed as mean ± standard deviation. Because the replicates of formulations 1 to 8 are analytical and not independent production batches, they were not treated as independent experimental replicates, and the comparisons among formulations by one-way analysis of variance (ANOVA) followed by Fisher's least significant difference (LSD) test ($p < 0.05$) reflect analytical variability and should be interpreted accordingly. The effects of the independent variables (coded as −1, 0 and +1) were evaluated by multiple linear regression of the treatment means (n = 10: eight factorial and two central points), fitting first-order models in Statistica 5.0 (StatSoft, USA). The two-factor interaction terms (x_1x_2, x_1x_3 and x_2x_3) were also tested and, since none was significant ($p > 0.05$) for any of the responses, they were not retained in the models. The central points were used to check for curvature. Because a 2^3 design with central points does not allow the individual quadratic terms to be estimated, the models describe only linear trends within the experimental domain; the response surfaces were generated to visualize these effects and not to optimize the formulation.

3. Results and Discussion

3.1 Flours Chemical Composition

The chemical composition of plant-based ingredients can vary considerably depending on varietal differences, hybridization, geographic origin, and environmental cultivation conditions. Table 2 presents the proximate composition, total phenolic content, and antioxidant activity of sorghum flour (SF), peach palm by-product flour (PF), and cassava by-product flour (CF). Moisture content is a critical parameter in flour characterization, as it influences both microbial stability and the rate of chemical reactions. All flours analyzed presented moisture levels within acceptable regulatory limits. SF exhibited the highest moisture content, followed by CF, while PF presented the lowest value.

Regarding ash content, SF contained 1.91 g/100 g, whereas PF had a notably higher ash content (3.97 g/100 g) and CF exhibited 1.20 g/100 g. The protein (1.90 g/100 g) and lipid (0.32 g/100 g) contents of CF were close to the ranges typically

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reported for cassava bran, 1.42–1.82 g/100 g protein and 0.31–0.43 g/100 g lipids (Ronko et al., 2020). SF presented 6.19 g/100 g protein and 5.54 g/100 g lipids. In contrast, PF presented lower protein (4.87 g/100 g) and lipid (1.35 g/100 g) contents than those reported by Danesi et al. (2018) for peach palm by-product flour (10.4 and 3.7 g/100 g, respectively), which may be related to differences in the portion of the stem used, in the raw material and in the processing. The flours derived from peach palm and cassava by-products stood out for their dietary fiber content (49.97 and 47.11 g/100 g, respectively, versus 9.42 g/100 g in SF). Their inclusion in food formulations may enhance dietary fiber intake while enabling partial substitution of wheat flour in pasta products, thus contributing to improved nutritional profiles.

Sorghum flour also presented a notable concentration of phenolic compounds (136.25 mg GAE/100 g), consistent with the range reported by Rao et al. (2018), which varied from 24 to 358 mg GAE/100 g across different sorghum genotypes. These phenolics include phenolic acids and flavonoids, particularly proanthocyanidins. PF presented a lower TPC (117.57 mg GAE/100 g) than the 233 mg GAE/100 g reported by Bolanho et al. (2014). In terms of antioxidant capacity, SF exhibited values of 1.84 mg TE/g (DPPH assay) and 3.07 mg TE/g (FRAP assay), which are higher than those reported for white sorghum varieties (0.33 and 2.31 mg TE/g, respectively) by Rao et al. (2018). For PF, the DPPH value (1.34 mg TE/g) was lower and the FRAP value (2.83 mg TE/g) was higher than those reported by Bolanho et al. (2014) (3.74 and 2.14 mg TE/g, respectively), differences that may be related to the raw material and to the extraction conditions.

Regarding the safety of the cassava by-product, cassava roots contain cyanogenic glycosides (mainly linamarin), which are substantially reduced by grating, pressing and drying during processing. The CF used here came from sweet cassava, which generally has low cyanogenic content, and was supplied already dehydrated from raw material declared by the manufacturer to be safe for consumption; it represented at most 15% of the formulations. Nevertheless, residual cyanogenic compounds (total cyanide) were not quantified in this study, which is a limitation. Before any application, the cyanide content of each lot of cassava by-product should be determined and compared with the limits established

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by the applicable food regulations.

3.2 Regression Coefficients and Statistical Analysis

Multiple linear regression analyses were conducted using the coded values of the independent variables. The regression coefficients and analysis of variance (ANOVA) for the dependent variables evaluated in the pasta formulations are presented in Table 3.

Table 3. Regression coefficients of the first-order models (coded variables), p-values of the coefficients (in parentheses), coefficient of determination (R^2) and overall p-value of the model (ANOVA) for the gluten-free pasta formulations

	Dietary fiber (g/100 g)	TPC* (mg GAE/100 g)	AA* (mg TE/g)	CL* (%)	Hardness (N)
Intercept	19.007	178.504	0.831	9.746	44.887
x_1	2.365 (0.017)	18.159 (0.035)	0.065 (0.008)	1.650 (0.047)	0.645 (0.756)
x_2	1.135 (0.168)	13.014 (0.100)	0.030 (0.121)	1.770 (0.036)	4.568 (0.061)
x_3	1.290 (0.125)	11.549 (0.135)	0.045 (0.035)	0.610 (0.391)	2.588 (0.240)
R^2	0.731	0.702	0.811	0.704	0.543
p (model)	0.038	0.051	0.014	0.050	0.169

x_1, x_2 and x_3 correspond to the coded values (−1, 0, +1) of PF, SF and CF, respectively. The models were fitted to the treatment means in Tables 2 and 4 (n = 10; 6 residual degrees of freedom), and the coefficients are expressed in the same units as the responses.

*TPC = total phenolic compounds; AA = antioxidant activity measured by the DPPH assay; CL = cooking loss. Numbers in parentheses indicate p-values; coefficients in red are statistically significant ($p < 0.05$).

The responses were fitted to first-order polynomial models. The two-factor interactions (x_1x_2, x_1x_3 and x_2x_3) were also tested but were not significant for any response ($p \geq 0.31$) and were therefore excluded from the models. The central points presented higher values than the mean of the factorial points for all responses (e.g.,

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hardness: 52.8 versus 42.9 N), which indicates curvature; the first-order models thus describe only the linear trend within the studied domain, and the quadratic terms cannot be individually estimated with this design. According to the global ANOVA (Table 3), the models for dietary fiber ($p = 0.038$) and for antioxidant activity by DPPH ($p = 0.014$) were statistically significant at the 5% level, the models for TPC ($p = 0.051$) and cooking loss ($p = 0.050$) were borderline, and the model for hardness was not significant ($p = 0.169$). Consequently, the TPC, CL and hardness equations must be regarded as exploratory trends rather than predictive models.

In Table 3, the significant linear terms ($p \leq 0.05$) were: PF for dietary fiber ($p = 0.017$), TPC ($p = 0.035$), AA by DPPH ($p = 0.008$) and CL ($p = 0.047$); SF for CL ($p = 0.036$); and CF for AA by DPPH ($p = 0.035$), as illustrated in the response surface plots (Figure 2). No linear term was significant for hardness (SF: $p = 0.061$). A positive coefficient indicates that the response increases with the proportion of the corresponding flour. This is nutritionally favorable for dietary fiber, TPC and AA but undesirable for CL, since a higher loss of solids during cooking indicates lower cooking quality; the increase of CL with PF and SF is therefore a technological drawback that offsets part of the nutritional gain obtained with these flours.

Figure 2. Response surface plots (first-order models) showing the effects of peach palm by-product flour (x_1), sorghum flour (x_2) and cassava by-product flour (x_3), in coded values, on dietary fiber (A, B), total phenolic compounds (TPC; C, D), antioxidant activity by DPPH (AA; E, F), cooking loss (CL; G, H) and hardness (I, J) of gluten-free pasta. In each panel, the third variable is fixed at its central level (0)

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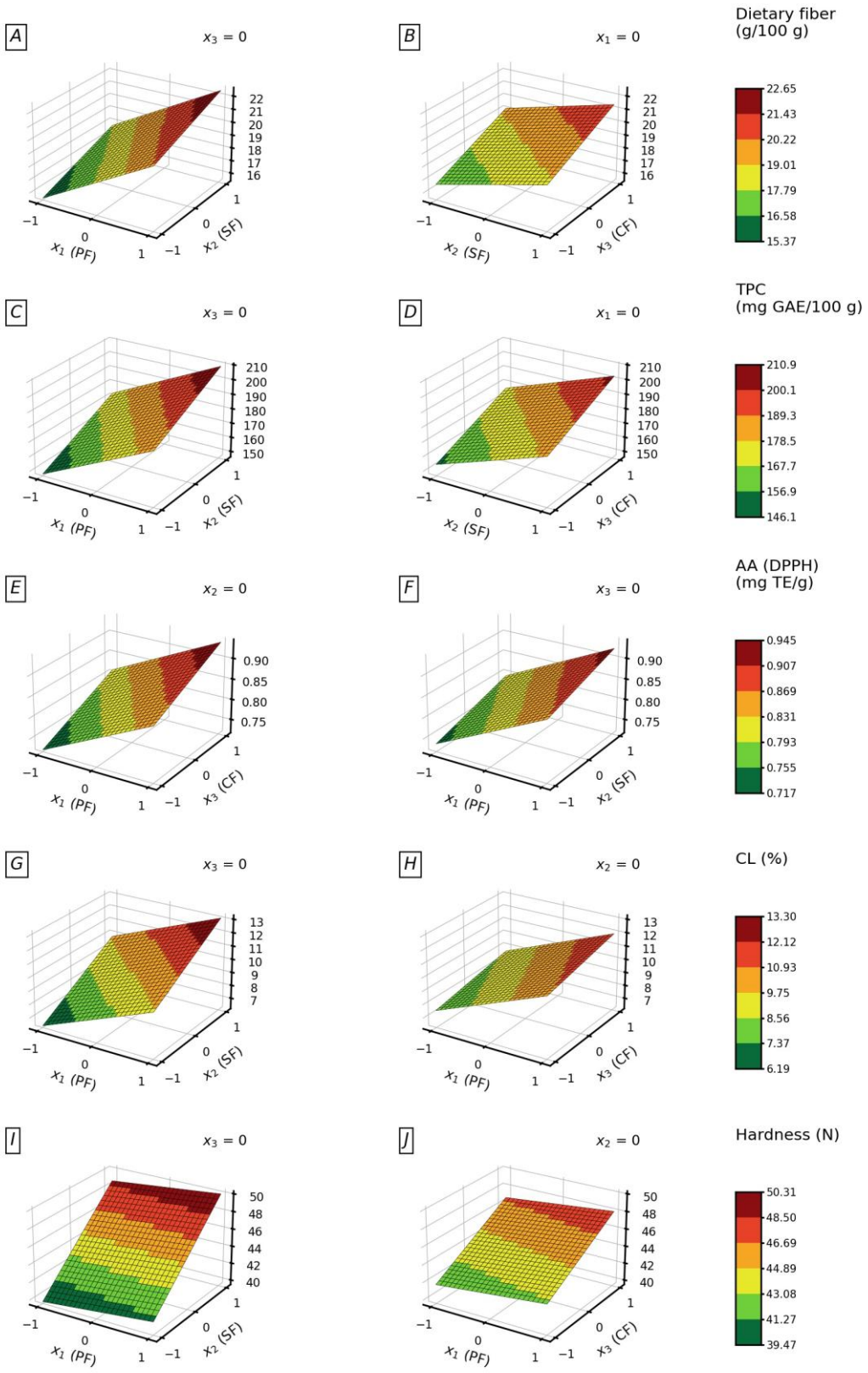

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3.3 Pasta Chemical Composition

The proximate composition and bioactive compound content of the gluten-free pasta formulations are presented in Table 2. Moisture content is a key parameter in determining the product's shelf stability. The values observed in this study are consistent with those reported by Bolarinwa and Oyesiji (2021), who found moisture levels ranging from 10.02% to 11.34% in pasta made from rice, soybean, and cassava starch.

Lipid content ranged from 3.83 to 5.32 g/100 g, with significant differences among formulations ($p < 0.05$). Higher lipid values were associated with higher proportions of SF (samples 3, 4, 7, 8, 9 and 10), reflecting the higher lipid content of SF (5.54 g/100 g) compared with PF (1.35 g/100 g) and CF (0.32 g/100 g). Carbohydrate content (calculated by difference and excluding dietary fiber) tended to be higher in formulations with more rice flour, attributable to its high starch content.

Protein content ranged from 6.94 to 8.29 g/100 g, with no significant differences among formulations ($p > 0.05$). Similar values were reported by Paiva et al. (2019) for gluten-free pasta based on corn (6.22 g/100 g) and sorghum flour (9.57 g/100 g).

Significant increases in dietary fiber content were observed in formulations with higher proportions of PF ($p = 0.017$) and, to a lesser extent, CF and SF (non-significant linear effects, Table 3), reaching a maximum value of 24.32 g/100 g in formulation 8, in which rice flour was entirely replaced by SF, PF, and CF. Fiber levels above 20 g/100 g were obtained only in formulations containing at least 10% PF (samples 4, 6, 8, 9 and 10), highlighting the influence of PF as confirmed by multivariate regression (Table 3). The observed fiber contents surpassed those reported in earlier studies on gluten-free pasta, including 1.32 g/100 g in soy-enriched pasta (Bolarinwa and Oyesiji, 2021) and 8.20 g/100 g in sorghum-based pasta (Paiva et al., 2019). This result is particularly relevant considering that conventional gluten-free products are frequently formulated with refined rice and corn flours or starches, which are poor sources of dietary fiber (Melini and Melini, 2019). Therefore, the high fiber content achieved in the present study, particularly

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in formulations containing higher proportions of PF and CF, may contribute to addressing one of the recognized nutritional limitations of gluten-free diets.

Figure 2 displays the response surface plots. Figure 2A shows the effects of PF (x_1) and SF (x_2) on dietary fiber content with CF (x_3) at the central level (10%). The highest predicted values (about 22.5 g/100 g) occurred at high x_1 and x_2 (+1); the experimental maximum (24.32 g/100 g, formulation 8) was obtained with the three flours at their highest levels. Figure 2B illustrates the combined effects of x_2 and x_3 with PF at the central level (10%), also showing higher fiber values at high concentrations of both flours, which is consistent with the high dietary fiber content of CF (47.11 g/100 g, Table 2). PF was identified as the most influential factor in the regression model (Equation 1):

$$\text{Dietary fiber} = 19.007 + 2.365x_1 + 1.135x_2 + 1.290x_3 \quad \text{(Equation 1)}$$

Total phenolic content (TPC) ranged from 122.54 to 211.56 mg GAE/100 g, consistent with values reported for multigrain pasta (Kamble et al., 2021). Figure 2C shows the influence of SF (x_2) and PF (x_1) on TPC, with CF fixed at the central level (10%). The highest TPC was observed when both SF and PF were at their highest levels (+1). Figure 2D shows the contribution of SF (x_2) and CF (x_3) with PF fixed at 10%. The TPC model (Equation 2) was borderline ($p = 0.051$) and only the coefficient of PF was significant (Table 3). The coefficients of PF and CF were larger, and that of SF smaller, than expected from the TPC of the flours in Table 2 assuming additive contributions and a negligible TPC of rice flour (PF: 117.57; SF: 136.25; CF: 35.78 mg GAE/100 g), which suggests that other factors, such as the release of phenolic compounds bound to the fiber matrix or changes during processing, may have contributed; this hypothesis should be tested in further studies.

$$\text{TPC} = 178.504 + 18.159x_1 + 13.014x_2 + 11.549x_3 \quad \text{(Equation 2)}$$

Antioxidant activity was measured using three methodologies: ABTS, FRAP,

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and DPPH. The formulations with the highest proportions of the by-product flours (samples 6 and 8) showed the highest AA values, with ABTS producing the highest values, followed by FRAP and DPPH. A similar trend was reported by Szydłowska-Tutaj et al. (2023) in mushroom-fortified pasta, where ABTS values ranged from 0.68 to 0.90 mg TE/g in alcoholic extracts, while DPPH values ranged from 0.04 to 0.43 mg TE/g. Regarding the effects of the different flours on the pasta, Figure 2E displays the effects of PF (x_1) and CF (x_3) on AA measured by DPPH, the assay used for the regression model in Table 3. Both variables had a significant positive effect ($p \leq 0.05$), whereas SF did not show a statistically significant effect (Figure 2F), although a positive trend was still evident. The regression model for AA by DPPH (mg TE/g) was (Equation 3):

$$AA\ (DPPH) = 0.831 + 0.065x_1 + 0.030x_2 + 0.045x_3 \quad \text{(Equation 3)}$$

3.4 Technological properties of the pasta

The results of technological properties are presented in Table 4.

Color is a fundamental parameter for product acceptance, particularly in dry and cooked pasta. The color of foods without additives depends primarily on the composition of the materials used in production, such as proteins, dietary fiber, phenols, and other compounds (Arribas et al. 2020). Significant differences ($p < 0.05$) were observed among some samples. Pastas with higher levels of PF and CF (samples 2, 5, and 6) showed lighter color, while those with higher sorghum flour (SF) levels (samples 3, 7, and 8) exhibited lower luminosity. The inclusion of different flours influenced the pasta's color pattern, with SF increasing the value of the a* parameter (red tones), while PF and CF contributed to higher yellow tones (b*) and luminosity.

The technological properties of pasta were determined through the following parameters: optimal cooking time (min), water absorption index (%), and cooking loss (%) (Table 4). Long cooking times and high solid losses are undesirable. Extended cooking times indicate higher energy consumption, while significant cooking loss during cooking suggests increased starch solubility, leading to

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structural deformation and lower product yield (Bolarinwa and Oyesiji, 2021).

The optimal cooking time is defined as the duration necessary to achieve complete starch gelatinization (Sissons et al. 2022). For the pastas produced, cooking time increased with greater incorporation of SF, PF, and CF, ranging from 5.0 to 7.65 min. These values are lower than those found by Espinosa-Solis et al. (2019), who reported 7.10 to 8.50 min for semolina pasta fortified with oat and apple flours, with the shortest time observed for pure wheat semolina pasta. This suggests that cooking time was not adversely affected by SF, PF and CF, although these comparisons involve different pasta matrices.

Weight gain or water absorption index is associated with the high content of non-starchy polysaccharides in the composition. It is also influenced by the starch granules' structure, the presence of more amorphous regions, protein matrix disintegration during cooking, and the pasta's geometric characteristics (Lucisano et al. 2012). The results of samples 2 and 7 showed significant differences from samples 8, 9, and 10, ranging from 93.05% to 111.71%. During cooking, water absorption must occur so that the cooked pasta's weight exceeds twice that of the raw product, resulting in absorption index values close to 100%, as observed in these samples (Brites et al. 2018).

Cooking loss (CL) is determined by measuring the amount of solids released into the cooking water (AACC, 2000). Reduced solid loss during cooking indicates better pasta quality, reflecting its efficiency in retaining starch granules and bioactive compounds throughout the cooking process (Nilusha et al. 2019). Usually, pasta with added fiber exhibits higher solid loss compared to those made with refined wheat flour (Bianchi et al. 2021). This behavior could also be observed in this study, since pastas with higher fiber content tended to show greater solid loss, varying from 5.54% to 15.39%. A similar effect was observed by Khatri et al. (2023), who reported higher solid loss in pasta fortified with quinoa and chia flours (8.72% to 14.34%) compared to control pasta made with semolina (7.34%).

The effect of SF and PF on cooking loss (CL) in pasta with CF fixed at the central level (10%) is illustrated in Figure 2G. The highest CL values, both predicted by the model (about 13%) and observed (15.39%, formulation 8), occurred with high

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PF (15%) combined with high SF (60%). This behavior may be attributed to the high fiber content of PF, which is associated with the release of soluble compounds from within the pasta structure. Rodrigues et al. (2023) reported a similar trend in gluten-containing pasta enriched with palm flour (5%, 10%, and 15%), with solid loss values of 4.0%, 7.6%, and 9.1%, respectively. Equation 4 represents the linear effects of the flours on CL (the model was borderline, p = 0.050, Table 3):

$$CL = 9.746 + 1.650x_1 + 1.770x_2 + 0.610x_3 \text{ (Equation 4)}$$

On the other hand, the effect of PF and CF on CL with SF fixed at the central level (45%) is shown in Figure 2H. With SF fixed, CL increased mainly with PF (x_1), whereas CF (x_3) had little influence, as supported by the low regression coefficient for CF (0.61, Table 3 and Equation 4) and its non-significant level (p = 0.391, Table 3). Therefore, within the range studied, CF did not significantly influence cooking loss. Taken together, these results show that the formulation with the greatest nutritional enrichment (sample 8) also had the highest loss of solids during cooking, the nutritional gain obtained with higher proportions of PF and SF came with a technological cost, a trade-off that must be considered when selecting a formulation.

According to Table 4, hardness tended to increase with the proportion of SF (formulations 3, 4, 7 and 8 versus 1, 2, 5 and 6), but the highest values were observed at the central points (52.78 and 52.89 N), above those of the factorial points (31.75 to 48.68 N), which indicates curvature that a first-order model cannot describe. The effect of SF and PF is shown in Figure 2I, with CF fixed at the central level (10%): higher predicted hardness values were observed with high SF, regardless of PF levels. In Figure 2J (SF at the central level, 45%), CF showed a positive trend with hardness, less pronounced than that of SF. Equation 5 presents the coefficients for the hardness response; however, this model was not statistically significant (p = 0.169, and no coefficient with $p \leq 0.05$, Table 3) and must therefore be regarded only as a descriptive trend:

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$$\text{Hardness} = 44.887 + 0.645x_1 + 4.568x_2 + 2.588x_3 \quad \text{(Equation 5)}$$

The hardness values ranged from 31.75 to 52.89 N, showing significant differences among samples. These results are similar to those observed in gluten-free pasta made with different proportions of rice and bean flours, which ranged from 35.28 to 70.36 N for 100% rice and 100% bean flours, respectively, and 31.26 N for commercial pasta (Arribas et al., 2020). Elasticity and adhesiveness did not differ significantly ($p > 0.05$) between samples. Adhesiveness was negative for all formulations (−0.45 to −1.13 N·s), as expected for the negative area of the force-time curve; Arribas et al. (2020) reported negative adhesiveness values up to −0.45 N·s, indicating resistance of the pasta to its contact surface. Elasticity (springiness, dimensionless) results were below those observed in wheat-based spaghetti (1.10) and in spaghetti enriched with apple and oat flours (1.24 and 1.16, respectively) (Espinosa-Solis et al., 2019). Similarly, chewiness (the energy required to chew a solid food until it is ready for swallowing) ranged from 6.66 to 11.62 N, which were comparable to the average value of 8.74 N reported by Palavecino et al. (2017) for gluten-free pasta made with sorghum.

4. Conclusion

The combination of sorghum flour with peach palm and cassava by-product flours proved technically feasible for producing gluten-free pasta with markedly improved nutritional characteristics.

Dietary fiber reached up to 24.32 g/100 g, well above the values reported for other gluten-free pasta in the literature, and was significantly increased by the proportion of peach palm by-product flour. Total phenolic content (up to 211.56 mg GAE/100 g) and in *vitro* antioxidant activity also increased with the incorporation of these flours, while protein content did not differ among formulations.

The pasta kept an optimal cooking time of 5.0 to 7.65 min, water absorption close to 100% and a light, yellowish color, and formulation 8 (15% PF, 60% SF, 15% CF, without rice flour) showed the greatest nutritional enrichment among those

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evaluated. In addition, the use of agro-industrial by-products adds value to residues of the heart-of-palm and cassava starch chains, supporting more sustainable food formulations.

As points to be addressed in future work, the highest enrichment was accompanied by higher cooking loss (15.39%), the models for total phenolics, cooking loss and hardness were borderline or not significant, and designs that allow quadratic terms are needed for formal optimization. Sensory evaluation with consumers and quantification of residual cyanogenic compounds in the cassava by-product are also recommended.

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Table 2. Proximate composition, total phenolic content (TPC) and antioxidant activity (AA) of the flours and gluten-free pasta formulations

Analysis (units)	Moisture (g/100 g)	Ash (g/100 g)	Protein (g/100 g)	Lipids (g/100 g)	Dietary fiber (g/100 g)	Carbohydrates (g/100 g)	TPC* (mg GAE/100 g)	AA ABTS* (mg TE/g)	AA DPPH* (mg TE/g)	AA FRAP* (mg TE/g)
PF	4.66 ± 0.14	3.97 ± 0.09	4.87 ± 0.26	1.35 ± 0.02	49.97 ± 0.31	35.18	117.57 ± 2.03	-	1.34 ± 0.04	2.83 ± 0.02
SF	7.44 ± 0.04	1.91 ± 0.05	6.19 ± 0.06	5.54 ± 0.16	9.42 ± 0.93	69.50	136.25 ± 3.74	-	1.84 ± 0.01	3.07 ± 0.06
CF	5.48 ± 0.42	1.20 ± 0.09	1.90 ± 0.03	0.32 ± 0.14	47.11 ± 0.67	43.99	35.78 ± 0.96	-	0.55 ± 0.00	0.36 ± 0.01
Sample 1	10.44^{AB}±0.03	1.91^{F}±0.01	8.05^{A} ±0.01	3.87^{B} ±0.09	15.42^{D} ±0.41	60.31	122.54^{E}±2.51	2.18^{D}±0.19	0.63^{E}±0.01	1.89^{E}±0.04
Sample 2	10.98^{A} ±0.06	2.46^{C} ±0.01	7.44^{A} ±0.09	4.07^{B} ±0.21	17.44^{C} ±0.07	57.61	162.29^{CD}±1.51	2.46^{D}±0.05	0.82^{BCD}±0.02	2.31^{CD}±0.03
Sample 3	10.12^{AB} ±0.06	2.14^{E} ±0.03	8.10^{A} ±0.16	4.72^{B} ±0.08	15.00^{D} ±0.39	59.92	157.15^{D}±0.25	2.99^{C}±0.08	0.80^{CD}±0.03	2.32^{CD}±0.10
Sample 4	10.04^{B} ±0.19	2.69^{B} ±0.01	8.29^{A} ±0.02	5.32^{A} ±0.05	20.34^{B} ±0.09	53.32	198.49^{AB}±4.52	3.40^{A}±0.05	0.85^{BCD}±0.02	2.50^{ABC}±0.08
Sample 5	10.01^{B} ±0.22	2.32^{D} ±0.01	7.46^{A} ±0.09	3.83^{C} ±0.05	15.24^{D} ±0.04	61.14	162.82^{CD}±3.26	3.01^{C}±0.15	0.78^{D}±0.01	2.08^{DE}±0.01
Sample 6	11.33^{A} ±0.75	2.66^{B} ±0.02	7.18^{A} ±0.35	4.08^{B} ±0.01	20.72^{B} ±0.11	54.03	186.96^{B}±2.28	3.32^{AB}±0.05	0.93^{A}±0.03	2.67^{AB}±0.09
Sample 7	10.23^{AB}±0.01	2.35^{D} ± 0.04	7.22^{A} ±0.05	4.55^{B} ±0.18	18.24^{C} ±0.22	57.41	171.52^{C}±1.51	3.17^{BC}±0.07	0.81^{BCD}±0.03	2.45^{BC}±0.06
Sample 8	10.34^{AB}±0.38	2.91^{A} ±0.01	6.94^{A} ±1.02	5.24^{A} ±0.16	24.32^{A} ±0.90	50.25	211.56^{A}±1.04	3.42^{A}±0.05	0.94^{A}±0.01	2.77^{A}±0.07
Sample 9	10.49^{AB} ±0.06	2.71^{B} ±0.01	7.98^{A} ±0.17	5.25^{A} ±0.01	21.37^{B} ±0.01	52.20	205.23^{A}±3.52	3.10^{C}±0.07	0.88^{AB}±0.03	2.47^{BC}±0.10
Sample 10	10.49^{AB} ±0.52	2.78^{AB}±0.02	7.99^{A} ±0.06	5.29^{A} ±0.04	21.98^{B}±0.75	51.47	206.48^{A}±2.76	3.12^{C}±0.06	0.87^{ABC}±0.01	2.52^{ABC}±0.01

PF = peach palm by-product flour; SF = sorghum flour; CF = cassava by-product flour; TPC = total phenolic compounds; GAE = gallic acid equivalents; AA = antioxidant activity; TE = Trolox equivalents; − = not determined. Carbohydrates were calculated by difference (100 − moisture − ash − protein − lipids − dietary fiber) and therefore exclude dietary fiber. Means followed by the same uppercase letter within a column do not differ significantly ($p > 0.05$). Formulations (PF, SF, CF; %): 1 (5, 30, 5); 2 (15, 30, 5); 3 (5, 60, 5); 4 (15, 60, 5); 5 (5, 30, 15); 6 (15, 30, 15); 7 (5, 60, 15); 8 (15, 60, 15); 9 and 10 (10, 45, 10).

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Table 4. Technological characteristics of the pasta formulations

	COLOR			COOKING PROPERTIES			TEXTURE ANALYSIS			
	L*	a*	b*	OCT (min)	WA (%)	CL (%)	Hardness (N)	Adhesiveness (N.s)	Elasticity (dimensionless)	Chewiness (N)
1	74.01^{BC} ±1.60	3.72^{BC} ±0.17	16.46^{AB} ±0.58	5.0^{C} ±0.0	98.43^{AB}±1.09	5.54^{E}±1.59	31.75^{D} ±2.84	-0.45^{A} ±0.20	0.54^{A} ±0.15	6.66^{D}±1.18
2	78.66^{A} ±0.37	3.23^{D} ±0.09	18.21^{A} ±0.04	6.25^{BC}±0.32	111.71^{A}±1.37	7.77^{DE}±0.46	36.33^{CD} ±3.36	-0.88^{A} ±0.17	0.74^{A} ±0.26	7.27^{CD}±3.03
3	74.38^{BC} ±0.81	4.02^{AB} ±0.07	17.07^{AB} ±0.04	7.0^{AB} ±0.11	102.11^{AB}±5.57	9.93^{BC}±0.34	46.50^{AB} ±1.78	-1.13^{A} ±0.27	0.64^{A} ±0.13	9.03^{BCD}±1.40
4	75.00^{B} ±0.85	4.12^{AB} ±0.11	17.93^{A} ±0.28	7.2^{AB}±0.28	100.13^{AB}±4.50	11.93^{B}±1.63	46.67^{AB} ±3.51	-0.77^{A} ±0.24	0.51^{A} ±0.13	7.76^{BCD}±1.85
5	77.93^{A} ±1.06	3.41^{CD} ±0.19	17.47^{A} ±0.32	6.72^{AB} ±0.81	105.33^{AB}±2.58	8.10^{CDE}±1.15	42.75^{BC} ±2.06	-0.94^{A} ±0.18	0.70^{A} ±0.07	11.07^{AB}±1.79
6	78.56^{A} ±1.44	3.46^{CD} ±0.25	18.39^{A} ±0.54	6.3^{ABC}±1.14	107.09^{AB}±2.50	9.12^{BCD}±0.67	42.50^{BC} ±5.29	-0.84^{A} ±0.30	0.64^{A} ±0.19	8.76^{BC}±3.14
7	74.23^{BC} ±0.36	4.22^{A} ±0.04	18.90^{A} ±0.17	6.8^{AB}±0.71	109.51^{A}±3.64	7.44^{DE}±1.10	48.02^{AB}±3.08	-1.01^{A} ±0.22	0.62^{A} ±0.02	8.42^{CD}±0.55
8	72.74^{C} ±0.28	4.32^{A} ±0.05	17.70^{B} ±0.16	7.65^{A}±0.50	93.05^{B}±1.08	15.39^{A}±1.83	48.68^{AB} ±1.61	-0.93^{A} ±0.26	0.53^{A} ±0.06	8.34^{CD}±0.262
9	75.54^{B} ±1.26	4.13^{AB} ±0.50	16.19^{AB} ±0.43	7.15^{AB}±0.21	94.52^{B}±1.78	11.50^{B}±1.17	52.78^{A} ±2.08	-1.01^{A} ±0.23	0.64^{A} ±0.03	11.01^{AB}±0.62
10	75.19^{B} ±0.84	4.04^{AB} ±0.42	16.99^{AB} ±0.41	7.15^{AB}±0.21	94.01^{B}±2.64	10.74^{BC}±0.12	52.89^{A} ±1.72	-1.05^{A} ±0.35	0.62^{A} ±0.1	11.62^{A}±1.19

L* = lightness (L* = 0, black; L* = 100, white); a* and b* are chromatic coordinates (+a* red and −a* green; +b* yellow and −b* blue).
OCT = optimal cooking time; WA = water absorption; CL = cooking loss. Elasticity is dimensionless.
Means followed by the same uppercase letter within a column do not differ significantly ($p > 0.05$).
Formulations (PF, SF, CF; %): 1 (5, 30, 5); 2 (15, 30, 5); 3 (5, 60, 5); 4 (15, 60, 5); 5 (5, 30, 15); 6 (15, 30, 15); 7 (5, 60, 15); 8 (15, 60, 15); 9 and 10 (10, 45, 10).