



EUROPEAN FOOD Science and Engineering (EFSE)

Volume: 5

Number: 1

Date: 2024

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## **European Food Science and Engineering**

Eur Food Sci Eng 2024, 5 (1), 1-7 doi: 10.55147/efse.1443464 https://dergipark.org.tr/tr/pub/efse

# Potential use of hazelnut (*Corylus avellana* L.) shell in muffin production by substitution of wheat flour: Color, bioactive, textural, and sensory properties

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#### ARTICLE INFO

Research Article Article History: Received: 27 February 2024 Accepted: 10 May 2024 Available Online: 30 June 2024 Keywords: Muffin Hazelnut Shell Functional ingredient By-products Textural changes Sensory evaluation

#### ABSTRACT

Herein, three muffin samples were produced by substituting 0, 5, and 10% (w:w) hazelnut shell (HS) into wheat flour (WF) and their color, bioactive, textural and sensory properties were determined. The results showed that both total phenolic content and DPPH (2.2-diphenyl-1-picrylhydrazyl) radical scavenging activity were not significantly affected by the addition of HS to the muffin formulation (P>0.05). Upon enrichment of muffins with HS, L\* (70.00-44.79 for crumb, 51.53-44.00 for crust) and  $b^*$  values of muffins (27.39-12.49 for crumb, 30.65-17.17 for crust) significantly decreased in both crumb and crust, while  $a^*$  values significantly increased in crumb (-0.26-6.97) and decreased in crust (8.39-14.30) (P<0.05). Textural analysis revealed that hardness (9.74-32.14 N), gumminess (7.17-14.83 N), and chewiness (6.40-12.55 N) significantly decreased (P<0.05) while the springiness (0.85-0.89%) and resilience insignificantly increased as the amount of HS increased in the muffin formulation (P>0.05). The substitution of WF with 5% (w:w) HS significantly received the highest crust (5.6), crumb (5.1), chewiness (6.0), taste/aroma (6.0) and overall acceptability scores (5.9) by the panelists (P<0.05). Overall, HSs, which are a waste and by-product of hazelnut processing, can be successfully used in functional muffin production, both expanding their potential areas of use and contributing to their economic value.

### 1. Introduction

According to Food and Agriculture Organization (FAO) statistical data in 2007, 1/3 of the edible food in the world is wasted and/or lost, amounting to 1.3 billion tons (Salihoglu et al., 2018). In undeveloped countries, food waste reveals at the initial stages of food production, while in developed countries it occurs mostly at the final stages. In Europe, 42% of total food waste comes from households, 39% from food production and processing facilities, 14% from the service and catering sector and 5% from the wholesale and retail sector. In Europe, consumers throw away 30-68% of the food they buy and the annual amount of food waste per capita reaches 280-300 kg (European Commission 2010; Gustavson et al., 2011; Gustavson et al., 2013). The amount of organic waste generated in the food industry as a result of physical processes such as sorting and peeling applied to fruits and vegetables is quite high. These wastes are remain as waste and usually use as

animal feed. However, latest studies revealed that these wastes are very rich in terms of nutritional value (Mirabella et al., 2014; Galanakis, 2012). For this reason, it has become a valuable, functional ingredient to reuse organic wastes such as fruit peels and seeds and to investigate ways of using them in different ways. It is extremely important to investigate the potential use of valuable and functional components for the reuse of organic waste such as fruit peels and seeds (Goel et al., 2020).

Hazelnut (*Corylus avellana* L.) has an important place in the economy of the Eastern Black Sea Region, which has a monoculture agricultural structure, especially in Trabzon, Giresun and Ordu, Türkiye (Gönenc et al., 2006). Türkiye, a significant producer, accounted for approximately 63% of global hazelnut production from 2016 to 2020, with Italy, Azerbaijan, and other countries following suit (Öztürk, 2023). Hazelnuts can be consumed fresh or roasted as a snack, but are also used in the food, pharmaceutical, and cosmetic industries for the preparation of chocolate, biscuits, confectionery, cakes, ice cream, refined edible oil, cleaning agents, moisturizers and pharmaceuticals (Topkafa, et al., 2015). Hazelnut shell, a waste and by-product of the hazelnut processing industry, accounts for 50% of the total hazelnut weight. Due to the burning of crop residues, their disposal poses an economic threat to producers as well as a significant environmental threat (Esposito et al., 2017). Hazelnut shells mostly consist of lignin (40-50%), hemicellulose (13-32%), and cellulose (16-27%) (Zhao et al., 2023). These lignocelluloses shells were rich in bioactive ingredients such as phenolic acids, flavonoids, tannins, diaryleptanoids, and lignans (Esposito et al., 2017; Di Michele et al., 2021; Zhao et al., 2023).

A worldwide known wheat-based snack, muffins are widely accepted and recognized as a convenient food in today's eating habits and food choices due to their ready-to-eat form (Olawuyi et al., 2019). It has been reported that many food additives are used in industrial cake production to improve cake properties, extend shelf life and prevent differences. Raw material properties and processing conditions used in muffin production have a significant impact on the product quality (Difonzo et al., 2022). Their quality properties are determined by physical and sensory characteristics such as crust and crumb color, height, weight, texture, volume, symmetry (Xu et al., 2020; Difonzo et al., 2022). Thus far, different functional muffin formulations were developed in the literature using different by-products or wastes such as mango pulp fibre waste (Sudha et al., 2015), tomato processing by-product (Mehta et al., 2018), Rosa damascena Mill. by-products and cocoa pod husks (Chochkov et al., 2022), and cocoa bean shell powder (Souza et al., 2022). However, to the best of our knowledge, no studies have been conducted so far on the use of hazelnut shell (HS) in muffin production through substitution of wheat flour and its potential for conversion from food waste to a functional food ingredient. In this context, the objectives of this study were (i) to prepare functional muffin samples containing different proportions of HS (0, 5 and 10%, w:w) to evaluate their crumb and crust color, bioactive, textural, and sensory characteristics.

#### 2. Materials and methods

#### 2.1. Materials

Hazelnut shells (HS) were obtained from Union of Hazelnut Sales Cooperative (FISKOBIRLIK). Other ingredients used in the preparation of the muffins such as sugar (Altınküp, Ankara, Türkiye), ultrahigh temperature (UHT) whole fat milk (Pinar Süt, Izmir, Türkiye), sunflower oil (Yudum oils, Balıkesir, Turkiye) and baking powder (Dr. Oetker, Izmir, Türkiye) were purchased from local markets. As well, all-purpose wheat flour (additive-free, 11.9% protein content, Eris Flour, Samsun, Türkiye) suitable for bread, cake, pie making *etc.* was purchased. Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) (Sigma-Aldrich, Steinheim, Germany), DPPH (Sigma-Aldrich, Germany), methanol (Merck, Germany), Folin-Ciocalteu phenol reagent (Sigma-Aldrich, Steinheim, Germany) were obtained.

#### 2.2. Preparation of HS

The shells underwent a thorough cleaning process using distilled water and scrubbing to remove any potential contaminants, and then subsequently dried using an oven (Memmert UF-110, Germany) at 110 °C for 2 h. Then, HSs were ground with a grinder (Tefal 8100.31 coffee grinder, France). The samples were then sealed and stored at room temperature until used in the muffin production and analysis.

#### 2.3. Muffin production

Muffin production was carried out with some modifications of Topkaya & Işık (2019). The proportions of HS to be incorporated into the muffin samples were determined as a result of preliminary experiments and sensory analysis. Moreover, the flowchart given in Figure 1 was followed for the preparation of muffins. HS was substituted into wheat flour at 0%, 5% and 10% (w:w) ratios and 3 different muffins were produced using the ingredients given in Table 1. For this purpose, eggs and sugar were mixed at high speed for 2 min using a mixer with dough hook (Bosch, MFQ 3030, 350 W). Then, milk and sunflower oil were added and mixed for another 2 min. After that, wheat flour, baking powder, and various proportions of HS were added. It was mixed for another 2 min. Then, muffin batter (12 g each) was transferred into pre-greased muffin cups. They were baked in a preheated oven at 180 °C for 30 min and left at room temperature for 1 h to cool down. Finally, they were named as HS-0, HS-5, and HS-10 at HS ratio of 0, 5, and 10%, respectively.



Figure 1. Flowcharts of muffin production.

**Table 1.** The used recipes for the production of HS-0, HS-5 and HS-10 muffins.

	<b>Muffin Samples</b>					
Ingredients (%, w/w)	HS-0	HS-5	HS-10			
Flour	32.00	30.40	28.80			
Hazelnut shell powder	0.00	1.60	3.20			
Egg	19.20	19.20	19.20			
Sugar	16.00	16.00	16.00			
Oil	16.00	16.00	16.00			
Milk	16.00	16.00	16.00			
Baking powder	0.80	0.80	0.80			

HS-0: The muffin samples without hazelnut shell; HS-5: The muffin samples including 5% substitution of hazelnut shell; HS-10: The muffin samples including 10% substitution of hazelnut shell.

#### 2.4. Color assessment

Both crumb and crust color characteristics of muffins were determined by the method previously reported by Yavuz et al. (2022).  $L^*(L^*=0, \text{black}; L^*=100, \text{white}), a^*(-a^* = \text{greenness}; +a^* = \text{redness})$  and  $b^*$  values (- $b^*$ =blueness; + $b^*$ =yellowness) of muffins for their crumb and crust were quantitatively measured employing a portable colorimeter (CR-400, Minolta Camera Co., Osaka, Japan). For this purpose, crust color values were measured from the tops of the muffins as a whole, while crumb colors were determined by cutting the muffins into two equal parts parallel to the ground using a knife. Total color difference ( $\Delta E^*$ ) was calculated as follows:

$$\Delta E^* = \sqrt{(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2}$$
(1)

#### 2.5. Bioactive properties

#### Preparation of muffin extract

Initially, muffin samples were mixed with 70% methanol in the ratio of 1:10 (w/v) using an Ultra-Turrax dispenser (Daihan, HG-15D, Gang-Won-Do, South Korea) for 1 min and then kept in ultrasonic water bath (Daihan, WUC-D10H, Seoul, South Korea) for 10 min at 40 °C. The mixture was mixed using a magnetic stirrer (IKA C-MAG HS 7, Germany) at 250 rpm for 15 min and then centrifuged (Centrifuge Multifuge X3 FR, Thermo Scientific, Heraeus, Germany) at 4 °C for 20 min at 6000 rpm. Afterwards, the clear supernatant was taken into a beaker, and then 70% methanol was added to the remaining precipitate and centrifuged once more under the same conditions. The collected supernatants were stored at -18 °C until measured on a spectrophotometer (UV-1800, Shimadzu, Kyoto, Japan).

#### Total phenolic content (TPC)

TPC of muffin samples as well as the extract of HS (HS-E) were determined based on the study previously reported by Bakkalbas1 et al. (2015) with minor modifications. To begin with, 0.5 mL of muffin extract were mixed with 2.5 mL Folin Ciocalteu reagent (1:10 diluted with distilled water) and 2 mL of 7.5% Na<sub>2</sub>CO<sub>3</sub> solution. Subsequently, the samples were kept in the dark at room temperature for half an hour. The calibration curve was generated using gallic acid solutions in the concentration range of 5-100 mg/L. Absorbance values were then read at 760 nm. The results were calculated as mg gallic acid equivalent per gram (mg GAE/g).

#### DPPH antioxidant activity

Modified version of DPPH antioxidant activity assay from the study of Bakkalbası et al. (2015) was applied to muffin extracts. Firstly, 0.1 mL of muffin extract and 4.9 mL of DPPH solution were mixed. After the samples were kept in the dark for half an hour, absorbance values were measured at 515 nm wavelength. Antioxidant activity values were calculated as  $\mu$ mol Trolox equivalent (TE)/g sample on dry matter basis.

#### 2.6. Texture profile analysis

The procedure previously reported by Pauter et al. (2018) were applied to determine the textural properties (hardness, springiness, cohesiveness, gumminess, chewiness, resilience) of the muffins using a texture analyser (TPA, Micro Stable

Systems TA.XDplus Texture Analyzer, England). For this aim, fresh muffin samples with 25 mm thickness were placed on to the instrument with a 5 kg load cell using a 35 mm diameter of a cylindrical plunger probe. The analysis were performed under following conditions: strain 40%, test speed: 1.7 mm/s and pretest speed: 1.0 mm/s.

#### 2.7. Sensorial attributes

After the cakes had cooled for 2 h, they were cut into two equal pieces. Eight semi-trained students from Yıldız Technical University, Faculty of Chemistry Metallurgy, Department of Food Engineering participated in the sensory analysis as panelists. Panelists rated the muffins on a scale of 1 (extremely poor) to 7 (excellent) for crust color, crumb color, pore structure, structure/texture, odor, chewiness, taste/aroma and overall acceptability. During the sensory evaluation, panelists were also offered water to neutralize the taste after each sample tasting (Topkaya & Işık, 2019).

#### 2.8. Statistical evaluation

The findings were given as mean  $\pm$  standard deviation of at least three replicates. Statistical analyses were conducted using JMP Statistical Software version 6 (SAS Institute, Cary, NC) by one-way analysis of variance (ANOVA) (Student's *t* test; P<0.05).

#### 3. Results and Discussion

#### 3.1. Crumb and crust color properties

Crumb and crust color properties, namely  $L^*$ ,  $a^*$ ,  $b^*$ , and  $\Delta E^*$  values were presented in Table 2. HS-0 (70.00±1.13) possessed significantly the highest  $L^*$  values for crumb color than that of HS-5 (48.56±0.46) and HS-10 (44.79±0.79) (P<0.05). As the proportion of HS in the formulation increased, the color darkness of the crumb of the muffin samples increased significantly compared to the control samples due to the brown color of HS (P<0.05). When fiber with a different sugar content was used in place of wheat flour, the non-enzymatic browning can become more apparent, which can cause this effect (Jeddou et al., 2017). Several studies, for instance Karp et al., 2017, Hassan et al., 2019, Heo et al., 2019, Marchetti et al., 2018, Marchetti et al., 2021, and Vo et al., 2023 had been reported similar findings in accordance with our findings. Moreover, when  $a^*$  values of crumb color were compared within each other, it was determined that HS-10 significantly showed highest  $a^*$  value of 6.97±0.09, followed by HS-5 (5.68±0.08) and lastly HS-0 with  $a^*$  value of -0.26±0.04 (P<0.05).  $a^*$  value measured for HS-0 showed the greenness of the samples, however,  $a^*$  value belonging to the HS-5 and HS-10 indicated that crumb color of muffins shifted towards red. These results were in line with previous research examining muffins fortified with coffee ground residual water extracts and powder (Kim et al., 2016).

Regarding the  $b^*$  values of crumb color, the HS-0 significantly had the highest  $b^*$  value (27.30±1.78), followed by HS-5 (14.85±0.27), and HS-10 (12.49±0.40), respectively (P<0.05). The highest yellowness was observed in the crumb of reference samples, however incorporation of HS led to decrease of the yellowness in muffin samples, which was in line with the findings of Karp et al. (2017) and Marchetti et al. (2021). Furthermore,  $\Delta E^*$  values for HS-5, and HS-10 regarding to crust color were determined as 25.49 and 30.12, respectively

compared to the HS-0, showing that the differences can be detectable by human eyes due to  $\Delta E>3$  when compared to HS-0 as previously reported by Yavuz et al. (2022), Akman et al. (2023) and Atlar et al. (2024).

**Table 2.** Crumb and crust color properties of HS-0, HS-5 andHS-10 muffins

Coded samples	$L^*$	<i>a</i> *	<i>b</i> *	$\Delta E^*$
	Crum	b color proper	ties of muffins	3
HS-0	70.00±1.13 <sup>a</sup>	-0.26±0.04°	$27.30{\pm}1.78^{a}$	0.00
HS-5	$48.56 \pm 0.46^{b}$	$5.68 {\pm} 0.08^{b}$	$14.85 \pm 0.27^{b}$	25.49
HS-10	44.79±0.79°	$6.97{\pm}0.09^{a}$	12.49±0.40°	30.12
	Crust	color propert	ties of muffins	
HS-0	51.53±0.38 <sup>a</sup>	14.30±0.31 <sup>a</sup>	30.65±1.18 <sup>a</sup>	0.00
HS-5	$46.07 \pm 1.08^{b}$	8.39±0.24°	$20.68 \pm 0.86^{b}$	12.81
HS-10	44.00±0.67°	$8.59 {\pm} 0.09^{b}$	$17.17 \pm 0.52^{\circ}$	16.46

<sup>a,b,c</sup>: Means with different letters in the same column are significantly different (P<0.05). HS-0: The muffin samples without hazelnut shell; HS-5: The muffin samples including 5% substitution of hazelnut shell; HS-10: The muffin samples including 10% substitution of hazelnut shell.

The following order was observed from low to high in terms of L\* values of crust color among muffin samples: HS-10 (44.00±0.67) <HS-5 (46.07±1.08) <HS-0 (51.53±0.38), indicating that enrichment of the muffins with HS led to a significant decrease in crust lightness (P<0.05). Additionally, the  $a^*$  values of crust color showed the following trend: HS-5  $(8.39\pm0.24) < \text{HS-10} (8.59\pm0.09) < \text{HS-0} (14.30\pm0.31).$ Moreover, as the level of HS addition increased from 0% to 10%, the  $b^*$  value of the crust color decreased significantly from 30.65±1.18 to 17.17±0.52, indicating that incorporation of HS in the muffins led to a reduction in the measured degree of yellowness (P<0.05). Likewise, kimchi by-product powder replacement into the muffins led to the reduction in  $L^*$ ,  $a^*$  and  $b^*$  values compared to the control samples (Heo et al., 2019). Similar observations were also made by Marchetti et al. (2018) who reported that fortification of muffin samples with pecan nut expeller meal resulted a higher darkness and redness and less yellowness compared to the control samples. In addition, the  $\Delta E^*$  value compared to the reference sample was 12.81 for HS-5 and 16.46 for HS-10. The results showed that color differences can be obtained by eye, similar to the  $\Delta E^*$  findings for crumb color. Considering all the results, we can conclude that both the resulting crumb and crust color characteristics of the muffins depended on the HS/wheat flour ratio in the formulation used.

#### 3.2. TPC and DPPH radical scavenging activity

Hazelnut shells are good source of bioactive compounds such as coumaroyl acid, feruloylquinic acid, galloylquinic acid, methyl gallat, myricetin, naringin, quinic acid, quercetin 3rhamnoside, taxifolin, vanillin, and veratric acid (Zhao et al., 2023). The analysis results demonstrated that TPC was not significantly different (P>0.05) in HS-0 (0.22 mg GAE/g), HS-5 (0.23 mg GAE/g) and HS-10 (0.22 mg GAE/g) muffin samples (Table 3), but significantly higher TPC findings were determined in HS-E (0.40 mg GAE/g) (P<0.05). Di Michele et al. (2021) reported the TPC of hazelnut shell extracts in the range of 1.34-4.66 mg/GAE g depending on different extraction method and parameters. Souza et al. (2022) reported the addition of cocoa shell powder decreased TPC values in cakes from 124 to 92 mg GAE/100 g up to 75% level, while 100% replacement caused an increase in TPC (96 mg GAE/100 g).

HS-E (0.08 mg TEAC/g) possessed the greatest DPPH antioxidant activity than that of HS-0 (0.03 mg TEAC/g), HS-5 (0.04 mg TEAC/g), and HS-10 (0.02 mg TEAC/g). The results showed that the DPPH radical scavenging activities of hazelnut shell extracts were lower than Di Michele et al. (2021) who noted the DPPH radical scavenging activities of hazelnut shell extracts in the range of 1.22-4.37 mg TEAC/g depending on different extraction method and parameters. The analysis results revealed that incorporation of HS into the muffin samples had no significant effect on the DPPH antioxidant activity (P>0.05). Souza et al. (2022) reported a decrease in DPPH antioxidant activity of muffins from 484 to 62 g sample/g DPPH with incorporation of cocoa shell powder level rose from 0 to 100%. However, Marchetti et al. (2021) reported 4 or 5 times higher DPPH antioxidant activity in enriched muffins compared to the reference muffins. Not all phenolic compounds exhibit identical antioxidant activity; some demonstrate strong antioxidative properties while others are comparatively weaker. Additionally, these compounds can interact in either antagonistic or synergistic ways with each other or with other constituents present in extracts (Zieliński & Kozłowska, 2000). Moreover, diverse extraction solvents, temperatures, and durations can lead to differing outcomes regarding the TPC and antioxidant activity (Addai et al., 2013).

**Table 3.** Some bioactive properties of HS-0, HS-5 and HS-10 muffins as well as HS-E.

Coded Samples	TPC (mg GAE/g)	DPPH radical scavenging activity (mg TEAC/g)
HS-0	0.22±0.01ª	$0.03{\pm}0.02^{a}$
HS-5	$0.23{\pm}0.00^{a}$	$0.04{\pm}0.01^{a}$
HS-10	$0.22{\pm}0.00^{a}$	$0.02{\pm}0.01^{a}$
HS-E	$0.40{\pm}0.00^{b}$	0.08±0.01ª

Control: The muffin samples without hazelnut shell; HS-5: The muffin samples including 5% substitution of hazelnut shell; HS-10: The muffin samples including 10% substitution of hazelnut shell; HS-E: the extract of hazelnut shell powder; TE: Trolox Equivalents; GAE: Gallic Acid Equivalents.

#### **3.3.** Textural properties

Internal structure properties of the muffin samples were evaluated using the hardness, springiness, cohesiveness, gumminess, chewiness, and resilience given in Table 4. The findings showed that HS-0 had the greatest hardness values (32.14 N). This was followed by HS-5 (21.69 N) and HS-10 (9.79 N), respectively, indicating that the higher the proportion of HS in the muffin formulation, the softer the texture in the fortificated specimens. Moreover, this decrease in hardness values can be related to the higher specific volume or fiber content of the supplemented muffin samples compared to the reference samples (Dhen et al., 2016). The results obtained in terms of hardness were similar to those reported by Marchetti et al. (2018), Jia et al. (2008), Bakkalbası et al. (2015) and Marchetti et al. (2021). Conversely, Heo et al. (2019) reported an increase in the hardness values with the incorporation of kimchi by-product powder into the muffins in comparison to the reference samples.

HS-0, HS-5, and HS-10 did not exhibit any significant differences in their springiness values (P>0.05), which ranged between 0.85-0.89 g. Similar trend was also observed by Huang & Jang (2019). However, adverse trends in terms of springiness and resilience had been reported by Heo et al. (2019) with the addition of kimchi by-product powder to muffin samples.

Table 4. Textural properties of HS-0, HS-5 and HS-10 muffins

Coded Samples	Hardness (N)	Springiness (%)	Cohesiveness	Gumminess (N)	Chewiness (N)	Resilience
HS-0	32.14±0.19 <sup>a</sup>	$0.85{\pm}0.03^{a}$	$0.46 \pm 0.03^{b}$	$14.83 \pm 1.20^{a}$	12.55±1.14 <sup>a</sup>	$0.19{\pm}0.00^{a}$
HS-5	21.69±0.17 <sup>b</sup>	$0.87{\pm}0.03^{a}$	$0.54{\pm}0.01^{b}$	$11.34 \pm 0.90^{b}$	$9.90{\pm}0.83^{b}$	$0.24{\pm}0.00^{a}$
HS-10	9.74±0.19°	$0.89{\pm}0.01^{a}$	$0.74{\pm}0.02^{a}$	7.17±1.12°	6.40±0.92°	$0.38{\pm}0.00^{a}$
a.b.c. Maana with	h different letters in the	some column are signifi	aantly different (D <0	05) IIC 0. The muffin	complex without he	almut shall IIC

a.b.c: Means with different letters in the same column are significantly different (P<0.05). HS-0: The muffin samples without hazelnut shell; HS-5: The muffin samples including 5% substitution of hazelnut shell; HS-10: The muffin samples including 10% substitution of hazelnut shell.

Table 5. Sensory properties of HS-0, HS-5 and HS-10 muffins

Coded Samples	Crust color	Crumb color	Structure/ Texture	Odor	Chewiness	Taste/ Aroma	Overall acceptability	Crust color
HS-0	5.0±0.93ª	4.8±0.71 <sup>b</sup>	4.4±0.52°	5.1±0.35°	5.0±0.76°	4.4±0.52°	$4.9 \pm 0.83^{\circ}$	5.0±0.93ª
HS-5	5.6±0.74ª	5.1±0.99ª	5.5±0.93 <sup>b</sup>	$5.4 \pm 0.74^{b}$	$6.0{\pm}0.76^{a}$	$6.0{\pm}0.76^{a}$	$5.9\pm0.64^{\rm a}$	5.6±0.74 <sup>a</sup>
HS-10	$5.4{\pm}0.74^{a}$	$5.1{\pm}0.83^{a}$	$6.1 \pm 0.64^{a}$	$5.8{\pm}0.46^{a}$	$5.8 {\pm} 0.71^{b}$	$5.9{\pm}0.64^{b}$	$5.8\pm0.46^{\rm b}$	$5.4{\pm}0.74^{a}$
a h a . h a	1 1 1100 1			1 11.00				1 1 11 110

a.b.c: Means with different letters in the same column are significantly different (P<0.05). HS-0: The muffin samples without hazelnut shell; HS-5: The muffin samples including 5% substitution of hazelnut shell; HS-10: The muffin samples including 10% substitution of hazelnut shell.

HS-10 (0.74) possessed significantly the highest cohesiveness than that of HS-0 (0.46) and HS-5 (0.54) (P<0.05), showing that HS makes muffins less tended to the disintegration (Souza et al., 2022). Such decrease in chewiness and hardness values with the incorporation of HS can probably be a result of the oil content of HS as in reported by Goswami et al. (2015). Demirbaş & Akdeniz (2001) reported the fat content of hazelnut shell as 1.4% and 3.2% on wet and dry basis, respectively. This probably resulted in more cohesive and softer muffin samples as previously reported by Marchetti et al. (2018).

In the TPC instrument, 14.83, 11.34 and 7.17 N gumminess values were determined for muffins containing 0%, 5% and 10% HS, respectively. There were statistical differences between gumminess results (P<0.05). In a related work, tomato processing residues supplementation into the muffin samples resulted in a decrease in hardness, gumminess and an increase in springiness, cohesiveness (Mehta et al., 2018).

The chewiness values of the muffin samples ranged from 6.40 to 12.55 N, with the highest chewiness value measured in the HS-0, probably due to water absorption with increasing fiber concentration as a result of increasing HS content in the muffin formulation (Heo et al., 2019). Furthermore, chewiness values also followed similar trend in line with the hardness values. Previously, hardness and chewiness were reported to be inversely related to cake quality (Huang & Yang, 2019). Therefore, in terms of the hardness and chewiness values, we can say that the addition of HS in the formulation at 5% and 10% (w:w) ratios positively affected the cake quality.

The resilience value was positively related with the consumer acceptance of a new product. The highest resilience value was measured in muffin formulation containing 10% HS (0.38), while the lowest value was in the reference muffin samples (0.19). The muffins containing 5% HS had an average resilience value (0.24), however, there was no statistical differences between resilience findings (P>0.05). Overall, the increase in HS content in the formulation exhibited positive effect on texture properties and led to a softer texture.

#### 3.4. Sensory evaluation

Sensory evaluation of cake quality is largely based on subjective qualitative assessment and personal judgment. The results are not always accurate, but they can be taken into account for customer preferences (Jeddou et al., 2017). The photograph images of muffin samples from different angles were given in Figure 2.



HS-0



HS-5



HS-10

**Figure 2.** Photograph images of muffin samples from different angles (HS-0: The muffin samples without hazelnut shell; HS-5: The muffin samples including 5% substitution of hazelnut shell; HS-10: The muffin samples including 10% substitution of hazelnut shell).

The sensory evaluation parameter scores given by panelists for HS-0, HS-5 and HS-10 including the crust color, crumb color, structure/texture, odor, chewiness, taste/aroma, and overall acceptability were presented in Table 5. The crust color scores of muffin samples indicated a random variation between 5.0 and 5.6, which were statistically non-significant (P>0.05). Regarding to crumb color findings, the panelists gave scores in the range of 4.8-5.1. Interestingly, the panelists did not identify crumb color differences between HS-5 and HS-10. Both crumb and crust color scores showed that darker samples were more desirable, but there was no statistically significant differences between crust colors (P>0.05). Moreover, for the parameter of structure/texture, there were significant differences between the three different muffin specimens: HS-0 = 4.4, HS-5 = 5.5 and HS-10 = 6.1 (P<0.05). The increase in chewiness and structure/texture parameter scores in muffins was thought to result from a decrease in hardness values obtained from TPA analysis. This suggested that panelists gave higher scores to softer muffin samples, as reported by Marchetti et al. (2018). Therefore, to obtain higher scores for control samples, more milk and sunflower oil can be added to the formulation (Goswami et al., 2015). Similar findings were also reported in muffins incorporated with pecan nut expeller meal by Marchetti et al. (2018), and they noted that darker crumb and softer texture were the most preferred by panelists. Additionally, the odor scores of muffins were between 5.1 and 5.8, which improved in both HS-5 and HS-10 samples. The HS-5 and HS-10 formulation was rated at almost the same level of acceptance by the panelists, but the reference muffins were the least accepted samples. Following the above-mentioned conclusions, we can say that the incorporation of HS in muffins resulted in uniform appearance, better odor, chewiness and taste/flavor, and improved the overall acceptability by the panelists.

#### 4. Conclusions

Incorporation of hazelnut shell, a by-product in the food industry, into the food formulations allows it to be transformed into a value-added product, increasing the possibilities of its use as a functional ingredient and contributing to the sustainability of the production chain. In the present study, hazelnut shell (0, 5, and 10, w:w) were substituted with wheat flour in order to produce functional muffin samples and their color, some bioactive, textural, and sensory properties were evaluated. The results showed that the reference muffin samples had lighter crust and crumb color compared to the muffins with added hazelnut shell, as the higher pigment content in the enriched muffins reduced the lightness of the composite flour. Furthermore, incorporation of different ratio of hazelnut shell (0, 5, and 10, w:w) had no statistically significant effect on DPPH radical scavenging activity and total phenolic content. Moreover, the increase of hazelnut shell in the formulation resulted in a softer texture. Both satisfactory textural and sensorial scores were provided with the incorporation of hazelnut shell into the muffins. However, further researches also require to investigate the effects of hazelnut shell on the starch retrogradation properties during the storage and whether people with hazelnut allergies can consume hazelnut shells. Also, new symbiotic recipe can be generated with the use of hazelnut shell as a prebiotic source in the future studies.

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## **European Food Science and Engineering**

Eur Food Sci Eng 2024, 5 (1), 8-19 doi: 10.55147/efse.1461684 https://dergipark.org.tr/tr/pub/efse

# Evaluation of the effect of coconut flour addition on the physicochemical and functional properties of wheat flour

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#### ARTICLE INFO

Research Article Article History: Received: 30 March 2024 Accepted: 27 May 2024 Available Online: 30 June 2024 Keywords: Chemical properties Wheat Coconuts Mixture design Principal component analysis

## ABSTRACT

The use of wheat flour alone in the formulation of many products in Africa not only causes a large deficit in the balance of trade, but also nutritional problems, as it is poor in many nutrients. Thus, the present article concern the physico-chemical and functional properties of flours formulated from coconut and wheat. Coconut pulp was used to formulate the flours in association with wheat flour. Using the two-factor centered mixture design, we generated five formulations (F1 to F5) and then the physico-chemical and functional properties were performed. Physico-chemical and functional properties revealed a variation in the various parameters with the substitution percentage. In fact, fiber, lipid, protein, and ash content, as well as energy density, increased with the percentage of coconut flour. The same phenomenon was observed with minerals. All the functional and physical properties were significantly (P<0.05) improved by substitution. Principal component analysis showed small-group correlation between some samples overall. These results show the need to partial substitute wheat with foods from other classes in order to improve the nutritional values and functional properties of derived products. In addition, they offer a way out for African policies to reduce wheat imports and thus retain foreign currency.

#### 1. Introduction

Coconut is a proteo-oleaginous fruit that grows mainly in the world's coastal areas (Srivastava, 2011). The coconut comes from the coconut tree (Cocos nucifera), one of the representatives of the Araceae or Palmaceae family. Often referred to as the "tree of life", the coconut palm probably originated in the Indo-Malaysian region: Fossil coconuts, dating back millions of years, have been found in New Zealand and India and it's now acclimatized in most tropical countries. It is highly prized for its richness in simple sugars, lipids, proteins, fat-soluble vitamins and essential fatty acids, making it a basic raw material for many industries (Trinidad et al., 2006). This composition makes the plant a basic ingredient in formulations aimed at reducing many malnutrition (Gunathilake et al., 2009). The fresh coconut contains a very complete food: the immature almond. Coconut water is rich in mineral salts and sugars. The nutritional value of 100 g of coconut corresponds to 353 kilocalories. On 100 grams, there are on average 3.4 grams of protein, 5.9 grams of carbohydrates, 35.1 grams of fat and 9.5 grams of fiber (Moore, 1986). Thus, the use of coconut flour in all these formulations or products would convey the many nutrients that compose it in order to fight malnutrition in all its forms, metabolic disorder

diseases such as diabetes and obesity (Hossain et al., 2016). The use of coconut flour in human and animal food as well as cosmetics is increasingly continuously in Africa given all the properties it has. Indeed, coconut oil is widely used in India for its scalp regenerating properties, attributed to the presence of a high proportion of the essential fatty acids such as linoleic and linolenic acids (Trinidad et al., 2006). In addition, its supply of essential amino acids makes this fruit an indispensable ingredient in the fight against nutritional deficiencies, especially for vegetarians (Trinidad et al., 2006). Coconut proteins are essentially made up of three classes: albumins, glutelins and globulins, which are recognized for their exceptional emulsifying, foaming and gelling properties (Kwon et al., 1996). It's also important to note that coconut is very rich in insoluble fiber (more than 50% of its fiber content), which makes this matrix a highly digestible food. In view of these properties, the incorporation of coconut flour as a substitute in numerous formulations such as cakes, breads and complementary flours becoming very important (Yalegama et al., 2013). Partial replacing many cereals with this fruit would not only improve their nutritional, sensory and biological properties, but also make them suitable for a wide range of formulations (Fife, 2011).

Wheat is the world's most widely grown and used cereal for both human and animal consumption (Akhtar et al., 2008). The whole wheat kernel is an essential source of B vitamins, minerals such as phosphorus, fiber, starch and protein, the main one being gluten which gives wheat its much sought-after properties in formulations and applications in the baking industry for the production (Bakke & Vickers, 2007; Akhtar et al., 2008). Despite its important properties, many governments in wheat-importing countries such as Cameroon are increasingly recommending reducing dependence on this cereal by using local products. Moreover, cereals are deficient in Lysine, Threonine, Calcium and Iron, and a combination with other nutritional sources like coconut is highly recommended (Adeoti et al., 2023). To this end, the determination of substitution proportions in order to fully benefit from both nutritional and techno-functional properties (i.e ability to be used in bakery) becomes a matter of some urgency and concern.

The composite flours introduced in 1964 by the FAO are the result of combining several ingredients from different classes to overcome the shortcomings of each matrix (Jisha et al., 2008; Hasmadi et al., 2020). This has not only helped to reduce malnutrition, but also poverty (Hasmadi et al., 2020). Furthermore to the nutritional and economic benefits, it should be noted that the supplementation of coconut flour with cereals tends to reduce their specific volume and improve their texture, given their high content in reducing sugar which is responsible for the maillard reaction that is essential for the product's acceptability (Adeoti et al., 2023). The work of Nilufer et al. (2008) showed that the properties of flours made from wheat and soy were much better than those of 100% wheat. Therefore, this work aimed to evaluate the physico-chemical and functional properties of mixture flours made up from wheat and coconuts.

#### 2. Materials and methods

#### 2.1. Research design

The research carried out for this study was experimental and quantitative (Trochim, 2005). It consisted in collecting quantitative data from the analysis of various samples.

#### 2.2. Materials

The material used in this work was coconut and wheat flour, which was purchased from traders at Market B in the town of Dschang (Menoua division, West Region of Cameroon). Once collected, these were transported to the Research Unit of Biochemistry of Medicinal Plants, Food Science and Nutrition where they underwent various transformations prior to analysis.

#### 2.3. Data source location

The coconuts and wheat flour were purchased from Market B in the town of Dschang (Altitude: 1350 m; 5°26.6382' North, 10°3.1992' East), West Cameroon Region while coconut flour and the various formulations were produced at the Research Unit of Biochemistry of Medicinal Plants, Food Science and Nutrition of the Department of Biochemistry, Faculty of Science, University of Dschang (Altitude 1500 m; 5°27' North, 10°04' East). The various analyzes were carried out both at the Soil Science Research Unit of the Faculty of Agronomy and Agricultural Sciences and at the Medicinal Plants, Food Science and Nutrition Biochemistry Research Unit of the Biochemistry Department of the Faculty of Science at the University of Dschang.

#### 2.4. Methods

#### Production of the coconut flour

Coconut endosperm after the removal of shell and paring, was shredded, grated and oven dried at 50 °C for 24 h in a hot air oven (model QUB 305010G, Gallenkamp, UK), milled using mill (model MXAC2105, Panasonic, Japan) to obtain coconut flour. The flour obtained was packaged in polyethylene bag before use.

#### Formulation of composite flour

A centered mixture design was applied to obtain the different proportion of flour. The design was carried out using software from Minitab® (version 18.1 Minitab Inc). According to mixture design, the coconut flour has been incorporated at 7.50%, 0.00%, 15.00%, 22.50% and 30.00% into the wheat flour. The formulations obtained were labelled F1, F2, F3, F4 and F5 (Table 1).

<b>Lable 1.</b> Formulation of composite flou
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Formulations	Wheat flour (%)	Coconut flour (%)
F1	92.50	7.50
F2	100.00	0.00
F3	85.00	15.00
F4	77.50	22.50
F5	70.00	30.00

Evaluation of functional properties of composite flour

The different functional parameters like water retention capacity (CRE), oil retention capacity (CRH), swelling power (TG) and ratio between CRH and CRE were evaluated according to Lin et al. (1974) method partially modified by Tambo et al. (2019a,b). One gram (1 g) of each sample was respectively mixed with 10 mL of sunflower oil or distilled water and incubated in a water bath at 30 °C for 30 min. The mixture was centrifuged at 4500 xg for 15 min. The volume of water or oil absorbed was measured. The CRE and CRH were calculated as follows (Eq. 1):

$$CRE/CRH (\%) = \frac{V_0 - V_1}{V_0} \times 100$$
 (1)

where  $V_0$ , initial volume of oil (mL);  $V_1$ : sedimented volume after centrifugation (mL); CRE, water retention capacity; CRH, oil retention capacity

The swelling power (TG) was deduced by difference between the mass of the sample having retained the water  $(M_1)$  and that of the start  $(M_0)$ . The swelling power is given by the Eq. 2:

Swelling power (%) = 
$$\frac{[(M_1 - M_0) \times 100]}{M_1}$$
 (2)

where  $M_1$ , mass of the pellet or mass after water intake (g);  $M_0$ , mass of the test sample (g)

#### Determination of physical properties

The physical properties like loose bulk density (LB), packed bulk density (PB), Porosity and hausner ratio (HR) were determined according to the method described by Okaka et al. (1991). In fact, an empty crucible of known volume was filled with flour (100 g) and weighed before and after packed one hundred times. The results obtained were expressed as follows (Eq. 3, 4 and 5): Losse bulk and Tapped density  $\left(\frac{g}{mL}\right) =$ weight of sample (g)

volume occupied by the sample

$$Hausner\ ratio = \frac{\text{Tapped density}}{\text{Loose bulk density}} \tag{4}$$

$$Porosity (\%) = \frac{\text{Tapped density} - \text{Loose bulk density}}{\text{Tapped density}} * 100$$
(5)

The standard method developed by AOAC (1990) was used to assessed the pH. In fact, one g of sample of each flour was weighed into centrifuge tubes and mixed with 10 mL of distilled water. The suspension obtained was stirred using a "Barnstead/Thermolyne" brand vortexer for 30 min and then centrifuged at 4000 rpm for 5 min with a refrigerated "Heraeus" brand centrifuge. The pH of the aqueous phase was measured using a calibrated pH meter, at 25.03±0.22 °C.

#### Chemical composition of the formulations

The formulations produced as described above were subjected to different chemical analyses. The proximate chemical composition (moisture, crude protein, crude fiber, total digestible carbohydrate, and energy calorie) was carried out according to the standard methods described by AOAC (1990). The soxhlet extraction method using hexane as solvent was used to quantify lipids in the formulations (IUPAC, 1979). The Kjeldahl method, which converts organic nitrogen into ammoniacal nitrogen in the presence of concentrated sulfuric acid, was used to determine total nitrogen. Proteins were deduced by multiplying the rate of nitrogen release by a factor of 6.25. Total digestible carbohydrates content was obtained by difference with moisture, ash, lipid, fiber and protein content (AOAC, 1990) according to Eq. 6:

%Total digestible carbohydrates = %Dry matter 
$$-$$
%Proteins  $-$ %Lipids  $-$ %Ashes  $-$ %Fibers (6)

The total metabolizable calorific energy (expressed in kcal/100 g) in 100 g of each formulation was obtained after multiplying the quantities of carbohydrates, lipids and proteins by the coefficients of Atwater & Rosa (1899), which are 4, 9 and 4 respectively (Eq. 7):

Calorific energy (kcal/100 g)	
$= (4 \times (\% \text{carbohydrates})) + (9)$	
$\times$ (%lipids)) + (4 $\times$ (%proteins))	(7)

Starch and amylose contents were determined using the methods described by Jarvis & Walker (1993), and Chrastyl (1987) respectively. In the case of starch, after extraction in a mixture of potassium hydroxide (KOH) and hydrochloric acid (HCl), an iodine-iodide solution (I<sub>2</sub>/KI) of lugol was used to determine the content in the various samples. Amylose content was determined using the same procedure, plus defatting. Amylopectin content was obtained by the difference between starch and amylose percentage.

The ash was determined after total destruction of the organic matter by incineration in a muffle furnace (Perkin Elmer, USA) at 560 °C under oxidizing atmosphere according to the protocol described by AOAC (1990). The same ashes were used to quantify the minerals (Ca, Mg, Na, K, Fe, Zn and Cu) after predigestion in a liquid HNO<sub>3</sub>/HCl mixture prior to reading with an atomic absorption spectrophotometer (BIOBASE BK-D590, spectrophotometer) at different wavelengths (AOAC, 1990).

#### 2.5. Statistical analyzes

(3)

The experimental data, reported as means±standard deviation was calculated using Excel 2016 software (Microsoft Inc). The graphs were draw using the same software. The Analysis of Variance (ANOVA) was used to establish the difference between the samples. When the difference was found, the Fisher test was applied at the 5% probability level to compare these means using MINITAB (version 18.1 Minitab Inc). The Principal Component Analysis between chemical, physical and functional properties of different formulations were done using XLSTAT® 2014 for IBM STATISTICS (add-in for Excel) and allowed us to correlate the different parameters (variables and observations).

#### 3. Results and Discussion

#### 3.1. Proximate chemical composition

The physico-chemical and functional property data for the formulations presented in this work are intended to demonstrate the importance of substituting wheat flour with coconut flour. The proximate chemical analysis presented in Table 2 shows that the moisture content of composite flours does not change statistically. The level of substitution does not significantly (P=0.355) affect by the moisture level. The moisture content is an indicator of a product's storage and transport stability, and is highly dependent on the nature of the product's constituents, drying time and temperature (Kumarakuru et al., 2024). The values obtained are less than 14%, which then make the flours suitable for the production of long shelf life foods, more stable or resistant to chemical-microbial deterioration during storage or transport (Ndangui, 2015; Ajjata et al., 2016). Lipids are responsible for food flavor but also for rapid degradation on the effect of oxidation (Muyanja et al., 2014; Shams et al., 2022). Lipid content fluctuated significantly (P=0.000) with the proportion of substitution and this is related to the contribution of coconut flour, which is an oil-rich matrix. The contents obtained are similar to those of Klang et al. (2019a) on corn-, soy- and moringa-based formulations. The addition of coconut flour enabled us to obtain flours with contents covering more than 100% of the recommended daily intake of this nutrient (FAO/WHO, 2006). The protein content is range from 15.05 (F4) to 16.80 (F2). It was significantly (P=0.004) reduced by supplementation, which is contrary to the observations of many authors (Klang et al., 2019a; Onipe et al., 2024). This may be due to the quantification method used. Onipe et al. (2024) obtained protein contents 11 times lower than those of this study in millet- and fruit-based formulations, proving that these flours would be suitable as complementary foods to fight against protein-energy malnutrition in children aged 6 to 59 months. Ash refers to the presence of minerals in a food (Sukainah et al., 2023). This parameter varied significantly (P=0.000) with the percentage of nutmeal intake, with values ranging from 1.05 (F5) to 9.10% (F4). The values obtained are not consistent with the substitution rate, probably due to the analysis technique or calcination time, or to losses suffered by the last sample during processing. The values obtained are higher than those of Aini et al. (2010) but similar to those of Klang et al. (2019a). The increase in ash content in all formulations with the exception of formulation 5 is in line with the work of Kohajdova et al. (2012), who reported an improvement in this parameter with the level of carrot powders in different formulations.

Table 2. Proximate chemical composition of different flours

Samples	F1	F2	F3	F4	F5	p-value
Moisture content (%)	$6.44 \pm 0.14^{a}$	6.38±0.12 <sup>a</sup>	$6.21 \pm 0.06^{a}$	$6.24{\pm}0.09^{a}$	$6.28 \pm 0.16^{a}$	0.355
Lipids content (%)	$7.08 \pm 0.27^{d}$	$2.58 \pm 0.00^{e}$	11.11±0.34°	15.76±0.03 <sup>b</sup>	$20.42 \pm 0.42^{a}$	0.000
Proteins content (%)	$16.19 \pm 0.25^{a}$	$16.80{\pm}0.25^{a}$	15.49±0.25 <sup>b</sup>	15.05±0.25 <sup>b</sup>	15.31±0.25 <sup>b</sup>	0.004
Ash content (%)	$3.10{\pm}0.14^{b}$	$2.05 \pm 0.07^{\circ}$	2.15±0.21°	$9.10{\pm}0.14^{a}$	$1.05 \pm 0.07^{d}$	0.000
Fibers content (%)	2.84±0.25°	$3.04 \pm 0.22^{\circ}$	$1.41 \pm 0.25^{d}$	$4.02 \pm 0.25^{b}$	$5.67 \pm 0.25^{a}$	0.000
Glucids content (%)	64.51±0.53 <sup>b</sup>	69.15±0.42 <sup>a</sup>	63.63±0.31 <sup>b</sup>	49.84±0.57°	51.11±1.12°	0.000
Energy value (kcal/100 g)	$386.51 \pm 3.55^{d}$	367.08±0.63 <sup>e</sup>	416.48±3.33 <sup>b</sup>	401.39±1.04°	449.46±0.31ª	0.000
Starch content (%)	$46.94 \pm 2.87^{b}$	52.42±1.48 <sup>a</sup>	$33.06 \pm 1.64^{d}$	37.50±1.14°	$31.72 \pm 1.14^{d}$	0.000
Amylose content (%)	12.92±0.79°	$13.81 \pm 0.42^{bc}$	14.45±1.11 <sup>b</sup>	$15.87 \pm 0.45^{a}$	$16.07 \pm 0.54^{a}$	0.001
Amylopectin content (%)	$87.08 \pm 0.79^{a}$	$86.19 \pm 0.42^{ab}$	85.55±1.11 <sup>b</sup>	84.13±0.45°	83.93±0.54°	0.001
Ratio amylose/amylopectin	0.15±0.01°	$0.16 \pm 0.00^{bc}$	$0.17 \pm 0.01^{b}$	$0.19{\pm}0.01^{a}$	$0.19{\pm}0.01^{a}$	0.001

F1=92.5% wheat flour + 7.5% coconut flour; F2=100% wheat flour; F3=85% wheat flour + 15% coconut flour; F4=77.5% wheat flour + 22.5% coconut flour; F5=70% wheat flour + 30% coconut flour.

These ash contents show that these formulations could be recommended in the preparation of cookies and cakes for the elderly or diabetics, as well as for children suffering from hidden hunger. The fiber plays a major role in the health of our organism, particularly the colon, ensuring good transit and, above all, preventing cancer, diabetes and obesity (Sheikh et al., 2019). The fiber content increases significantly (P=0.000) with the substitution rate (except F3), ranging from 1.41 (F3) to 5.67% (F5). The reduction observed with formulation F3 is thought to be due to partial digestion of the fibres in this formulation, resulting in poor quantification. These variations concur with the observations of Malomo et al. (2011), who observed a positive evolution of fiber in bread with the substitution rate of the fruit formulation. The use of these formulated flours, in addition to their energy and nutritional values, would also have significant health benefits due to their fiber content. This positive trend in fiber content is testimony to the contribution of coconut flour, as demonstrated by Yalegama et al. (2013). The levels obtained are higher than those of Onipe et al. (2024), which ranged from 0.24 to 3.15%. The carbohydrates are the main form of energy that can be directly metabolized by the body and serve as the brain's energy raw material (Tambo et al., 2019a,b).

Furthermore, they determine the functional properties, color (particularly through non-enzymatic browning), texture and flavor of the products (Sukainah et al., 2023). This parameter decreased with the substitution rate from 69.15% (F2 or wheat flour) to 49.84% (F4). Coconut is a low-carbohydrate proteinoleaginous plant, although it is a source of simple sugars, and this would explain these results (Sukainah et al., 2023). Bello & Esin (2023) also reported a drop in carbohydrate content in a formulation based on corn, cowpea and coconut when the proportions of the last two ingredients were increased. Moreover, this parameter is negatively (r=-0.9305; P<0.05) correlated with lipids, as shown by the results in Table 8. The values obtained are lower than those of Bello & Esin (2023), which ranged from 76.08 to 78.36. Calorific energy, which is related to nutritional composition, also increased with coconut content, while the opposite was observed for starch content. This parameter was significantly (P=0.000) boosted by the addition of coconut flour, as shown in Table 2. Indeed, the energy density of the flours increased from 367.08 kcal (F2) to 449.46 kcal (F5). This contribution is in line with the high lipid content provided by coconut, and thus concurs with the work of Bello & Esin (2023) who observed a positive evolution of the flours formulated when the coconut flour substitution rate increased. Furthermore, these results are confirmed by the positive correlation (r=0.9089; P<0.05) between lipids content and energy density (Table 8). The energy values reported are similar to those of Bello & Esin (2023), which ranged from

404.97 to 413 kcal. The results obtained also show that the consumption or use of these flours in the formulation of products intended for supplemental feeding would make it possible to meet a deficit of more than 70% of their daily requirements for a 100 g portion (FAO/WHO, 2006; Klang et al. 2019a,b). Starch, amylose, amylopectin and the amylose/amylopectin ratio are parameters that significantly influence the various techno-functional properties of a flour, such as water retention capacity and physical stability (Klang et al., 2019b; Dongmo et al., 2020). One example is the positive correlation between starch content and swelling rate (r=0.1648; P>0.05). Starch and amylopectin contents decreased substantially with the coconut flour substitution rate, while amylose content, which is responsible for the retrogradation and hence thermal instability of starch, increased with substitution. Cisse et al. (2023) also observed a decrease in starch content with the rate of substitution of attiéké by legumes. Coconut is low in starch, which would explain its negative effect on starch content. These results are also confirmed by the negative correlation (r=-0.8821; P<0.05) between the lipid content of coconut and starch content (see Table 8). The results obtained for starch content are lower than those of Cisse et al. (2023), which ranged from 69.79 to 77.91% in flour formulations based on Attieke & Voandzou. These results also demonstrate the need for other cereal sources to benefit from starch properties. The amylose/amylopectin ratios of the various formulations are positively related with the substitution rates, but remained below 1, demonstrating that these flours will retrograde little and consequently maintain the techno-functional properties of starch. The data obtained are in the same range as those of Dongmo et al. (2020), which varied between 0.13 and 0.26 for corn flours subjected to different treatments.

## 3.2. Proximate mineral composition in different blending flours

The influence of the substitution percentage on the mineral composition of formulations is shown in Table 3. The minerals are so essential for the proper functioning of the organism, participating in homeostasis, enzymatic activity, muscle contraction, cell growth and activity, as well as the technofunctional properties of flours (Klang et al., 2019a,b; Dongmo et al., 2020; Tambo et al., 2023). With regard to functional properties, the work of Tambo et al. (2019a,b) revealed a positive correlation between Ca and water retention capacity. The same applies to this study, where a positive correlation (r=0.7682; P<0.05) was found between the two parameters. In this study, seven minerals were evaluated, of which four are major (Ca, Mg, Na and K) and three are microelements (Fe, Cu

and Zn). With the exception of K, all ions show a negative trend with coconut content. These observations does not concur with those of Klang et al. (2019a). These results can be explained by the composition of the ash used to quantify the minerals. Poor mineralisation and the presence of organic matter in certain formulations, particularly F5, are thought to be responsible for these variations. In addition, the presence of phytates and oxalates (complexing agents for divalent cations) in coconuts could explain a drop in these elements with substitution. All the micro and macro elements were significantly (P<0.05) affected by the percentage of substitution, and this can be explained by the significant contribution of coconut flour. The Calcium levels contributed over 50% of the recommended daily requirement (2 g/day) for the F1 formulation, and 26% for the F5 formulation. These values are much higher than the 3.5% obtained by Diallo et al. (2024) with Bambara groundnuts. The contribution of magnesium ranged from 26% to 36%, while that of copper (1 mg/day) was over 300%. These results suggest that the consumption of these formulated flours should be recommended for athletes, pregnant women, the elderly and children (Omotoso, 2006). Iron is an important element in hemoglobin synthesis and oxygen transport in the body (Omotoso, 2006). These activities make iron an indispensable for women. Iron intake mineral decreases with supplementation, but remains above 100% of daily contribution for the F1 formulation. The levels of these minerals were higher than those of Botella-Martinez et al. (2023), with the exception of zinc. Calcium and sodium availability can be assessed by determining Ca/Mg and Na/K ratios. These ratios show that supplementation reduces Ca availability from 11.18 Mg (F1) to 4.33 Mg (F5), and Na availability from 0.22 K (F1) to 0.01 K (F5). Although these results are decreasing, they nevertheless demonstrate that consumption of the different blending would provide a large quantity of available and therefore bioavailable Calcium, and would also be safe for hypertensive patients, given the low availability of Na (Tambo et al., 2019a,b).

Table 3.	Proximate	mineral	composition	of	different	flours
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## **3.3.** Influence of blending ratio on functional properties of flours

The functional properties were evaluated and results are presented in Table 4. The water absorption capacity is a parameter highly dependent on flour composition, in particular protein, starch, amylose and amylopectin content, but also on the conformational structure of these molecules (Onipe et al., 2024). This parameter represents a flour's ability to bind water molecules under stress conditions and form a more or less stable gel consistency (Bajo et al., 2021). The water retention decreased with substitution and is lesser than those of wheat flour. These observations are contrary to those of Adeoti et al. (2023), who observed an improvement in water retention capacity with increasing substitution by cocoa powder. This is the consequence of a reduction in the hydrophilic group content by lowering the starch content. Indeed, substitution with coconut flour increases the lipid and protein content, which forms complexes on the starch surface and with amylose, thus hindering hydrophilic interactions (Bajo et al., 2021). It would also be linked to an increase in the amylose content of formulated flours and a complexification of the starch structure. Unlike amylopectin, amylose is less soluble, as it interacts with insoluble molecules such as lipids (Adeoti et al., 2023). The negative correlation (r=-0.3692) with amylose content confirms these observations (Dongmo et al., 2020). These results suggest the use of these flours in the formulation of foods that do not require dough elasticity, such as cookies.

The swelling capacity depends both on intrinsic factors such as molecular composition and organization, and extrinsic factors such as culinary treatments and water stress (Onipe et al., 2024). It measures a flour's ability to form a consistent and stable gel with water molecules. The analysis of this table shows that this parameter was significantly (P=0.025) influenced by the substitution rate, and decreased with it. The values ranged from 53.70 (F4) to 65.36 (F1).

Samples	F1	F2	F3	<b>F4</b>	F5	p-value
Ca (mg/100 g)	1360.16±0.22 <sup>a</sup>	720.16±0.22°	640.17±0.23 <sup>d</sup>	800.18±0.25 <sup>b</sup>	400.15±0.21 <sup>e</sup>	0.000
Mg (mg/100 g)	121.68±0.25 <sup>b</sup>	$102.21 \pm 0.21^{d}$	111.96±0.25°	131.40±0.25 <sup>a</sup>	92.52±0.25 <sup>e</sup>	0.000
Cu (mg/100 g)	3.56±0.22ª	$1.07 \pm 0.17^{b}$	$1.12 \pm 0.37^{b}$	1.19±0.21 <sup>b</sup>	$1.59 \pm 0.67^{b}$	0.005
Fe (mg/100 g)	$14.11 \pm 0.00^{a}$	$0.99{\pm}0.00^{\circ}$	$3.22 \pm 0.00^{b}$	$0.96{\pm}0.00^{d}$	$0.13 \pm 0.00^{e}$	0.000
Zn (mg/100 g)	15.15±0.22 <sup>a</sup>	$0.37 \pm 0.06^{\circ}$	$0.35 \pm 0.00^{\circ}$	$0.30{\pm}0.00^{\circ}$	$1.74{\pm}0.00^{b}$	0.000
Na (mg/100 g)	36.82±0.22°	50.15±0.21 <sup>b</sup>	65.17±0.23ª	25.18±0.25 <sup>d</sup>	25.18±0.25 <sup>d</sup>	0.000
K (mg/100 g)	167.85±0.22°	1237.65±599.80 <sup>b</sup>	1431.23±325.56 <sup>b</sup>	1200.82±0.21 <sup>b</sup>	2421.84±319.20 <sup>a</sup>	0.010
Ca/Mg	$11.18 \pm 0.02^{a}$	$7.04{\pm}0.01^{b}$	$5.71 \pm 0.01^{d}$	6.09±0.01°	4.33±0.01 <sup>e</sup>	0.000
Na/K	$0.22{\pm}0.001^{a}$	$0.05 {\pm} 0.02^{b}$	$0.05 \pm 0.01^{b}$	$0.02 \pm 0.00^{bc}$	$0.01 \pm 0.001^{\circ}$	0.000

F1=92.5% wheat flour + 7.5% coconut flour; F2=100% wheat flour; F3=85% wheat flour +15% coconut flour; F4=77.5% wheat flour + 22.5% coconut flour; F5=70% wheat flour + 30% coconut flour.

**Table 4.** Functional properties of different flours

Samples	WRC (%)	SP (%)	<b>OHC</b> (%)	Ratio OHC/WRC
F1	22.00±0.00ª	65.36±1.53ª	63.52±0.79 <sup>a</sup>	$2.89{\pm}0.04^{a}$
F2	$27.00 \pm 4.24^{a}$	$65.16 \pm 0.53^{a}$	$63.76{\pm}0.74^{\rm a}$	$2.39{\pm}0.40^{a}$
F3	22.00±2.83ª	$54.02 \pm 6.39^{b}$	$60.46 \pm 0.43^{b}$	$2.77{\pm}0.38^{a}$
F4	21.00±1.41ª	53.70±0.61 <sup>b</sup>	$60.31 \pm 0.66^{b}$	$2.88{\pm}0.16^{a}$
F5	$22.00{\pm}2.83^{a}$	$61.38{\pm}1.05^{ab}$	61.00±0.33 <sup>b</sup>	$2.80{\pm}0.37^{a}$
p-value	0.310	0.025	0.005	0.540

WRC: Water retention Capacity; SP: Swelling power; OHC: Oil holding capacity. F1=92.5% wheat flour + 7.5% coconut flour; F2=100% wheat flour; F3=85% wheat flour + 15% coconut flour; F4=77.5% wheat flour + 22.5% coconut flour; F5=70% wheat flour + 30% coconut flour.

In fact, the addition of a small quantity of coconut not only provides polar amino acids in the proteins, but also limits molecular saturation, which facilitates interactions between polar molecules and water (Tambo et al., 2023). These results are contrary to those of many authors who have demonstrated that the presence of fibres improves water retention capacity and consequently swelling rate (Fida et al., 2020; Onipe et al., 2024). This suggests that coconut fibers are mainly insoluble and would therefore be recommended for people suffering from diabetes and obesity. In addition, the richness of minerals such as Ca and Mg capable of forming strong and stable ionic bonds with the water molecules would also explain these results. In addition, a positive correlation (r=0.6356; P<0.05) was observed between swelling power and water retention capacity, indicating the high solubility of these flours. Furthermore, these observations reveal that partial substitution at 7.5% rate with coconut flour would be beneficial to manufacturers in terms of swelling power.

The oil retention capacity, on the other hand, decreased significantly (P=0.005) with the substitution percentage, thus showing the interest of substitution in reducing oil intake, since coconut is already a source of lipid. Indeed, the richness of polar amino acids in coconut flours and a molecular arrangement orienting these towards the interior of the chain would be the consequence of weak hydrophobic interactions with the hydrocarbon chains of fatty acids (Mubaiwa et al., 2018). Similarly, an increased intake of coconut flour would lead to saturation of amylose-lipid interactions, thus reducing interactions with additional fats (Chandra et al., 2015). The capacities obtained are lower than the 69-92% range obtained by Yusufu & Ejeh (2018) in different wheat- and bambarabased flour formulations. This therefore suggests that the use of different formulations would limit the absorption of oil, which could be a danger to the product's shelf life or preservation. Indeed, oils are subject to oxidation, responsible for the formation of rancid odors and toxic compounds, thus limiting storage. In addition, these results also show that the use of these flours would be beneficial for private individuals, as they would limit expenditure on the purchase of oil and, above all, energy, as they would take less time to cook.

The OHC/WRC ratio, like emulsifying capacity, provides information on the ability of matrix constituents to form table mixtures by modifying the interfacial tensions of the heterogeneous mixture constituents (Matidza et al., 2023). This ratio was not affected (P=0.540) by the substitution rate, although the highest values were found with formulated flours. Indeed, Matidza et al (2023) demonstrated that the addition of oil-rich matrices would facilitate the stability of the mixture, as these lipids would play an amphiphilic role. The results obtained show that these flours are suitable for the formulation of cakes and breads, as they promote interactions between the hydrophobic zones of the gluten and the starch of the flours, thus contributing to the creation of a more compact and stable molecular network (Thakaeng et al., 2021). Furthermore, these results show that substitution with coconut flour would improve the flavor and taste of foods in view of the higher ratios.

## **3.4.** Influence of blending ratio on physical properties of flours

The physical properties are presented in Table 5. The physical properties are also among the parameters governing a flour's applicability. The table shows that pH varied between 7.07 (F4) and 7.35 (F2). It was not significantly influenced (P= 0.122) by the rate of substitution by coconut flour, although the substituted flours presented the lowest values. The richness of coconut in acidic amino acids would be responsible for these observations (Owusu-Kwarteng et al., 2022). These results would also explain the data on water retention capacity. Indeed, Tambo et al. (2019a) reported that a reduction in pH due to the presence of organic acids lowers the water-holding capacity of a flour. The values obtained are all above 7, demonstrating that there is no need to use acidity-correcting agents during formulation, and that these macronutrients are readily accepted and, above all, digestible. In addition, products formulated from these flours are more stable (as they are less conducive to microbial growth) and easier to store (Matidza et al., 2023). The results obtained are superior to those of Matidza et al. (2023), which ranged from 5.27 to 5.79 for banana- and wheat-based flours.

The porosity measures the interstices that can be found in a heterogeneous mix, and is related to macronutrient composition. Indeed, the richer a mixture is in macronutrients such as proteins and starch, the less porous it is. Indeed, proteins and starch, being high molecular weight macromolecules, will not more rationally occupy all the spaces, thus facilitating the passage of air and favoring packaging (Jha & Sit, 2024). These observations are confirmed by the negative correlation between porosity and protein (r=-0.3949) and starch (r=-0.5996; P<0.05) contents respectively. The porosity ranged from 50 (F2) to 36.97% (F3). It was significantly (P=0.000) reduced by the coconut flour substitution rate. In fact, coconut substitution increases the lipid content, which promotes cohesion of the molecules by acting as a binder, thus reducing particle dispersion and making them resistant to any flow movement (Jha & Sit, 2024).

The values obtained are higher than those of Jha & Sit (2024), which ranged from 7 to 18% in a mixture of zein and corn starch. These results show that substituting coconut flour for 7.5% would result in a product that would not pose any packaging problems. In fact, these data provide further evidence that the addition of coconut produces flours suitable for bread-making (Djikeng et al., 2022).

The bulk densities depend on particle size, nutrient composition (especially protein) and pellet shape (Dongmo et al., 2020; Djikeng et al., 2022; Tambo et al., 2023). They influence food intake, preservation, transport and packaging (Dongmo et al., 2020). Indeed, Jha & Sit (2024) reported that a flour with a high bulk density would be beneficial as it would occupy less pace during packaging.

Table 5. Physical properties of different flours

Samples	F1	F2	F3	F4	F5	n-value
pH	7.32±0.06ª	7.35±0.04ª	7.26±0.10 <sup>ab</sup>	7.07±0.01 <sup>b</sup>	7.21±0.16 <sup>ab</sup>	0.122
Porosity (%)	$46.67 \pm 0.00^{b}$	$50.00 \pm 0.00^{a}$	36.97±2.10°	38.08±1.39°	37.98±0.68°	0.000
Loose bulk density (g/mL)	$0.40{\pm}0.00^{\circ}$	$0.43 \pm 0.00^{b}$	$0.47{\pm}0.01^{a}$	$0.48{\pm}0.01^{a}$	$0.47{\pm}0.00^{a}$	0.001
Packed bulk density (g/mL)	$0.75 \pm 0.00^{\circ}$	$0.86{\pm}0.00^{a}$	0.75±0.32°	$0.77 \pm 0.00^{b}$	$0.75{\pm}0.00^{\circ}$	0.000
Hausner ratio	1.47±0.02°	$1.71 \pm 0.00^{b}$	$1.93{\pm}0.00^{\rm a}$	$1.71 \pm 0.00^{b}$	$1.77 {\pm} 0.08^{b}$	0.001

F1=92.5% wheat flour + 7.5% coconut flour; F2=100% wheat flour; F3=85% wheat flour + 15% coconut flour; F4=77.5% wheat flour + 22.5% coconut flour; F5=70% wheat flour + 30% coconut flour.

The Loose bulk density varied between 0.48 (F4) and 0.43 g/mL (F2) and was significantly (P=0.001) improved by coconut substitution rate, while the opposite effect was observed with the tapped bulk density. In fact, the higher lipid content with coconut flour substitution led to particle aggregation, making them denser and less porous. There was also an inverse variation between loose bulk density and tapped bulk density, contrary to the observations of Djikeng et al. (2022). This observation is confirmed by the negative correlation with porosity (r=-0.7421; P<0.05). Onipe et al (2024) also reported an improvement in bulk density with the percentage of Parinari curatellifolia flour. The values obtained are respectively lower for the loose bulk and tapped bulk densities than those of Onipe et al. (2024), which ranged from 0.82 to 0.85 g/mL, and those of Bello & Esin (2023), which varied from 0.72 to 0.96 g/mL. These results demonstrate that these flours are suitable for the preparation of supplementary feeds with adequate viscosity, easy digestibility and high energy density (Tambo et al., 2022).

The evaluation of compression and compaction resulting from intermolecular friction between the constituents of a blend are possible through to the determination of the Hausner ratio (Shumaila et al., 2015). Indeed, compared to the control (F2), coconut substitution increased significantly (P<0.05) with 15% substitution. This parameter was unaffected by the 22.5% and 30% substitution, while it dropped significantly (P<0.05) at

Table 6. Contributions and squared cosines of variables

7.5% to reach a value of 1.47 (F1).

Overall, this parameter was affected by substitution (P=0.001). These ratios are higher than the range of 1.10 to 1.40 obtained by Djikeng et al. (2022) on snail meal. In fact, the difference is due to a difference in composition directly linked to the coconut flour content. All these values are higher than 1, which has been defined as the value of excellence for judging good flowability, tenderness and above all resistance to shearing phenomena (Jha & Sit, 2024). These results demonstrate the need to control the percentage of wheat substitution by legumes in order to benefit from all their techno-functional properties.

# **3.5.** Principal component analysis between the nutritional, physical and functional properties of the different blending flours

The correlation between the various physico-chemical and functional parameters was assessed using principal component analysis. This multivariate analysis was also used to classify the formulations according to their similarities. This analysis showed that variables and observations are correlated in small groups. Formulations F1, F4 and F5 form the F1 axis, while formulations F2 and F3 form the F2 axis (Table 6).

	Variable	s contributi	ons (%)	Variab	les Squares (	Cosines
Parameters	F1	F2	F3	F1	F2	F3
Water retention capacity (WRC)	2.1140	4.3247	4.1673	0.3240	0.3353	0.1338
Swelling power	0.9163	10.2316	0.1005	0.1404	0.7933	0.0032
Oil retention capacity (ORC)	5.1324	0.6011	4.7631	0.7866	0.0466	0.1529
ORC/WRC	0.3247	11.2853	1.4370	0.0498	0.8750	0.0461
pH	0.5842	8.7194	7.2477	0.0895	0.6760	0.2326
Moisture	4.6583	0.3884	0.1087	0.7140	0.0301	0.0035
Lipids	3.7227	3.9318	3.5351	0.5706	0.3048	0.1135
Porosity	4.0724	4.2909	0.5607	0.6242	0.3327	0.0180
Loose bulk	5.5518	0.1325	4.2975	0.8509	0.0103	0.1379
Packed bulk	0.0074	6.4742	13.8317	0.0011	0.5020	0.4440
Hausner ratio	3.6493	4.6029	1.2684	0.5593	0.3569	0.0407
Starch	3.5381	0.4873	8.8737	0.5423	0.0378	0.2848
Amylose	5.8621	1.2822	0.0630	0.8985	0.0994	0.0020
Amylopectin	5.8621	1.2822	0.0630	0.8985	0.0994	0.0020
Amylose/Amylopectin	5.8306	1.3482	0.0549	0.8936	0.1045	0.0018
Proteins	3.1317	2.6799	1.4076	0.4800	0.2078	0.0452
Glucids	3.2220	6.2787	0.0000	0.4938	0.4868	0.0000
Fibers	1.5432	5.6699	0.0270	0.2365	0.4396	0.0009
Energy	3.0668	1.1755	13.6669	0.4700	0.0911	0.4387
Ashes	0.1500	2.5369	14.3365	0.0230	0.1967	0.4602
Na	0.3301	10.8523	1.1966	0.0506	0.8414	0.0384
К	5.1856	0.1101	2.0562	0.7948	0.0085	0.0660
Ca	5.3337	1.1797	0.4975	0.8175	0.0915	0.0160
Mg	0.5868	1.4546	6.0919	0.0899	0.1128	0.1955
Cu	3.9745	3.5562	3.5828	0.6092	0.2757	0.1150
Fe	5.1780	1.0437	2.5200	0.7936	0.0809	0.0809
Zn	4.5544	2.7464	2.7547	0.6980	0.2129	0.0884
Ca/Mg	6.1640	0.5284	0.1294	0.9447	0.0410	0.0042
Na/K	5.7528	0.8049	1.3606	0.8817	0.0624	0.0437

Values in **bold** for each variable correspond to the factor for which the cosine squared is the greatest.

Table 7. Contributions and squared cosines of observations

	F1	F2	F3	F1	F2	F3
F1	61.2316	13.8567	4.4906	0.8841	0.1012	0.0136
F2	2.7104	26.8649	23.1719	0.1044	0.5233	0.1868
F3	1.8798	32.9740	16.6136	0.0694	0.6159	0.1285
F4	10.3839	16.2203	35.0907	0.3559	0.2812	0.2519
F5	23.7944	10.0841	20.6332	0.6308	0.1352	0.1145

Values in bold for each observation correspond to the factor for which the cosine squared is the greatest. F1=92.5% wheat flour + 7.5% coconut flour; F2=100% wheat flour; F3=85% wheat flour +15% coconut flour; F4=77.5% wheat flour + 22.5% coconut flour; F5=70% wheat flour + 30% coconut flour.

Axis F3 is not formed by any of the observations. The formation of these different axes by variables and observations is confirmed by biplots (Figure 1). The dendograms (Figure 2) used to link formulations on the basis of their physico-chemical properties revealed that formulations F2, F3 and F4 belong to one class or have the same properties, while formulations F1 and F5 form two respective classes. These results show that formulations F3 and F4 can be used as an alternative to 100% wheat. Classification of the variables (Figure 2) revealed the existence of 6 classes. The first class is made up of Cu, Fe, Zn, Ca/Mg, Na/K, CRE/CRH and amylose/amylopectin ratios, amylose, fiber, protein, ash, water content, water retention capacity (CRE), and physical properties such as mass density and Hausner ratio. The second class is made up of swelling

ratio, oil retention capacity (ORC), porosity, carbohydrate, starch and Na contents.

Classes 3, 4, 5 and 6 are respectively formed by amylose and amylopectin content, energy value, K content and Ca content. The negative correlation (r=-0.369) between amylose content and water retention capacity (Table 8) is in line with the work of Tambo et al. (2019a,b), who demonstrated that amylose retrogrades more rapidly, leading to a reduction in water retention capacity. The positive correlation (r=0.5293) between Loose bulk density and protein content is in line with that of Tambo et al. (2019a,b). This multivariate analysis shows that the choice of formulation must take into account both its chemical and functional properties, for greater efficiency.



**Figure 1.** Biplot of correlation between contributions and variables (F1= 92.5% wheat flour + 7.5% coconut flour; F2= 100% wheat flour; F3= 85% wheat flour + 15% coconut flour; F4= 77.5% wheat flour + 22.5% coconut flour; F5= 70% wheat flour + 30% coconut flour. CRE: Water retention capacity; TG: Swelling power; CRH: Oil retention capacity; TE: Moisture content; LB: Loose bulk; PB: Packed bulk; HR: Hausner ratio.)



**Figure 2.** Dendogram that show the different classes of variables (CRE: Water retention capacity; TG: Swelling power; CRH: Oil retention capacity; TE: Moisture content; LB: Loose bulk; PB: Packed bulk; HR: Hausner ratio)



**Figure 3.** Dendogram that show the different classes of observations (F1= 92.5% wheat flour + 7.5% coconut flour; F2= 100% wheat flour; F3= 85% wheat flour + 15% coconut flour; F4= 77.5% wheat flour + 22.5% coconut flour; F5= 70% wheat flour + 30% coconut flour)

#### Table 8. Pearson correlation matrix of variables

Ode         1	Variables	CRE	TG	CRH	CRH/ CRE	pH	TE	Lipids	Porosity	LB	РВ	HR	Starch	Amyl	Amyp	Amyl/ Amyp	Proteins	Glucids	Fibers	Energy	Ashes	Na	K	Ca	Mg	Cu	Fe	Zn	Ca/ Mg	Na K
TechTec	CRE	1																												
Gene Orde <	TG	0.635	1																											
Grim Dist <	CRH	0.469	0.192	1																										
pict 0.68 0.69 0.70 <	CRH/ CRE	-0.259	-0.780	0.464	1																									
Image: Field of the state of	pH	0.463	0.658	-0.249	-0.739	1																								
Indic Out <	TE	0.331	0.594	0.748	-0.072	-0.117	1																							
Prote       0.60	Lipids	0.061	0.201	-0.669	-0.595	0.838	-0.615	1																						
Image: Disc structure Image: Disc	Porosity	-0.663	-0.842	-0.542	0.419	-0.179	-0.855	0.340	1																					
PA 0.408 0.40 0.40 0.40 <td>LB</td> <td>0.706</td> <td>0.465</td> <td>0.945</td> <td>0.186</td> <td>-0.012</td> <td>0.790</td> <td>-0.519</td> <td>-0.742</td> <td>1</td> <td></td>	LB	0.706	0.465	0.945	0.186	-0.012	0.790	-0.519	-0.742	1																				
HR 6.88 6.88 6.89 6.98 6.9	PB	-0.043	-0.638	0.416	0.853	-0.281	-0.249	-0.168	0.508	0.199	1																			
State       0.80	HR	-0.603	-0.853	-0.479	0.471	-0.179	-0.852	0.325	0.995	-0.682	0.580	1																		
And 6.00 <t< td=""><td>Starch</td><td>-0.056</td><td>0.165</td><td>0.529</td><td>0.169</td><td>-0.622</td><td>0.805</td><td>-0.882</td><td>-0.599</td><td>0.472</td><td>-0.278</td><td>-0.619</td><td>1</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>	Starch	-0.056	0.165	0.529	0.169	-0.622	0.805	-0.882	-0.599	0.472	-0.278	-0.619	1																	
And Out <th< td=""><td>Amyl</td><td>-0.369</td><td>-0.079</td><td>-0.927</td><td>-0.514</td><td>0.521</td><td>-0.748</td><td>0.876</td><td>0.563</td><td>-0.859</td><td>-0.283</td><td>0.514</td><td>-0.739</td><td>1</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></th<>	Amyl	-0.369	-0.079	-0.927	-0.514	0.521	-0.748	0.876	0.563	-0.859	-0.283	0.514	-0.739	1																
And       A	Amyp	0.369	0.079	0.927	0.514	-0.521	0.748	-0.876	-0.563	0.859	0.283	-0.514	0.739	-1.000	1															
Preter 0.402 0.402 0.404 0	Amyl/ Amyp	-0.362	-0.071	-0.926	-0.521	0.528	-0.745	0.879	0.556	-0.855	-0.287	0.507	-0.739	1.000	-1.000	1														
And <th< td=""><td>Proteins</td><td>-0.182</td><td>-0.029</td><td>0.691</td><td>0.447</td><td>-0.663</td><td>0.778</td><td>-0.901</td><td>-0.395</td><td>0.529</td><td>0.086</td><td>-0.396</td><td>0.902</td><td>-0.796</td><td>0.796</td><td>-0.798</td><td>1</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></th<>	Proteins	-0.182	-0.029	0.691	0.447	-0.663	0.778	-0.901	-0.395	0.529	0.086	-0.396	0.902	-0.796	0.796	-0.798	1													
Filters 0.10 0.81 0.81 0.82 0.70 0.02 0.70 0.02 0.70 0.02 0.70 <td>Glucids</td> <td>-0.067</td> <td>-0.323</td> <td>0.791</td> <td>0.786</td> <td>-0.778</td> <td>0.543</td> <td>-0.931</td> <td>-0.175</td> <td>0.582</td> <td>0.486</td> <td>-0.137</td> <td>0.704</td> <td>-0.888</td> <td>0.888</td> <td>-0.891</td> <td>0.877</td> <td>1</td> <td></td>	Glucids	-0.067	-0.323	0.791	0.786	-0.778	0.543	-0.931	-0.175	0.582	0.486	-0.137	0.704	-0.888	0.888	-0.891	0.877	1												
Energy 0.03 0.47 0.48 0.49	Fibers	-0.162	0.549	-0.519	-0.832	0.701	-0.009	0.664	-0.092	-0.375	-0.636	-0.156	-0.262	0.666	-0.666	0.670	-0.339	-0.725	1											
Ashes 0.178 0.188 0.498 0.498 0.498 0.059 0.198 0.498 0.499 0.	Energy	0.032	0.047	-0.416	-0.291	0.772	-0.572	0.909	0.458	-0.356	0.207	0.468	-0.921	0.715	-0.715	0.718	-0.759	-0.694	0.507	1										
Na 0.421 0.48 0.49 <t< td=""><td>Ashes</td><td>0.178</td><td>0.158</td><td>-0.562</td><td>-0.498</td><td>0.059</td><td>-0.295</td><td>0.191</td><td>-0.137</td><td>-0.364</td><td>-0.641</td><td>-0.171</td><td>-0.044</td><td>0.319</td><td>-0.319</td><td>0.320</td><td>-0.455</td><td>-0.495</td><td>0.066</td><td>-0.205</td><td>1</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>	Ashes	0.178	0.158	-0.562	-0.498	0.059	-0.295	0.191	-0.137	-0.364	-0.641	-0.171	-0.044	0.319	-0.319	0.320	-0.455	-0.495	0.066	-0.205	1									
K       0.634       0.611       0.628       0.619       0.639       0.639       0.639       0.639       0.643       0.649       0.643       0.649       0.	Na	-0.212	-0.788	0.443	0.995	-0.738	-0.113	-0.583	0.419	0.179	0.849	0.474	0.142	-0.509	0.509	-0.515	0.396	0.762	-0.873	-0.298	-0.425	1								
Ca       0.768       0.532       0.655       0.062       0.094       0.686       0.529       0.820       0.820       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.753       0.751       0	K	-0.634	-0.311	-0.628	-0.119	0.330	-0.603	0.671	0.735	-0.726	0.124	0.699	-0.643	0.801	-0.801	0.799	-0.443	-0.512	0.570	0.749	-0.285	-0.161	1							
Mg       0.556       0.192       0.071       0.209       0.059       0.052       0.108       0.123       0.034       0.035       0.151       0.132       0.035       0.038       0.842       0.123       0.641       1	Ca	0.768	0.532	0.655	-0.062	-0.094	0.686	-0.529	-0.862	0.809	-0.205	-0.825	0.574	-0.753	0.753	-0.749	0.374	0.386	-0.391	-0.609	0.238	-0.026	-0.965	1						
Cu       0.868       0.782       0.713       -0.246       0.362       0.736       -0.186       -0.876       0.899       -0.122       -0.831       0.296       -0.589       0.589       -0.582       0.235       0.184       -0.035       -0.121       -0.242       -0.653       0.819       0.252       1	Mg	0.556	0.192	-0.071	-0.209	-0.059	-0.052	-0.108	-0.367	0.123	-0.344	-0.353	0.106	-0.151	0.151	-0.149	-0.252	-0.132	-0.351	-0.388	0.842	-0.123	-0.692	0.641	1					
Fe       0.872       0.550       0.815       0.029       0.096       0.679       0.398       0.049       0.066       0.735       0.371       0.765       0.765       0.765       0.318       0.398       -0.333       -0.082       0.051       0.824       0.913       0.401       0.939       1         Zn       0.863       0.735       0.755       -0.177       0.272       0.765       -0.293       0.659       0.659       0.659       0.659       0.262       -0.123       -0.324       0.913       0.401       0.939       1       -         Ca/Mg       0.693       0.516       0.783       0.031       -0.692       0.6123       -0.6123       -0.613       0.041       0.939       0.955       0.965       1         Na/K       0.806       0.559       0.848       0.039       0.011       -0.173       0.403       0.417       -0.938       0.965       0.455       0.411       0.411       0.401	Cu	0.868	0.782	0.713	-0.246	0.362	0.736	-0.186	-0.876	0.899	-0.122	-0.831	0.296	-0.589	0.589	-0.582	0.235	0.184	-0.035	-0.152	-0.121	-0.242	-0.653	0.819	0.252	1				
Zn       0.863       0.735       -0.177       0.272       0.756       -0.273       -0.882       0.927       -0.095       -0.835       0.659       -0.653       0.293       0.262       -0.110       -0.171       -0.70       0.864       0.293       0.995       0.965       1         Ca/Mg       0.693       0.516       0.783       0.031       -0.159       0.804       0.927       -0.882       0.927       -0.835       0.659       0.659       0.659       0.293       0.262       -0.123       -0.236       -0.110       -0.171       -0.720       0.864       0.293       0.995       0.965       1         Ca/Mg       0.693       0.516       0.783       0.031       -0.159       0.890       -0.613       0.659       0.659       0.659       0.528       -0.394       -0.647       0.043       0.047       -0.938       0.966       0.862       0.966       0.492       0.911       0.052       -0.847       0.928       0.359       0.965       0.965       0.961       1         Na/K       0.806       0.559       0.848       0.029       0.910       0.766       0.491       -0.819       0.435       0.435       0.470       0.360       0.429 <t< td=""><td>Fe</td><td>0.872</td><td>0.550</td><td>0.815</td><td>0.029</td><td>0.096</td><td>0.679</td><td>-0.398</td><td>-0.796</td><td>0.949</td><td>0.066</td><td>-0.735</td><td>0.371</td><td>-0.765</td><td>0.765</td><td>-0.760</td><td>0.318</td><td>0.398</td><td>-0.373</td><td>-0.334</td><td>-0.082</td><td>0.051</td><td>-0.824</td><td>0.913</td><td>0.401</td><td>0.939</td><td>1</td><td></td><td></td><td></td></t<>	Fe	0.872	0.550	0.815	0.029	0.096	0.679	-0.398	-0.796	0.949	0.066	-0.735	0.371	-0.765	0.765	-0.760	0.318	0.398	-0.373	-0.334	-0.082	0.051	-0.824	0.913	0.401	0.939	1			
Ca/Mg 0.693 0.516 0.783 0.031 -0.159 0.809 -0.634 -0.877 0.890 -0.131 -0.839 0.674 -0.854 0.854 -0.850 0.543 0.528 -0.394 -0.647 0.043 0.047 -0.938 0.976 0.466 0.842 0.926 0.889 1 Na/K 0.806 0.559 0.848 0.039 0.021 0.769 -0.489 -0.840 0.966 0.019 -0.786 0.491 -0.819 0.819 -0.815 0.435 0.470 -0.360 -0.429 -0.111 0.052 -0.847 0.928 0.359 0.934 0.990 0.965 0.961 1	Zn	0.863	0.735	0.755	-0.177	0.272	0.756	-0.273	-0.882	0.927	-0.095	-0.835	0.358	-0.659	0.659	-0.653	0.293	0.262	-0.123	-0.236	-0.110	-0.171	-0.720	0.864	0.293	0.995	0.965	1		
Na/K 0.806 0.559 0.848 0.039 0.021 0.769 -0.489 -0.840 0.966 0.019 -0.786 0.491 -0.819 0.819 -0.815 0.435 0.470 -0.360 -0.429 -0.111 0.052 -0.847 0.928 0.359 0.934 0.990 0.965 0.961 1	Ca/Mg	0.693	0.516	0.783	0.031	-0.159	0.809	-0.634	-0.877	0.890	-0.131	-0.839	0.674	-0.854	0.854	-0.850	0.543	0.528	-0.394	-0.647	0.043	0.047	-0.938	0.976	0.466	0.842	0.926	0.889	1	
	Na/K	0.806	0.559	0.848	0.039	0.021	0.769	-0.489	-0.840	0.966	0.019	-0.786	0.491	-0.819	0.819	-0.815	0.435	0.470	-0.360	-0.429	-0.111	0.052	-0.847	0.928	0.359	0.934	0.990	0.965	0.961	1

Pearson coefficients in bold are those that are significant at the 5% level. Amyl: Amylose; Amyp : Amylopectine; CRE: Water retention capacity; TG: Swelling power; CRH: Oil retention capacity; TE: Moisture content; LB: Loose bulk; PB: Packed bulk; HR: Hausner ratio

#### 4. Conclusions

At the end of this work, which aimed to highlight the influence of substituting wheat flour by coconut flour on the nutritional and techno-functional properties of blends, it was found that substitution did not affect water and protein content. It positively improves lipid and fiber content. The energy density increased with the percentage of coconut flour added. With the exception of potassium, the 7.5% coconut flour substitution resulted in the best mineral profile and calcium availability. The functional properties (swelling rate, OHC/WRC ratio, water and oil retention capacities) were better when coconut flour was incorporated at 7.5%. In terms of physical properties, pH was similar in all flours. The porosity decreased with coconut flour percentage, while the opposite effect was observed with loose bulk density and Hausner ratio. The tapped bulk density was higher with 100% wheat flour, and the addition of coconut flour had no effect on this parameter. The principal Component Analysis (PCA) revealed a similarity between the F2, F3 and F4 formulations, while the F1 formulation was more correlated with minerals and functional properties. These results thus demonstrate the contribution of coconut flour as a substitution agent on the physico-chemical and functional properties of the resulting products.

#### Limitations

Data on the amino acid composition, rheological properties and texture of the flours obtained should be completed.

Data on breadmaking trials with these flours should also be completed.

#### **Ethics statements**

The authors have read and followed the ethical requirements for publication and confirm that the current work does not involve human subjects, animal experiments, or any data collected from social media platforms.

#### Author' contributions

Stephano Tambo Tene: Conceptualization, Methodology, Software, Writing- Original draft preparation.

#### Acknowledgments

We thanks all the team of the Research Unit of Biochemistry of Medicinal Plants, Food Sciences and Nutrition.

#### Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

#### **Declaration of interests**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## **European Food Science and Engineering**

Eur Food Sci Eng 2024, 5 (1), 20-25 doi: 10.55147/efse.1477768 https://dergipark.org.tr/tr/pub/efse

# Improving the quality of sunn pest (*Eurygaster integriceps*)-damaged wheat flour by atmospheric air cold plasma

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#### ARTICLE INFO

Research Article Article History: Received: 3 May 2024 Accepted: 11 June 2024 Available Online: 30 June 2024 Keywords: Cold plasma Improving dough rheological properties Sunn pest-damaged wheat flour

## ABSTRACT

This study aimed to improve the quality of wheat flour affected by sunn bugs (*Eurygaster integriceps*) by examining the effects of cold plasma on its physicochemical and dough rheological properties. The sun-damaged wheat flour was treated with Surface Dielectric Barrier Discharge using atmospheric air cold plasma at different application times. The results demonstrated that modified sedimentation volumes increased with longer plasma treatment times and maximized in 60 s without causing sunn pest-damage. The alveograph tenacity (P) increased with plasma application, and as a result, the energy (W) reached approximately three times that of the application of 60 s. The results showed that cold plasma has a big potential for improving sunn pest-damage flour (SPDF) and dough properties. The plasma application caused this positive effect on dough properties at  $\leq 60.0$  s.

### 1. Introduction

Plasma, the fourth state of matter distinct from gas, is an ionized medium characterized by the loss of electrons from gasphase atoms or molecules when sufficient energy is supplied, forming positively charged ions and free electrons. Plasmas are commonly found in natural phenomena such as stars, solar winds, and lightning, constituting about 99% of the universe. They can be categorized into two main types based on their energy densities and temperatures: thermal and cold plasmas. Thermal plasmas are typically found at high temperatures, often in the millions of Kelvins, and are primarily studied in fusion research and astrophysical events. On the other hand, cold plasmas operate at low temperatures are much higher than those of ion and neutral gases (Chu & Lu, 2013).

In laboratory settings, cold plasma production is achieved using various energy sources. Typically, gases such as helium, neon, and argon are ionized by applying high electrical voltages (Ilik & Akan, 2016). However, in industrial applications, atmospheric air cold plasmas, which utilize air as the gas source, offer cost and ease of production advantages. In atmospheric air cold plasmas, a wide variety of particles are generated, including electrons, ions such as  $O_2^+$  (oxygen ion),  $N_2^+$  (nitrogen ion),  $NO^+$  (nitric oxide ion),  $O^+$  (oxygen atom ion),  $N^+$  (nitrogen atom ion),  $NO_2^-$  (nitrite),  $NO_3^-$  (nitrate), molecules such as  $O_2$  (molecular oxygen),  $N_2$  (molecular

nitrogen), NO (nitric oxide), O3 (ozone), N2O (nitrous oxide), neutral atoms such as O (oxygen atom), N (nitrogen atom), radicals such as OH (hydroxyl radical), O (oxygen radical), NO (nitric oxide radical), NO2 (nitrogen dioxide radical), excited species such as O\* (excited oxygen atom), N\* (excited nitrogen atom), O2\* (excited molecular oxygen), N2\* (excited molecular nitrogen), reactive oxygen species such as O2- (superoxide anion), O<sub>3</sub> (ozone), H<sub>2</sub>O<sub>2</sub> (hydrogen peroxide), reactive nitrogen species such as NO2 (nitrogen dioxide), N2O (nitrous oxide), ONOO<sup>-</sup> (peroxynitrite), and ONOOH (peroxynitrous acid), as well as photons such as infrared, visible, and ultraviolet radiation. These species interact with the matter with which the cold plasma is in contact, leading to various surface and molecular structural changes. Plasmas have demonstrated efficacy in surface modification and coating processes, enhancing the functionality and durability of various materials in materials science and engineering (Akan et al., 2007; Musa et al., 2007). In biomedical applications, they have shown effectiveness in wound healing, blood coagulation, cancer/tumor treatment, and inactivation of bacteria, fungi, and viruses, as well as in the treatment of acne, eczema, venous ulcers, chronic leg ulcers, burn wounds, and skin regeneration (Akan et al., 2006; Fridman et al., 2008). The adverse effects of thermal treatments on foods, such as non-enzymatic browning, protein denaturation, changes in sensory properties, vitamin loss, and consumers' expectations for high-nutrient-content foods, have prompted researchers to explore non-thermal technologies. The efficacy of cold plasma technology has been

demonstrated as an alternative tool for food disinfection, shelflife extension, enzyme inactivation, toxin removal, and food packaging modifications (Barbhuiya et al., 2021; Asl et al., 2022). Cold plasma treatments have been observed to have minimal or no effect on various nutritional and sensory properties of different products (Saremnezhad et al., 2021). By pathogens effectively inactivating and spoilage microorganisms on food surfaces, cold plasma processes enhance food safety and extend shelf life. Additionally, they contribute to increased antioxidant activity in foods, slowing down oxidation processes to preserve freshness and quality. Furthermore, cold plasma treatments enhance surface disinfection of foods, thereby improving cleanliness and hygiene (Desai et al., 2024).

In the flour industry, AACP technology offers various advantages such as reducing the microbial load of flour, enzyme control, preserving nutritional value, improving functional properties, and extending shelf life. Its utilization enhances quality and safety in flour production while reducing the need for chemical additives. Further research is needed to better understand the effects of this technology on flour and baked goods, but existing findings are highly promising (Misra et al., 2015). Sunn pest (Eurygaster spp.) is a pre-harvest pest that causes occasional substantial loss of wheat both in yield and quality. The proteolytic enzymes that these insects release into the wheat grain cause the gluten proteins to break down. When damaged grains are used to make dough, the dough gets soft and sticky and sometimes disintegrates entirely. These doughs ultimately cause low-volume and poor-quality breads with defective texture and pore properties (Armstrong et al., 2019). Several methods are being used to minimize or improve sun-damaged flour's capability to be used for bread-making. These strategies include steam tempering, short-term microwave applications, using inhibitory substances (like potassium dihydrogen phosphate, sodium or calcium chloride, and sodium salicylate), and applying certain substances during the bread-making process (like bread additives like potassium bromate, L-ascorbic acid, vital gluten, DATEM, changing the conditions of the process, adding organic acids, and using sourdough) (Dizlek & Özer, 2021). These approaches can improve the rheological qualities of dough to some extent, but they cannot lead to broadly available advancements. The quality of the gluten mostly determines the viscoelastic qualities of the dough (Guzman et al., 2016). The amount of disulfide bonds (SS) and sulfhydryl groups (SH) in gluten is the main determining factor for the structure of the dough (Shewry & Jones, 2020). By oxidizing the thiol groups to disulfide bonds in sunn pest-damaged flour, the AACP approach is particularly successful at strengthening the gluten network and improving the dough's suitability for breadmaking (Tavakoli Lahijani et al., 2022). In this study, the possibility of the AACP approach to improve dough qualities with substantial sunn insect damage was examined.

#### 2. Materials and Methods

#### 2.1. Materials

The sunn pest-damaged wheat grains of the Ekiz bread wheat variety were provided by the Eskişehir Commodity Exchange. The Chopin CD1 laboratory flour mill (Chopin Technologies, Paris, France) was used to obtain refined white flour. The flour was stored in polyethylene bags and kept in a cold place ( $\sim +10$  °C) for further analysis.

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#### Plasma treatment

In this study, a surface dielectric barrier discharge (SDBD) system was used as AACP. The SDBD plasma is produced in the lid of a closed container with a volume of 1 L (Figure 1). A 1.5-mm-thick copper cable was mounted 11 times in a zig-zag pattern on the inside of the lid to serve as the energy electrode. The distance of each mounting was 10 cm. Another 1.5 mm thick copper cable was laid in a zig-zag pattern on the outer part of the lid to serve as the ground electrode, opposite to the zigzag copper cable inside. The thickness of the polypropylene lid is 2 mm. As the container's depth is 5 cm, AACP application on the flours can be accepted at a height of 4-5 cm. An alternative current (AC) high-voltage power supply with a voltage of 17.6 kV and a frequency of 12.5 kHz was used to generate cold plasma as micro-discharges (Akan & Durmus, 2023). Since no additional gas is used in this treatment, purple-colored atmospheric air plasma is generated between the inside copper cable and lid when the system is energized (Figure 1)

The AACP was applied to the flours for different durations of 10, 45, and 60 s. Optical emission spectroscopy (OES) results from cold plasma systems provide information about the reactions and chemical types of plasma. OES measurements were taken from 1-2 mm to the high-voltage electrode. The optical emission spectra of the atmospheric air cold plasma generated as micro-discharges are given in Figure 2.

The spectrum was taken with the Ocean Optics USB2000+XR1-ES device. Figure 2 displays emission spectra of the SDBD micro-discharges generated in the air, showcasing strong bands of the second positive system (SPS) of N<sub>2</sub>, including  $C^{3}\Pi_{u} \rightarrow B^{3}\Pi_{g}$  and the first negative system (FNS) of  $N_2^+$ , including  $B^2\Sigma_u^+ \rightarrow X^2\Sigma_g^+$ , in the UV spectral range. The VIS-NIR range showed characteristic sequences of bands of the first positive system (FPS) of N<sub>2</sub>, including  $B^3\Pi_g \rightarrow A^3\Sigma_u^+$ . Due to the atmospheric environment in which the SDBDs were generated, many nitrogen reactions may have occurred, leading to the presence of nitrogen ions and excited species in the micro-discharge plasmas. The atomic oxygen emission line was observed at 777 nm, and the NO molecule emission line was seen between wavelengths 200-300 nm, both of which exhibit significant chemical activities. SDBD plasmas are also known to produce high levels of ozone (Pekarek, 2012). When SDBDs have a plasma energy density between 800 and 1000 J/L, they generate more than 250 ppm of ozone (Pemen et al., 2009).

#### Physico-chemical analysis

Protein content and hardness of flours were determined using near-infrared spectroscopy (NIR 6500, Foss, Hillerød, Denmark), calibrated according to the American Association of Cereal Chemists (AACC) methods 46-19.01 and 55-31.01 (AACCI, 2010). The Single Kernel Characterization System (Perten SKCS4100) devices was used to measure the hardness values of the grain samples. The grains were then ground into flour, and the flour hardness value was established using calibration files in the NIR device. Therefore, using flour samples, the hardness value can be determined directly. The Zelenv sedimentation test was conducted according to the ICC Standard Method 116/1 (ICC, 2011). In the modified sedimentation test, as in the Zeleny sedimentation analysis, after bromphenol blue was added, the test tubes were incubated at 37 °C for 2 h. After incubation, 25 mL of sedimentation test solution was added, and the sediment volume was read after exactly 5 min. Dough rheological properties were determined by using Alveo-Link (Chopin Technologies, Paris, France) as

modified by the American Association of Cereal Chemists International (AACCI, 2010). The final water content (25 °C) was adjusted using the developed equations for 60 g of flour by Karaduman et al. (2023). After kneading for a total of 8 min with a mixer (Bastak Instruments, Ankara, Turkey), the dough was opened to the proper thickness with the standard mold of the device. The Alveo-AH test was applied to the dough samples after a rest of 20 min.

#### 2.3. Statistical analyzes

The results were evaluated with the JMP statistical program (SAS Institute, 1998). An analysis of variance (ANOVA) was applied to the flour properties in a completely randomized design with three replications. The means were compared using a Tukey's HSD test (P<0.05).



Figure 1. The system of application of AACP to the flour



Figure 2. The optical emission spectrum in the atmospheric air SDBD

#### 3. Results and Discussion

#### 3.1. Proximate chemical composition

The physico-chemical properties (alveograph) of sunn pestdamaged wheat flour treated with different times of AACP are given in Table 1. Flour protein content is an important component in determining the final product quality (Shewry, 2024). In the study, the protein contents of the flours ranged from 11.17% to 11.40%, and as was normally expected, there was no variation between the flours when exposed to prolonged cold plasm. Ultimately, there should be positive developments in protein quality, which determines the final product quality rather than the protein content. Sedimentation tests can potentially help characterize the overall protein quality because of the ease of the measuring methods (Tömösközi et al., 2009). A high sedimentation volume indicates high gluten quality (Guzman et al., 2016). The average sedimentation volume of SPDF in the study was 34.50 mL, however, when the modified sedimentation test was used, it decreased to 12.50 mL because the sunn pest protease activity broke down the peptide linkages in the gluten. When the AACP was applied to SPDF for increasing periods, normal sedimentation volumes were close to each other, and modified sedimentation volumes increased linearly. After being treated with AACP for 60 s, it reached to a level where sunn pest-damaged does not occur (30.33 mL). One of the interesting findings in the study is that the hardness value increased with plasma application compared to the control flour at all application times. The degree of protein adherence to starch and the integrity of the protein matrix are related to hardness (Hrušková & Švec, 2009). The hardness of flour seems to be increased by the application of plasma, which strengthens the protein matrix and enhances especially

intramolecular and intermolecular disulfide linkages in gluten.

The dough rheological properties (alveograph) of sunn pestdamaged wheat flour treated with different times of AACP are given in Table 2. Alveograph parameters are frequently used to determine the dough rheological properties of flours (Dubois et al., 2008). The length of the obtained curve between the start and end points represents its extensibility (A value), its height represents its elasticity (P value) (Jødal & Larsen, 2021), and its area represents the total energy required to explode (W value). The gluten balance value is obtained by dividing the P value by the L value (Guzman et al., 2016). A high energy (W) value and a P/L (gluten) of roughly 1.0-1.3 are required for viscoelastic dough having excellent gluten quality and high bakery quality (Jødal & Larsen, 2021). Once more, the breakdown of the gluten networks by the enzyme resulted in a considerable decrease in the tenacity (P), energy (W), and gluten balance (P/L) of the SPDF dough in the alveograph. The W value increased statistically in each AACP treatment, increasing from 51.67 10<sup>-4</sup> x J to 137.67 10<sup>-4</sup> x J in 60 s (r around 1.5 times in 10 s, twice in 45 s, and 3 times in 60 s). In the study, another important factor is that the gluten balance (P/L), which establishes the properties of flour at very close amounts of protein that are used for the production of the end product (Guzman et al., 2016), progressively increases from the mean level of soft wheat products (0.5) to the level that is necessary for producing bread (1.0-1.3). The AACP treatment strengthened the gluten network and enhanced the dough's ability for bread-making by oxidizing the thiol groups in SPDF to disulfide bonds (Tavakoli Lahijani et al., 2022).

The tenacity (P) values of the flours also increased from 30.0 mm to 70.0 mm with increasing plasma application time and the elasticity of gluten increased.

Plasma treatment time (s)	Protein content (%)	Flour hardness value (HI)	Sedimentation volume (mL)	Modified sedimentation volume (mL)
Control	$11.38{\pm}0.04^{a}$	43.44±0.90°	34.50±0.50ª	$12.50 \pm 0.50^{d}$
10	$11.17{\pm}0.07^{b}$	58.18±1.22ª	31.67±0.29 <sup>b</sup>	20.50±0.50°
45	$11.27{\pm}0.05^{ab}$	54.61±1.11 <sup>b</sup>	31.83±0.29 <sup>b</sup>	25.67±0.58 <sup>b</sup>
60	$11.40{\pm}0.07^{a}$	$55.27 \pm 0.59^{b}$	28.33±0.58°	30.33±0.29ª
Mean	11.31	52.87	31.58	22.25
LSD0.05	0.15**	2.54**	1.33**	1.39**

Table 1. The physico-chemical properties of sunn pest-damaged wheat flour treated with different AACP treated times

The means of flour properties in the same column marked with different lowercase letters are statistically different from each other (P<0.05). The significance between flour properties at the 1% level is indicated by two asterisks (\*\*); Control: sunn pest-damaged flour (SPDF); HI: Hardness index

Table 2. The alveograph properties of sunn pest-damaged wheat flour treated with different AACP treated times

Plasma treatment time	Alveograph tenacity	Alveograph extensibility	Alveograph energy	Alveograph P/L value
<u>(s)</u>	( <b>P</b> , mm)	(L, mm)	$(W, 10^{-4} x J)$	
Control	$30.67 \pm 0.58^{\circ}$	$57.33 {\pm} 0.58^{b}$	$51.67 \pm 1.53^{d}$	$0.535{\pm}0.009^{d}$
10	$46.33 \pm 0.58^{b}$	51.67±0.58°	74.33±1.15°	$0.897{\pm}0.011^{b}$
45	$48.33 \pm 0.58^{b}$	68.00±1.00 <sup>a</sup>	91.33±1.53 <sup>b</sup>	0.711±0.005°
60	$68.67 \pm 1.15^{a}$	$53.00 \pm 0.00^{\circ}$	137.67±1.53ª	1.296±0.022ª
Mean	48.50	57.50	88.75	0.860
LSD0.05	2.49**	1.56**	4.19**	0.039**

The means of flour properties in the same column marked with different lowercase letters are statistically different from each other (P<0.05). The significance between flour properties at the 1% level is indicated by two asterisks (\*\*); Control: sunn pest-damaged flour (SPDF)

#### 4. Discussion

Atmospheric pressure cold air plasma generates reactive species that can interact with the components of flour, leading to various chemical modifications. Flour primarily consists of starch (the main carbohydrate component, primarily composed of glucose polymers), proteins (includes glutenin and gliadin, which together form the gluten network), lipids (composed of phospholipids, triglycerides, and free fatty acids), and a small amount of minerals (present in small quantities as inorganic compounds). The reactive oxygen species (ROS) and reactive nitrogen species (RNS) produced by plasma can react with the side chains of amino acids in proteins, causing oxidative modifications. This can lead to cross-linking, oxidation, or degradation of proteins. Amino acids such as methionine, cysteine, tyrosine, and tryptophan are particularly susceptible to these reactive species, resulting in structural changes in the proteins. When gluten proteins undergo oxidation, disulfide bonds may break or new ones may form, leading to alterations in the gluten network's elasticity and viscoelastic properties. Starch, a polymer of glucose units, can also be affected by ROS and RNS through depolymerization or oxidation, causing the breakdown of starch molecules. This impacts the gelatinization and water-holding capacity of starch. Lipids can undergo peroxidation due to ROS, resulting in oxidative degradation and the formation of free radicals. Lipid oxidation affects the shelf life and flavor of the flour. Additionally, electrons and anions (such as sulfide or oxygen anions) produced by AACP can interact with specific regions of proteins or starch, causing chemical modifications. Electrons can break specific bonds in proteins or starch chains or form new bonds. Ultraviolet (UV) radiation generated by AACP can induce photochemical reactions in proteins and starch. leading to photo-oxidation and structural changes. UV radiation can also damage the DNA of microorganisms, reducing the microbial load in the flour. These modifications can affect the rheological and functional properties of the flour. Changes in the gluten structure can alter the dough's elasticity, viscoelasticity, and consequently its behavior in bread-making processes (Thirumdas et al., 2012; Menkovska et al., 2014; Pankaj et al., 2014; Misra et al., 2016). The treatment of AACP with an oxidation-reduction potential increases the formation of disulfide bonds between glutenin proteins, hence enhancing the strength of the dough (Sandhu et al., 2011; Menkovska et al., 2014). In the study, gluten quality increased by encouraging the formation of gluten disulfide bonds by applying AACP to sunn pest-damaged flour. This situation was reflected in the sedimentation values, and even in 10 s application, the sedimentation value increased approximately twice. With 60 s of application, the sedimentation level with no sunn pest-damage was reached. It was thought that with plasma application, there was an improvement in the structure of the gluten protein, especially due to the development of intermolecular disulfide bonds, and the sedimentation values of the flour particles increased by absorbing more water. The increase in hardness value supported this, and the bonds increased by oxidation in the gluten structure strengthened the gluten and increased the hardness of the flour. The extent to which this improvement in the physicochemical properties of the flour is reflected in the properties of the dough is extremely critical before obtaining the product. In the study, the effect of AACP application on dough properties was determined by an alveograph, which is the most common test used for this purpose. The high alveograph energy (W) value is the basic parameter that determines the suitability of the dough for bread-making. The study showed that the alveograph energy value increased with

each dose and that AACP application was very effective in strengthening the dough and improving its viscoelastic properties. The increase in the dough alveograph energy value sometimes occurs with a high tenacity (P) value, and the extensibility (L) value required for a volume increase in a bakery decreases. In other words, highly elastic gluten emerges, and the resulting breads are low in volume. This situation can be seen with the gluten balance (P/L value) on the alveograph. A high P/L value (>1.3) creates a highly elastic (tenacious) dough, while a small P/L value (<0.8) creates a highly extensible dough. With its high energy value, this ratio is desired to be between 1.0 and 1.3 in bread making. In our study, the positive improvement in P/L value with the increased energy value with plasma application and reaching optimum limits in 60 s were found valuable. As a result, plasma application improved the dough's rheological properties in a very positive way. The most important point in the study is that this positive effect on dough properties was obtained at  $\leq 60.0$ s with the plasma application system developed differently from the literature.

#### 5. Conclusions

The results show that with increasing time of plasma treatment, modified sedimentation volumes increased and reached a level that did not cause any sunn pest-damage in the 60 s of treatment. The AACP application increased the tenacity (P) of the dough, which had intense sunn pest-damage. Thus, its energy value (W) increased approximately three times, and the gluten balance (P/L) reached the desired level in the application of 60 s. As a result, it was seen that the SPDF dough has gained better viscoelastic properties with the application of AACP.

#### **Ethics statements**

The authors have read and followed the ethical requirements for publication and confirm that the current work does not involve human subjects, animal experiments, or any data collected from social media platforms.

#### Author' contributions

**Arzu Akın:** Collected the data, Investigation, Data curation, Formal analysis, Conceptualization, and Writing - original draft.

Yasar Karaduman: Supervision, Collected the data, Investigation, Data curation, Formal analysis, Conceptualization, and Writing - the original draft.

**Tamer Akan:** Collected the data, Investigation, Data curation, Formal analysis, Conceptualization, Writing - review & editing.

#### Acknowledgments

The authors greatly thank the Eskişehir Commodity Exchange Presidency for supplying sunn pest-damaged wheat grains.

#### Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

#### **Declaration of interests**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## **European Food Science and Engineering**

Eur Food Sci Eng 2024, 5 (1), 26-34 doi: 10.55147/efse.1481485 https://dergipark.org.tr/tr/pub/efse

## Development of a new vegan muffin formulation: Assessing its quality and sensory characteristics

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#### ARTICLE INFO

Research Article Article History: Received: 9 May 2024 Accepted: 22 June 2024 Available Online: 30 June 2024 Keywords: Vegan-muffin Aquafaba Black chickpea flour Lactose-free Sensory attributes

#### ABSTRACT

This study aimed to develop a healthy and lactose-free muffin containing different levels of black chickpea flour (0%, 50%, 75%, and 100%, w/w) in the formulation while maintaining sensory appeal. Four different formulations were developed: while the control muffins contain cow milk, chicken egg, and wheat flour, the other three formulations include almond milk, aquafaba, and black chickpea flour at replacement ratios of 50% (M-1), 75% (M-2), and 100% (M-3), respectively. Results showed pH values ranging from 6.45 to 6.95 for batter and 6.76 to 7.10 for baked muffins, with dry matter content between 63.71% and 65.54%, and baking loss between 8.89% and 12.22%. Calorie values were highest in M-0 (330.69 kcal/100 g), reduced to 272.83-269.72 kcal/100 g with the addition of chickpea flour, aquafaba, and almond milk. Muffin height and volume decreased insignificantly in M-1, M-2, and M-3 compared to reference muffins (P>0.05). The uniformity index, volume, symmetry index, and volume index significantly decreased with chickpea flour addition (P<0.05). Sensory evaluation showed no statistically significant differences in overall acceptance among muffin samples (P>0.05). Overall, this demonstrates the potential to create sensorially pleasing vegan muffins by replacing traditional ingredients with alternatives like black chickpea flour, aquafaba, and almond milk.

#### **1. Introduction**

The quality of processed foods is directly impacted by the raw ingredients utilized. Coupled with advancements in processing technology, there has been a notable increase in processed food manufacturing. Bakery products constitute a substantial proportion of overall food consumption, with soft bakery items particularly favored despite their limited shelf life due to their delectable taste. Muffins, in particular, garner special attention due to their diverse combinations of nutritious components and sensory attributes (Dizlek, 2015; Ali et al., 2023; Shukla et al., 2024).

Chickpea (*Cicer arietinum* L.) is an annual leguminous plant belonging to the *Fabaceae* family (Rachwa-Rosiak et al., 2015). Chickpeas are valued for their high carbohydrate and protein content, with their protein quality often regarded as superior to that of other legumes (Hirdyani, 2014). When contrasted with conventional chickpeas, black chickpeas are distinguished by their black outer coat, smaller size, and irregular, wrinkled shape. They are abundant in proteins, fibers, and bioactive compounds. Several recent research findings indicate that black chickpeas hold significant promise for the creation of functional food formulations (Yaver, 2022). It holds

significant promise within the consumer market, being suitable for a wide range of products such as baked goods, snacks, soups, and ready-to-eat foods (Kumar et al., 2020).

Eggs play crucial roles in food preparation due to their abilities in gelation, foaming and emulsification. They represent one of the most widely utilized food components across the globe, making them indispensable in a variety of bakery items (Mustafa et al., 2018; Boukid & Gagaoua, 2022). However, in recent times, there has been a surge in interest towards plant-based proteins as a substitute for animal-derived proteins. This trend stems from the growing vegetarian market. According to a recent report by Bloomberg Intelligence (2021), the plant-based protein market is projected to comprise up to 7.7% of the global protein market by 2030, with an estimated value exceeding \$162 billion, a significant increase from \$29.4 billion recorded in 2020 (Kim et al., 2022). The rise of plantbased food ingredients and products mirrors a growing effort to replicate and substitute animal-derived sources like meat, milk, and eggs. This shift aligns with the considerable expansion of vegetarian and vegan markets, driven by consumer desires for healthier and more environmentally friendly food options. Individuals are increasingly open to modifying their dietary habits and embracing accountability for climate change by minimizing their carbon emissions. This entails opting for

plant-based alternatives over animal-based ones (He et al., 2021; Raikos et al., 2021; Ozcan et al., 2023).

Aquafaba, originating from the Latin words "aqua" meaning water and "faba" representing the Fabaceae family, is recognized as a valuable ingredient with functional capabilities like foaming, emulsifying, and gelling. Studies and practical applications demonstrate its potential as a substitute for eggs and milk in vegan products, offering diverse formulation options (Buhl et al., 2019; Bekiroglu et al., 2023; Viana et al., 2023; Erem et al., 2023; Erem et al., 2024). It's a viscous liquid typically extracted and discarded following the cooking of legumes, with chickpeas being the most commonly utilized source (Shim et al., 2018; Mustafa & Reaney, 2020; Echeverria-Jaramillo & Shin, 2023). Aquafaba is used in various scientific reports for the preparation of vegetable-based origin mayonnaise (Raikos et al., 2020; He et al., 2021), scrambled eggs (Dever, 2016), merengues (Fuentes-Choya et al., 2023; Tufaro & Cappa, 2023), cakes & muffins (Mustafa et al., 2018; Aslan & Ertaş, 2020; Sengar, 2021; Grossi Bovi Karatay et al., 2022; Edleman & Hall, 2023), cookies (Edleman & Hall, 2023), and mousses (Mehren et al., 2023). However, to the best of our knowledge, there is no study evaluating the effects of black chickpea flour, almond milk and aquafaba.

#### 2. Materials and methods

#### 2.1. Materials

Black chickpea flour (Cey Natural Foods, Istanbul, Türkiye), almond milk (Nilky Beverage and Food Industry and Trade Inc., Istanbul, Türkiye), sugar (Irmak Şeker, Ismen Food Company, İstanbul, Türkiye), commercial chickpea cans (Yayla Agro Food Industry and Transportation Inc., Mersin, Türkiye), sunflower oil (Yudum oils, Balıkesir, Türkiye), walnut (Peyman Company, Balikesir, Türkiye), cacao powder (Dr. Oetker, İzmir, Türkiye), baking powder (Dr. Oetker, Izmir, Türkiye), vanilla sugar (Dr. Oetker, İzmir, Turkiye), cinnamon powder (Bağdat Baharat, Ankara, Turkiye), UHT whole-fat cow milk (Icim, Ak Food Co., Sakarya, Türkiye), wheat flour (Söke flour, Söke Milling Industry and Trade Inc., Aydın, Türkiye) were obtained. Also, carrots and chicken eggs were purchased from a local market.

#### 2.2. Aquafaba production

To prepare aquafaba, commercial chickpea cans were first drained using a sieve. Then, 0.5 grams of salt was added to the drained chickpea water, and it was whisked with a hand mixer for 4 min until it reached a foam consistency (similar to whipped cream, rather than stiff peaks) (Mustafa et al., 2018).

#### **2.3. Muffin production**

The block flow diagram illustrating the production process of muffins is depicted in Figure 1. At first, the sugar and chicken eggs/aquafaba were mixed in a mixing bowl for 7 min using a stainless-steel wire whisk until it reached a smooth and consistent texture. Once the sugar and egg/aquafaba mixture was ready, sunflower oil and milk were slowly added into the bowl. Then, the ingredients were thoroughly mixed for 3 min to ensure they were evenly incorporated. The grated carrot, chopped walnuts, and cinnamon were added and mixed for 1 min. Subsequently, cocoa, baking powder, vanilla, wheat flour and/or chickpea flour were added and mixed for 2 min until a uniform batter was formed. The mixture was portioned into the equal sizes (60 g portions) to promote consistent baking. Portioned batter into the muffin molds (Dolphin GG-muffin cake capsule with paper surface covered with PET film, China) were baked in the preheated countertop electric mini oven (SUF 4000 MEB, Arçelik, Türkiye) at 180 °C for 22 min. Following this procedure, 4 different muffin samples were produced, and their recipes were provided in Table 1. Also, the block flow diagram was used to produce muffins. The muffin samples, including egg (11.1%), cow milk (19.4%), and wheat flour (22.2%), were named as M-0. Meanwhile, the muffin formulations containing aquafaba (11.1%), almond milk (19.4%), and wheat flour: chickpea flour (11.1 g:11.1 g for M-1, 5.6 g:16.7 g for M-2, and 0 g:22.2 g for M-3) were coded as M-1, M-2, and M-3, respectively. Finally, the images of the produced muffins were given in Figure 2.

#### 2.4. Chemical analysis

#### pH analysis

The pH values of the samples were determined using a pH meter (Ohaus AB23PH-F, China) calibrated with appropriate buffer solutions prior to analysis. 10 grams of sample was weighed using an analytical scale (readability: 0.001 g; PLJ 1200-3A-2020a, Kern & Sohn GmbH), 30 mL of distilled water was added and homogenized using a high-speed laboratory homogenizer (Model D-160, hand-held homogenizer, DLAB Scientific Co., Ltd., Beijing, China) for 3 min. Then, pH measurements and temperatures of the upper part were determined by immersing the probe into the samples (Elgün et al, 2012).

#### Dry matter analysis

The dry matter content of muffin samples was ascertained by using the AOAC (1990) standard technique. About 4 g of muffin samples were weighed into the weighing containers using an analytical scale (readability: 0.001 g; PLJ 1200-3A-2020a, Kern & Sohn GmbH). After they were constricted and brought to consistent weight, they were dried in a drying oven (WGL-65B, Tianjin Test Instrument Co., Ltd., Tianjin, China) at 105 °C for 3 h. Following the procedure, they were brought to the desiccator. Using the following formula and the computed dry matter percentages, the weights of the samples that had been cooled to room temperature were ascertained.

Dry matter percentage of the sample (% = ((Dry sample after drying + weighing container tare)–(Weighing container tare)

(( )	I I I I I		. 0	0	,	(	0	,
	((Sample	+ weighin	g conta	iner tare)	–(Weighi	ng conta	iner tare))	
100							(1	1)

#### Calculation of caloric value

The specific amounts of protein and carbohydrates are multiplied by the factor of 4 kcal/g, while the amount of fat is multiplied by the factor of 9 kcal/g, and as a result of these calculations, the calorie values of muffin samples determined by examining the labels of all brands individually have been calculated. The results are presented as kcal/100g.

#### 2.5. Physical analyses

#### The weights of produced muffins

The weights of the produced muffins were determined in grams by weighing them on a sensitive scale after they reached room temperature.



Figure 1. The block flow diagram used to produce muffins.

Table 1. The recipes utilized in the manufacturing of M-0, M-1, M-2, and M-3.

L		Muffin sa	mples	
Ingredients (%)	M-0	M-1	M-2	M-3
Wheat flour	22.2	11.1	5.6	-
Black chickpea flour	-	11.1	16.7	22.2
Milk	19.4	-	-	-
Almond milk	-	19.4	19.4	19.4
Carrot	16.7	16.7	16.7	16.7
Egg	11.1	-	-	-
Aquafaba	-	11.1	11.1	11.1
Sugar	11.1	11.1	11.1	11.1
Sunflower oil	8.3	8.3	8.3	8.3
Walnut	8.3	8.3	8.3	8.3
Cacao powder	1.9	1.9	1.9	1.9
Vanilla powder	1.4	1.4	1.4	1.4
Baking powder	1.4	1.4	1.4	1.4
Cinnamon	0.8	0.8	0.8	0.8



Figure 2. The images of produced muffins.

#### Muffin volume

The volume values of muffins were determined based on the displacement principle of rapeseed (AACC, Method 10-05.01). Rapeseed was poured into the container at a constant speed and distance to determine the empty volume of the container. Then, the seeds in the container were transferred to a graduated measuring cylinder to determine the container volume (V<sub>1</sub>). Afterwards, muffin samples were placed into the container, and the seeds with volume V<sub>1</sub> were emptied onto them, and the top of the container was leveled using a ruler. The value obtained by transferring the seeds overflowing from the container to the graduated measuring cylinder was recorded (V<sub>2</sub>). The value read from the graduated measuring cylinder where the overflowing seeds were placed provided the muffin volume value in milliliters (V<sub>2</sub>).

#### Muffin specific volume

The specific volume values of the muffin samples (mL/g) were calculated by dividing the muffin volume by the muffin weight (Lin et al., 2017).

$$Specific \ volume = \frac{Muffin \ volume}{Muffin \ weight}$$
(2)

#### Muffin density

The density values of the produced muffins (g/cm<sup>3</sup>) were calculated by dividing the muffin weight by the muffin volume (Gómez et al., 2008).

$$Density = \frac{\text{Muffin weight}}{\text{Muffin volume}}$$
(3)

#### Baking loss

The baking loss values of the produced muffins were calculated as a percentage using equation (Rodríguez-García et al., 2012).

$$Baking \ loss \ (\%) = \frac{(Batter \ weight) - (Muffin \ weight)}{(Batter \ weight)} * 100$$
(4)

#### Muffin height

The height of the muffin samples was calculated by determining the highest point of the center section of the muffin and dividing it vertically with a knife. Measurements were then made with a digital caliper (Piranha PDC 1850 Digital Caliper, China) with an accuracy of 0.1 mm based on the highest point of the sample (Martínez-Cervera et al., 2012).

# Determination of uniformity index (UI), upside shrinkage value (USV), symmetry index (SI), volume index (VI), and shrinkage value (SV)

VI, SI, UI, SV, and USV (shrinkage occurs on the upside portion of the muffin) of muffin samples were determined using a layer cake measurement template following the AACC 10-91.01 method, with measurements expressed in millimeters as detailed in equations (5,6,7,8, and 9; respectively). The layer cake measurement template was appropriately adjusted for a single muffin baking cup, with dimensions of 50 mm bottom diameter, 70 mm top diameter, and 35 mm height as previously modified by Dizlek (2015). In the modified muffin measurement template, the length of the template was 70 mm, and point C indicated the center. Points B and D were positioned 21 mm from both the left and right sides of the center, while points A and E were located 35 mm from both sides. Finally, the heights |BB'|, |CC'|, and |DD'|, and lengths |AE| and |A'E'| were measured from the template and utilized for the calculation of the index and shrinkage values, respectively.

To calculate the index values according to AACC Method 10–91.01, the height of the muffin was determined to the nearest 1 mm at vertical lines B, C, and D. For determining muffin SV and USV, the diameter was determined (from A to E and A' to E', respectively) to the nearest 1 mm. Then, the diameter was deducted the from 50 mm to find SV (Eq. 8), and subtracted from 70 mm to find USV (Eq. 9).

$$VI(mm) = |BB'| + |CC'| + |DD'|$$
(5)

SI(mm) = 2|CC'| - |BB'| - |DD'| (6)

$$UI(mm) = |BB'| - |DD'| \tag{7}$$

SV(mm) = 50 mm - |AE|(8)

USV(mm) = 70 mm - |A'E'| (9)

#### 2.6. Sensory analysis

Sensory analyses were carried out by 15 female and 7 male panelists aged between 20-42 years, semi-educated, consisting of Ankara Medipol University Department of Gastronomy and Culinary Arts lecturers and third-year undergraduate students who had taken the Food Formulation and Sensory Analysis course. In these analyses, the samples were presented to the panelists at room temperature for sensory analysis. The order of presentation of the samples varied among the panelists in order not to influence their choices. Each sample was cut into two equal parts with a stainless-steel knife (Pirge ecco, 26 cm, chef knife, Türkiye). The samples were served to the panelists in plastic plates numbered (M-0:289, M-1:675, M-2:378, and M-3:743) for sensory evaluation. In addition, water was given to the panelists in disposable plastic cups as a palate cleanser during the sensory analyses. The sensory characteristics were determined by 11 different sensory parameters (crust & crumb color, softness, crumbliness, moistness, elasticity, porosity, odor, taste, volume, and overall acceptability) using a 9-point hedonic scale (1, not at all like; 9, very much like) (Yalcin et al., 2021).

#### 2.7. Statistical evaluation

JMP 6.0 statistical analysis software (SAS Institute, Inc., Cary, NC, USA) was utilized for performing one-way ANOVA. Additionally, Student's t-test was employed to evaluate the impacts of independent variables, including pH, dry matter, physical, and sensory analysis (*e.g.* crust & crumb color, crumbliness, softness, moistness, volume, elasticity, porosity, odor, taste, and overall acceptability). The results were analyzed at a significance level of P<0.05.

#### 3. Results and Discussion

#### 3.1. Physicochemical properties

The batter pH values were measured between 6.45 and 6.95 (Table 2). However, despite minor differences in the results, these variations were found to be insignificant (P>0.05). The pH values of the muffin samples ranged between 6.76 and 7.10 (Table 2). The highest pH was observed in the M-0 samples, while the lowest pH was determined in the M-2 samples. Additionally, the effect of changes in the formulation on pH was found to be statistically significant (P<0.05). In other words, variations in the ratios of the flours in the formulation significantly affected the pH values (P<0.05). The addition of black chickpea flour instead of wheat flour resulted in a decrease in pH. The pH values of flour in water suspension are crucial as certain functional properties, primarily associated with protein, such as emulsion properties and nitrogen solubility, are greatly influenced by variations in pH (Alvarez et al., 2017). Substituting wheat flour with pulse flour resulted in a higher density of cake batters. This change may be attributed to the functional properties of bean flour proteins, such as foam stability and emulsification (Singh et al., 2015). Similarly, Mustafa et al. (2018) reported that the pH of sponge cake manufactured with egg white was higher than that manufactured with aquafaba.

The dry matter values of M-0, M-1, M-2, and M-3 were determined to be 65.54%, 64.81%, 63.71%, and 65.50%, respectively (Table 2). The dry matter content of M-0 samples was slightly higher compared to the other samples; however, statistically, this difference was not significant (P>0.05). In other words, replacing chicken eggs with aquafaba, black chickpea flour with wheat flour, and cow milk with almond milk did not alter the moisture level of the prepared muffins. Similarly, the muffin enriched with chickpea flour has a lower pH and higher moisture content compared to muffins incorporated with wheat flour (Alvarez et al., 2017). Increased moisture levels may be linked to greater water absorption, attributable to the existence of two separate sources of protein and starch in these batters (Alvarez et al., 2017).

The reduction in bake loss holds significance in industrial settings as it directly impacts yield and product weight, with lower bake loss correlating to higher yields. Bake loss occurs due to the evaporation of water from the product during baking, a process influenced by the water retention capability of the ingredients used (Grasso & Methven, 2020). The baking loss values for muffins were found in descending order as follows: M-3 (12.22%) > M-0 (10.56%) > M-2 (10.00%) > M-1 (8.89%)

as shown in Table 2. Differences in baking loss percentages between muffin formulations were found to be significant (P<0.05). Due to lower baking loss in M-1 samples among the muffin samples, they had a higher yield.

The calorie contents of the muffins were provided in Table 2 and these values ranged from 269.72 to 330.69 kcal/100 g. The presence of chicken egg in the formulation (for M-0) increased the calorie content compared to muffins containing aquafaba (M-1, M-2, and M-3). Compared to the control samples, the reformulated vegan muffin samples showed a lower energy value, reaching a decrease in the range of 17.50% to 18.4% compared to the control muffins. Despite this quantitatively significant decrease, it was not sufficient for the product to claim "reduced energy" according to Regulation (EC) No 1924/2006 of the European Parliament and of the Council, which requires a minimum decrease of 30% (Anserona et al., 2022).

#### 3.2. Physical attributes

The height values of the muffins vary between 35.07 and 36.11 mm (Table 3). Height was lower in muffins enriched with black chickpea flour. However, the differences in height values were not found to be statistically significant (P>0.05).

The volume values of the muffins for M-0, M-1, M-2, and M-3 were measured as 120.58, 102.52, 95.83, and 96.00 mL, respectively (Table 3). The differences between the measured values for M-1, M-2, and M-3 were found to be insignificant (P>0.05). The measured results between M-1/M-2/M-3 and control muffin samples were found to be significant (P < 0.05). Several interconnected factors contribute to the final volume in baking: the rheological characteristics of the batter (which are influenced by the ingredients used), the degree of air integration, and the duration and speed of mixing and homogenization (Martínez-Cervera et al., 2012). Chickpea flour exhibited elevated protein content as well as distinct amino acid composition compared to wheat flour, factors that could potentially influence cake attributes, particularly its volume (Gómez et al., 2008). The notable decrease in muffin volume indicates a reduced amount of air retained within the cake during baking and generated denser muffins (Gómez et al., 2008; Ahmad et al., 2021; Sunwar, 2022). This could be attributed to the inability of black chickpea flour and/or aquafaba to effectively enhance air retention and promote batter aeration. The other reason could also be attributed to the elevated fiber content in chickpea flour, which potentially limits water availability for the formation of the starch-protein network during baking. Consequently, this could lead to a reduction in muffin volume (Herranz et al., 2016).

The specific volume values of the samples varied in the range of 1.77 (for M-2) to 2.25 mL/g (for M-0) (Table 3). While the differences between M-1, M-2, and M-3 samples were not statistically significant (P>0.05), the differences between the mentioned samples and the M-0 samples were found to be significant (P<0.05). Likewise, Herranz et al. (2016) observed a notable reduction in specific volume measurements in gluten-free muffins made with chickpea flour.

According to the calculated findings, the density values of the samples were determined to be in the range of 0.45-0.56 g/mL (Table 3). Since density and volume were inversely related, the M-2 muffin had the highest density. The results between M-1, M-2, and M-3 samples were found to be statistically insignificant (P>0.05). However, the differences between these samples and M-0 were found to be significant (P<0.05).

The VI values, which gave clues about the overall dimensions of the muffin (Dizlek, 2015), were ranked from smallest to largest in the tested samples as follows: M-2 (9.38 mm) < M-3 (9.68 mm) < M-1 (10.00 mm) < M-0 (12.33 mm) and these differences were statistically significant (P<0.05) (Table 3).

SI values above zero suggested an expansion in the center of bakery products, which was a desirable trait in muffin samples. Conversely, negative values indicate a concavity in the center (Moreira et al., 2023). SI values of the muffins were calculated to range from -0.14 to 1.08. Specifically, the SI values were determined as 1.08 for M-0, 0.58 for M-1, 0.54 for M-2, and -0.14 for M-3 (Table 3). While the differences between M-0 and M-1 were not found to be statistically significant (P>0.05), it was observed that the differences in SI values among the other samples were statistically significant (P<0.05).

Table 2. pH,	dry matter, a	and baking loss	content of muff	ïn samples.
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Samples	pH of batter	pH of muffin	Dry matter (%)	Baking loss (%)	Calorie content (kcal/100g)
M-0	6.95±0.01ª	7.10±0.01ª	$65.54 \pm 3.10^{a}$	10.56±0.96 <sup>b</sup>	330.69
M-1	6.81±0.01ª	6.94±0.01°	$64.81 \pm 4.34^{a}$	$8.89 \pm 0.96^{\circ}$	272.83
M-2	$6.45 \pm 0.59^{a}$	$6.76 \pm 0.01^{d}$	63.71±4.42 <sup>a</sup>	$10.00 \pm 0.00^{bc}$	271.28
M-3	$6.77 \pm 0.02^{a}$	$6.96 \pm 0.02^{b}$	$65.50 \pm 3.34^{a}$	$12.22 \pm 0.96^{a}$	269.72

Columns labeled with distinct letters (*e.g.*, a, d) indicate statistically significant differences (P<0.05)

**Table 3.** The influences of different muffin formulations on the muffin quality.

Properties	<b>M-0</b>	M-1	M-2	M-3
Height (mm)	36.11±3.46 <sup>a</sup>	36.09±3.47 <sup>a</sup>	35.07±1.17 <sup>a</sup>	35.56±2.19 <sup>a</sup>
Volume (mL)	$120.58 \pm 3.76^{a}$	102.52±9.01 <sup>b</sup>	$95.83 \pm 5.64^{b}$	96.00±12.17 <sup>b</sup>
Specific volume (mL/g)	$2.25 \pm 0.06^{a}$	1.88±0.15 <sup>b</sup>	$1.77 \pm 0.09^{b}$	$1.82{\pm}0.02^{b}$
Density (g/mL)	$0.45 \pm 0.01^{b}$	$0.54{\pm}0.05^{a}$	$0.56{\pm}0.03^{a}$	$0.55{\pm}0.07^{a}$
Volume index (mm)	12.33±0.45 <sup>a</sup>	$10.00 \pm 0.05^{b}$	9.38±0.24°	$9.68 \pm 0.52^{bc}$
Symmetry index (mm)	$1.08 \pm 0.43^{a}$	$0.58{\pm}0.63^{a}$	$0.54{\pm}0.87^{ m ab}$	-0.14±0.22 <sup>b</sup>
Uniformity index (mm)	$0.04{\pm}0.37^{ab}$	-0.23±0.09b	-5.42±1.20 <sup>b</sup>	15.25±2.12 <sup>bc</sup>
Shrinkage value (mm)	$-2.42\pm1.56^{a}$	0.21±0.31ª	$-3.75 \pm 1.94^{ab}$	19.33±3.39 <sup>a</sup>
Upside shrinkage (mm)	13.08±1.86°	-0.14±0.22 <sup>b</sup>	-3.18±1.82 <sup>a</sup>	17.33±1.75 <sup>ab</sup>

Rows labeled with distinct letters (*e.g.*, a, d) indicate statistically significant differences (P<0.05)

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Correspondingly, Gomez et al. (2008) reported that sponge cakes with chickpea flour addition have less symmetry and consequently, less gas retention capacity and final volume. The computed UI values, serving as an indication of cake symmetry, varied between -0.23 and 0.21 mm (Table 3), with statistically significant differences between them (P<0.05). For a perfect cake, this index is ideally recorded as zero, because positive or negative values happen when one side of the cake is raised above the other (Dizlek et al., 2008).

The SV values of the muffins were calculated negatively, with the smallest value determined for M-1 (-5.42 mm) and the highest value determined for M-0 (-2.42 mm) (Table 3). The differences between these determined values were found to be statistically significant (P<0.05). Previously, Moreira et al. (2023) reported that the technological attributes of bakery goods, including firmness, SV, SI, and consistency, impact the sensory approval of the product by consumers.

The highest USV value among the samples (19.33 mm) was determined for M-2. This was followed by M-3 (17.33 mm), M-1 (15.25 mm), and M-0 (13.08 mm), respectively (Table 3). The differences in USV values arising from differences in recipes were found to be statistically significant (P<0.05).

The sensory characteristics are significant factors in determining the approval of a product (Ahmad et al., 2021; Yavuz et al., 2022; Demirkan et al., 2024). The results of sensory evaluations are shown in Table 4. Figure 3 depicts the spider diagram illustrating the sensory evaluation of the four different muffin samples. The color of baked goods originates from two factors: the inherent color contributed by individual ingredients and the developed color that emerges from the interaction between ingredients (Sunwar et al., 2022). According to these findings, crust color values were scored in the range of 5.91-6.86 by the panelists. The crumb color of M-0 was at the lowest level, while formulations M-1, M-2, and M-3 received higher scores. However, the differences in these scores were not statistically significant (P>0.05). Additionally,

3.3. Sensory evaluation

scores were not statistically significant (P>0.05). Additionally, odor scores for muffin samples ranged from 5.82 to 6.50. However, in formulations M-2 and M-3, a higher amount of black chickpea flour (16.7%) led to lower odor scores for muffins according to the panelists. Nevertheless, these differences were not statistically significant (P>0.05).

Table 4. Sensory	properties	of muffin	samples.
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Sensory parameters	<b>M-0</b>	M-1	M-2	M-3
Crust color	$5.91{\pm}1.77^{a}$	6.45±1.79 <sup>a</sup>	6.86±1.52 <sup>a</sup>	6.55±1.63ª
Crumb color	$5.64{\pm}1.87^{a}$	$6.45 \pm 1.44^{a}$	$6.59{\pm}1.50^{a}$	$6.18 \pm 1.74^{a}$
Odor	$6.50{\pm}1.50^{a}$	6.68±1.39 <sup>a</sup>	6.14±1.39 <sup>a</sup>	5.82±1.62ª
Taste	5.95±2.01ª	5.77±1.60 <sup>a</sup>	$5.73{\pm}1.80^{a}$	5.36±1.89ª
Softness	$6.82{\pm}1.74^{a}$	$6.55 {\pm} 1.57^{ab}$	5.55±1.87°	5.64±1.43 <sup>bc</sup>
Moistness	5.95±2.13ª	6.14±1.98 <sup>a</sup>	5.82±1.62 <sup>a</sup>	5.82±1.59ª
Crumbliness	$5.52{\pm}1.69^{a}$	5.50±1.30 <sup>a</sup>	$5.45 \pm 1.50^{a}$	$5.71 \pm 1.45^{a}$
Elasticity	$6.50{\pm}1.68^{a}$	4.90±1.95 <sup>b</sup>	$5.24 \pm 2.12^{b}$	$4.59 \pm 1.89^{b}$
Porosity	$4.86{\pm}2.17^{a}$	5.32±1.70 <sup>a</sup>	5.59±2.06ª	$5.14{\pm}1.88^{a}$
Volume	6.23±1.57ª	5.00±1.63 <sup>b</sup>	6.41±1.71ª	5.86±1.61 <sup>ab</sup>
Overall acceptability	6.57±1.36ª	$6.36{\pm}1.50^{a}$	$6.05 \pm 1.77^{a}$	$5.76 \pm 1.45^{a}$

Rows labeled with distinct letters (e.g., a, d) indicate statistically significant differences (P<0.05)



Figure 3. Spider web chart comparison of sensory analysis.

In terms of taste scores, muffin samples were found to range from 5.36 to 5.95. The highest taste score was observed in sample M-0, followed by M-1, M-2, and M-3, respectively. Hence, vegan-type produced muffins received lower scores than control muffins, but these differences were not statistically significant (P>0.05).

Softness scores ranged from 5.55 to 6.83, with sample M-2 receiving the lowest softness score and sample M-0 receiving the highest. Additionally, the differences in softness scores were found to be statistically significant (P<0.05).

The moisture parameter, which reflected the level of perceived moisture, varied between 5.82 and 6.14, which indicates that the highest moisture score was given to sample M-1 and the lowest scores were given to samples M-2 and M-3. However, these scores were not statistically significant (P>0.05). Crumbliness values ranged between 5.45 and 5.71, but no statistically significant differences were found among the crumbliness scores (P>0.05).

Elasticity values ranged from 4.90 to 6.50, with significant differences observed between samples M-0 and M-1/M-2/M-3 (P<0.05). Also, sensory evaluators rated porosity scores between 4.86 and 5.59, with no statistically significant differences found among these scores (P>0.05). Moreover, volume values were determined to range from 5.00 to 6.23. The volume scores from smallest to largest were as follows: M-1<M-3<M-0<M-2. However, the differences between the scores of M-0 and M-2 were not statistically significant (P>0.05). Additionally, overall acceptability scores ranged from 5.76 to 6.57. However, these results did not show statistically significant differences (P>0.05), showing that all muffin formulations were acceptable for the panelists.

#### 4. Conclusions

As bakery products like muffins gain increasing popularity globally, there's a growing demand for items that are not only delicious but also low in calories and offer health benefits. Hence, the aim of this study was to assess the characteristics of muffin samples and illustrate how incorporating almond milk, aquafaba, and black chickpea flour influenced their sensory attributes and overall quality, catering to consumers who prioritize mindful eating habits. According to the findings, while the muffin formulation did not significantly affect the pH and dry matter of the muffin batter, there were notable decreases in the pH of baked muffins compared to the control. Additionally, the lowest baking loss was observed in M-1 samples. The highest calorie value was found in the control samples, whereas increasing the concentration of black chickpea flour in the formulation, substituting aquafaba for eggs, or using almond milk instead of cow's milk reduced the calorie content of the produced muffins. Moreover, height and density values were not significantly affected by the muffin formulation (P>0.05). However, compared to the control, other samples showed significantly lower values for uniformity index, volume, shrinkage value, symmetry index, volume index, and specific volume, while their density values were higher (P<0.05). Furthermore, among the sensory parameters, there was no statistically significant effect on moistness, crumb color, crust color, porosity, taste, crumbliness, odor, and overall acceptability. However, the effect was significant on other parameters (softness, elasticity, porosity, and volume) (P<0.05). Panelists rated vegan cakes lower in softness, elasticity, and volume scores. In conclusion, the muffin samples produced not only cater to individuals suffering from egg, cow milk and gluten intolerance and allergies but also offer a delightful option for those adhering to a vegan diet. This versatility underscores their potential to accommodate diverse dietary preferences and requirements while providing a tasty treat for all and aiding in the advancement of clean-label food products with the substitution of animal-based ingredients.

### Author Contributions

Gozde Kutlu: Writing – original draft, review&editing, methodology, visualization, validation, supervision. Safa Yilmaz: Methodology, investigation. Ahmet Eray Karabulut: Methodology, investigation.

#### Acknowledgments

This article was prepared based on the results of the research conducted by the 2<sup>nd</sup> and 3<sup>rd</sup> authors within the scope of the Food Formulation and Sensory Analysis course. We would like to thank Mr. Hüseyin Karacan, MSc biologist and laboratory supervisor at Ankara Medipol University, for , for assisting in the chemical analysis.

#### **Conflicts of Interest**

The authors state that they have no conflicts of interest.

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## **European Food Science and Engineering**

Eur Food Sci Eng 2024, 5 (1), 35-43 doi: 10.55147/efse.1495662 https://dergipark.org.tr/tr/pub/efse

## Multiresponse optimization of value-added sesame seed candy

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#### ARTICLE INFO

Research Article Article History: Received: 4 June 2024 Accepted: 26 June 2024 Available Online: 30 June 2024 Keywords: Sesame seed Candy Response surface methodology (RSM) Optimization D-optimal Desirability function

#### ABSTRACT

Sesame seed candy, traditionally produced from sesame seeds and sugar, is a confectionery product. This study is aimed at developing value added candy from sesame seed, ginger, cinnamon, honey; and optimizing employing desirability function technique. Experiments were conducted using a four-component constrained D-optimal mixture-process experimental design and the formulated candies were characterized. The formulation design constraints were: sesame seed (40-70%), ginger (10-30%), cinnamon (10-30%), honey (10-30%); while the processing factors were roasting temperature (100-150 °C) and roasting time (10-30 min). Consumer acceptability was measured by 50 semi-trained regular consumers using a 9-point hedonic scale. Optimal candy of highest desirability index of 0.506 was obtained from 43.53% sesame seed, 10% ginger, 16.47% cinnamon, and 30% honey, with 110 °C roasting temperature. and 27 min roasting time. Quality properties of the optimal candy were 7.95% moisture content, 20.76% dietary fiber, 37.89% protein, 10.40% carbohydrate, 17.20% fat content, 249.71 mg calcium, 0.029 mg/100g vitamin C, 353.54 kcal energy value, 5.78 hardness, 6 taste, 5.74 colour, 5.87 chewiness, 5.92 gumminess, 5.6 crispness, 6.11 flavour, and overall acceptability of 6.20 score; based on 9-point hedonic scale. Compared with the traditional sesame seed candy, the optimal value-added sesame seed candy was of higher quality.

### 1. Introduction

The Sesame, a genus of Sesamum, is a member of the Pedaliaceae family. According to the difference in germplasm color, sesame can be classified as white sesame, black sesame, and yellow sesame, among which black and white sesame are the more common and widely grown dominant species. Sesame seeds are often used to make a variety of foods, such as sesame oil, sesame paste, or to decorate other foods. Sesame seeds are rich in fat, protein, minerals, vitamins, and dietary fiber. Sesame oil, which is obtained through traditional oil production methods, is rich in unsaturated fatty acids, fatsoluble vitamins, amino acids, etc. Studies have found that sesame seeds contain 21.9% protein and 61.7% fat, and are rich in minerals such as Fe and Ca. Sesame seeds are rich in nutrients and have the reputation of being an "all-purpose nutrient bank" and the "crown of eight grains (Wei et al., 2022).

Sesame seed candy is traditionally made from sesame, sugar and/or honey. It is popular from the Middle East through South Asia to East Asia; in Nigeria it is mostly consumed by children. The texture varies from chewy to crisp (Richardson,

2008). Sesame seed is one of the oldest oilseed crops known. It has one of the highest oil contents of any seed. It has been regarded as a health food for aging prevention and energy increasing (Obiamaka, 2019). However, sesame seed, the major ingredient in the candy production is deficient in many nutrients that the body needs. Hence, there is need to fortify the product by adding ingredients that will inject vitamins and minerals during processing to increase their nutritional value (Olson et al., 2021). Fortification is a proven, safe and costeffective strategy for improving diets and for the prevention and control of micronutrient deficiencies. Malnutrition is commonly recognized as being among the most widespread and pernicious causes of human suffering throughout the world. FAO estimated that a total of 842 million people were undernourished in 2011-2013 and current statistics have not shown any improvement on this. About 45% of the 6.9 million child deaths in 2011 were linked to malnutrition. Some 162 million children under five years of age are stunted owing to chronic undernutrition and 99 million children are underweight (FAO, 2013). Multi-disciplinary, multifaceted, nutrition-sensitive food manufacturing approach is one of the effective vehicles in combating and tackling malnutrition that is prevalent in the rural communities of Africa. The objective of this study was to optimize the development of value-added sesame seed candy that can serve as an effective vehicle in combating and tackling malnutrition that is prevalent in the rural communities of Africa. Studies have shown that candies can be used to increase the nutritional status of consumers by incorporating nutrients such as protein and fiber from plant source which have health benefits. Sesame seed, the major ingredient in the candy production, has a composition of about 50-52% oil, 17-19% protein, 48.5% crude oil, 9.4% crude fiber, 4.2% ash, and 16-18% carbohydrate (Adebayo-Oyetoro et al., 2017).

This study presents a novel optimization procedure using RSM to optimize multiple responses, generate the optimal parameter-setting. and to determine the optimum ratio of sesame seed/ginger/cinnamon/honey formulations. Optimization is concerned with determining the best possible formulation from a set of factors (ingredients and & or process factors that meets desire responses (studied quality indices). Once the formulation experiments have been carried out and the experimental data gotten, based on the design matrix, multi-variate regression analysis on the experimental data are performed. Optimization commences after the multi-variate regression analysis, using the same experimental data from the formulation experiment. Quite a number of optimization techniques are usually employed. Numerical optimization will optimize any combination of one or more goals. The goals may apply to either factors (ingredients and processing parameters) or responses. The possible goals are: maximize, minimize, target, within range, none (for responses only) and set to an exact value (factors only). Weights (0.1 to 10) are assigned to goals to adjust the shapes of their desirability functions. Increased weight (up to 10) moves the result towards the goal. Also, the "importance" of a goal can be changed in relation to the other goals. If the researcher wants all goals to be equally important, at a setting of 3 pluses (+++) is given. If one goal to be most important, a setting of 5 pluses (+++++) is given (ReliaSoft, 2015). Using numerical optimization, via objective functions, optimal blends are predicted in form of optimization solutions indicating components combinations that meet specifications (optimal formulation conditions) and the "overall desirability index". A graph of desirability can be generated for any of the solutions found via numerical optimization. Individual response may also be graphed. Such individual response graph can be useful to see how a single response behaves in the vicinity of a particular optimum. In addition, the numerical optimization histograms, often referred to as the bar graph, can be generated. It is a simple view that shows the desirability for each factor and each response individually. Usually, the bottom histogram bar is the combined desirability of all the factors and responses. With multiple responses, regions where requirements simultaneously meet the critical properties are sought. By overlaying critical response contours on a contour plot the best compromise can be picked (the sweet spot). Graphical optimization displays the area of feasible response values in the factor space. Regions that do not fit the optimization criteria are shaded gray. Any "window" that is not gray shaded satisfies the goals for every response. In the graphical optimization solution overlay plot, the bright yellow (default) now shows where the entire range of all intervals meet the specified criteria. The dark gold corresponds to where the point estimate meets the criteria requirements, but part of an interval estimate does not (Goos & Jones, 2011; ReliaSoft, 2015). Optimization is concerned

with determining the best possible formulation from a set of factors (ingredients and/or process factors that meets desire responses (studied quality indices). In a multi-response study, the degree to which all the optimization goals are met is "a compromised value referred to as overall desirability index" (Smith, 2005; Tsai et al., 2010; Wang & Fang, 2010). The aim of this research was to develop and optimize value added sesame seed candy. The objective was to find an optimum sesame seed/ginger/cinnamon/honey confection that would have improved nutritional values and consumer acceptance close to that of commercial sesame seed candy.

#### 2. Materials and methods

#### 2.1. Materials

Samples of white sesame seed (*Sesamum indicum L.*), of the Pedaliaceae family, common ginger (also known as Indian or Chinese ginger), cinnamon (cassia) and honey. were purchased from Kure market in Minna, Niger State, Nigeria. The preparation of sesame seed candy was carried out at the department of Agricultural and Bioresources Engineering laboratory, while the analyses were carried out at the department of Food Science and Technology laboratory at Federal University of Technology, Minna.

#### Reagents and apparatus

The analytical grade reagents used during the experiment include: ferric chloride, ammonium thiocyanate, ammonium solution, sulphuric acid (H<sub>2</sub>SO<sub>4</sub>). sodium hydroxide, ethanol, petroleum ether, hydrogen chloride (HCL), n-hexane and distilled water. The apparatus used during the experiment include: Kenwood electric oven (70L Convection Steel), burette (Boro 3.3. To DIN EN ISO 385), desiccator (Boro 3.3. DIN 12491), 1200 °C (2190 °F) laboratory muffle furnace, measuring cylinder (with glass hexagonal base), pipette (CellTreat 10 mL Serological), crucible (SKU: 034-5920230), stopwatch (A601X), digital weighting balance (Reshy Lab scale 3000 g), digital thermometer (Model: ACC550DIG), conical glass flask 500 mL, pyrex glass petri dishes, Maxi 60 60 4 gas cooker, stirrers (Scilogex SCI-120-HS) frying pan (Misen 5-Ply Stainless Steel Pan), stainless steel knife, stainless steel flat plates, spectrophotometer (UV-Vis 9300 / 9600) and parchment paper.

#### 2.2. Methods

#### Sesame seed candy preparation

The sesame seed, ginger and cinnamon were washed to remove foreign materials and were then sundried. Ginger and cinnamon were milled and packaged separately. The first stage in the production of sesame seed candy is roasting of the seed. Though roasting is done traditionally by placing the seeds inside a frying pan on top of a stove, but in this work, a temperature-regulated oven was employed. The honey was placed in a saucepan over medium heat to froth and bubbled up after about 5 min. Guided by the matrix design (Table 1), the mixtures of roasted sesame seed, ginger, cinnamon were then poured gradually inside the honey syrup with a large spoon and gently stirred until a uniform sample was obtained.

Table 1.	Design	matrix	for the	value-added	l sesame	seed o	candv	formulation	experiments.
I abit I	Design	manna	ior the	varue uuuee	i bebuille	beeu e	cund y	ioiiiiaiaiioii	experimento.

Run	Sesame seed (%)	Ginger (%)	Cinnamon (%)	Honey (%)	Roasting	Roasting time
					temperature (°C)	(min)
1	40.0	10.0	30.0	20.0	150	10
2	40.0	20.0	30.0	10.0	150	10
3	40.0	20.3	19.1	20.6	100	10
4	40.0	20.3	19.1	20.6	100	10
5	40.0	19.7	20.7	19.5	150	10
6	70.0	10.0	10.0	10.0	150	10
7	56.8	20.2	13.0	10.0	150	30
8	56.4	10.0	12.1	21.5	150	10
9	55.5	21.6	11.4	11.4	100	30
10	40.0	10.0	30.0	20.0	150	30
11	42.3	12.8	14.9	30.0	150	10
12	55.5	11.6	22.9	10.0	100	30
13	43.1	16.9	10.0	30.0	100	10
14	40.0	20.0	10.0	30.0	150	30
15	40.0	19.5	19.8	20.7	150	30
16	41.3	13.3	30.0	15.4	100	30
17	40.0	27.0	10.0	23.0	150	10
18	43.6	10.0	16.4	30.0	150	30
19	55.3	10.0	13.9	20.8	100	30
20	40.0	28.8	21.2	10.0	100	10
21	55.4	10.0	22.3	12.3	150	30
22	56.0	11.6	22.3	10.0	100	10
23	40.0	21.7	28.3	10.0	150	30
24	40.0	21.5	28.5	10.0	124	27
25	40.0	10.0	20.8	29.3	100	10
26	40.0	19.7	20.7	19.5	150	10
27	55.8	10.0	13.2	21.0	100	10
28	70.0	10.0	10.0	10.0	150	30
29	41.4	30.0	10.0	18.7	100	10
30	40.9	30.0	10.0	19.1	100	30
31	40.0	28.2	21.8	10.0	100	30
32	40.0	20.2	19.2	20.6	100	30
33	43.8	16.2	10.0	30.0	100	30
34	43.3	30.0	16.7	10.0	150	10
35	47.7	12.3	10.0	30.0	119	22
36	55.4	21.4	11.5	11.7	100	10
37	70.0	10.0	10.0	10.0	100	10
38	56.0	20.8	10.0	13.2	150	10
39	55.2	13.7	10.0	21.0	150	30
40	70.0	10.0	10.0	10.0	100	30
41	40.0	19.5	19.8	20.7	150	30
42	40.0	10.0	20.0	30.0	100	30
43	47.8	10.0	20.8	21.4	144	20
44	50.0	10.0	30.0	10.0	107	20
45	48.1	20.6	19.9	11.5	125	13
46	55.2	12.3	21.6	10.9	150	10
47	43.5	30.0	12.4	14.1	150	30
48	41.6	13.8	30.0	14.6	100	10
49	40.0	20.2	19.2	20.6	100	30
50	55.5	21.6	11.4	11.4	100	30

The sesame seed candy dough was then cooled, formed and cut into desired shape with the aid of a knife. This preparation of the sesame seed candy was based on the design matrix.

## *Experimental design for the value-added sesame seed candy formulation experiments*

Design-Expert software (version 13, Stat-Ease Inc., USA) was used for experimental design and statistical evaluation of data. A four-component constrained D-optimal mixture-process experimental design, totaling 50 randomized experimental runs, was employed. Four major variable components with two processing factors were investigated. The respective formulation design constraints were: sesame seed (40%-70%), ginger (10%-30%), cinnamon (10%-30%), and honey (10%-

30%). The processing factors investigated were roasting temperature (100-150 °C) and roasting time (10-30 min). The quality properties of the value-added sesame seed candy monitored were: moisture content, dietary fiber, protein, carbohydrate, fat content, calcium, vitamin C, energy value, hardness, taste, colour, chewiness, gumminess, crispness, flavour, and overall acceptability. The design matrix for the D-Optimal mixture – process design is presented in Table 1. The formulation of the blend with the processing parameters were based on the design matrix.

#### Proximate analysis and sensory evaluations

Most of the quality characteristics of the value-added sesame

seed candy which were measured using the methods described by the Association of Analytical Chemist with some modifications by some researchers (AOAC, 2002; Crisan & Sands, 2008; El-Ishaq & Obirinakem, 2015; Christian et al., 2019). The sensory evaluation of the samples was conducted using a total of 30 semi-trained panelists. The samples were evaluated for taste, flavour, sweetness, colour, texture and overall acceptability. A 9-point hedonic scale ranging from 9 =like extremely and 1= dislike extremely was used to evaluate the samples.

#### 2.3. Experimental data

Formulated value-added sesame seed candy samples were analyzed and evaluated for the moisture content, dietary fiber, protein, carbohydrate, fat content, calcium, vitamin C, energy value, hardness, taste, colour, chewiness, gumminess, crispness, flavour, and overall acceptability (Tables 2 and 3).

Table 2. Quality properties of the formulated value-added sesame seed candy.

Run	Moisture	Dietary	Protein (%)	Carbohydrate	Fat content	Calcium	Vitamin C	Energy
	content (%)	fiber (%)		(%)	(%)	( <b>mg</b> )	(mg/100g)	value (kcal)
1	6.80	23.61	20.25	19.06	26.28	273.00	0.00	393.76
2	7.40	20.14	21.13	21.13	26.20	269.00	0.00	304.84
3	7.60	20.63	24.85	17.31	25.11	233.00	0.00	398.23
4	9.60	21.11	20.65	19.30	25.34	234.00	0.04	387.90
5	6.40	22.63	19.25	21.54	26.18	216.00	0.02	398.78
6	4.60	19.84	21.00	25.95	24.11	222.00	0.03	304.78
7	5.20	19.72	25.76	20.49	24.33	241.00	0.00	399.97
8	4.20	21.66	19.56	26.46	23.62	256.00	0.00	396.66
9	7.40	22.06	20.88	23.58	21.14	278.00	0.06	368.10
10	8.20	21.72	20.65	25.21	20.22	281.00	0.03	365.42
11	7.20	20.11	30.80	13.91	21.48	283.00	0.03	372.16
12	10.60	20.63	28.70	14.46	20.11	291.00	0.04	353.63
13	7.00	19.84	38.50	10.24	19.92	206.00	0.00	369.74
14	9.80	19.33	42.00	4.84	19.33	218.00	0.00	360.13
15	6.20	19.78	43.50	3.11	17.41	216.00	0.00	343.13
16	8.40	21.62	41.65	3.00	20.33	233.00	0.07	361.57
17	8.60	21.63	31.50	11.84	21.63	247.00	0.07	368.03
18	7.60	20.11	33.25	15.26	19.48	272.00	0.05	369.36
19	7.20	20.22	35.00	14.02	18.36	262.00	0.04	361.32
20	6.20	20.63	33.60	13.61	20.66	252.00	0.02	375.18
21	9.00	23.24	28.72	16.22	18.22	271.00	0.00	365.43
22	5.20	22.11	29.33	18.33	20.63	273.00	0.00	376.31
23	6.80	23.24	25.66	19.80	19.11	222.00	0.00	354.19
24	7.40	23.00	25.38	20.60	18.32	227.00	0.00	348.80
25	6.00	22.14	26.11	21.92	18.73	229.00	0.00	360.69
26	6.90	21.43	24.74	18.48	23.72	240.00	0.02	386.33
27	6.10	20.77	27.79	19.18	21.68	256.00	0.03	382.97
28	6.10	20.54	24.63	22.73	21.16	264.00	0.05	379.91
29	7.10	20.22	32.96	12.61	21.86	221.00	0.03	378.99
30	8.50	19.91	40.27	7.19	18.10	222.00	0.05	352.76
31	8.40	21.21	34.65	10.99	19.07	235.00	0.04	354.21
32	8.50	20.86	37.52	9.49	17.95	240.00	0.04	349.56
33	8.50	19.88	41.37	7.61	16.96	238.00	0.00	348.56
34	6.60	21.12	23.45	19.21	24.73	228.00	0.02	393.24
35	7.60	20.10	35.52	12.43	19.16	245.00	0.04	364.29
36	6.00	20.65	26.82	19.14	22.66	242.00	0.00	387.78
37	5.00	20.74	22.52	24.66	22.91	264.00	0.00	394.94
38	5.80	20.59	22.50	22.08	24.45	241.00	0.03	398.38
39	7.30	20.23	31.20	16.09	19.92	249.00	0.05	368.46
40	6.30	20.44	29.58	19.48	19.21	265.00	0.05	369.16
41	8.30	21.02	32.37	12.91	19.89	241.00	0.04	360.12
42	8.70	20.88	38.95	9.09	16.95	252.00	0.04	344.74
43	7.10	21.39	26.75	18.52	21.49	257.00	0.03	374.47
44	6.90	22.40	24.38	20.51	21.25	269.00	0.00	370.83
45	6.60	21.43	24.65	19.55	23.07	245.00	0.00	384.46
46	5.70	21.82	18.16	25.39	24.70	261.00	0.00	396.55
47	8.00	20.34	32.74	12.54	20.60	225.00	0.05	366.50
48	6.90	22.37	24.99	18.96	22.30	258.00	0.00	376.51
49	8.50	20.86	37.52	9.49	17.95	240.00	0.04	349.56
50	7.40	20.35	33.80	14.03	18.99	243.00	0.05	362.26

**Table 3**. Sensory properties of the formulated value-added sesame seed candy.

Run	Hardness	Taste	Colour	Chewiness	Gumminess	Crispness	Flavour	Overall acceptability
1	6.4	6.1	6.3	5.7	6.4	5.9	5.9	6.2
2	4.8	4.6	5.3	5.5	5.7	4.6	5.1	5.7
3	5.2	5.1	5.7	5.7	6.0	4.9	5.6	5.5
4	4.9	5.1	5.4	6.0	5.5	5.2	5.7	5.6
5	5.8	5.3	6.6	6.0	6.0	5.6	5.7	5.9
6	6.2	4.4	5.7	5.2	4.8	6.2	4.9	5.4
7	6.9	4.9	5.9	5.5	5.1	6.1	6.1	6.1
8	6.0	6.3	6.9	6.0	6.2	5.8	6.7	6.9
9	6.0	5.8	6.3	6.0	5.2	6.2	5.9	6.2
10	71	67	6.6	6.6	64	6.5	64	6.6
11	68	64	63	62	6.5	6.1	6.6	67
12	64	61	6.6	6.2	5 5	53	57	6.5
12	63	63	6.6	63	67	5.5	63	6.5
13	67	6.5	6.0	6.9	6.0	6.6	6.8	7.2
15	63	5.5	6.0	5.8	57	5.1	5.9	6.5
15	6.2	5.5	5.0	5.0	53	57	5.9	6.1
17	6.6	5.5	63	5.7	5.5	5.7	5.0	6.2
17	6.0	5.8	0.3 6.4	6.0	0.2 6.3	5.5	5.9	6.5
10	5.8	5.6	5.9	5.8	0.3 6 1	5.0	6.0	5.8
20	5.0	5.0	5.9	5.6	5.5	5.5	0.0 5 5	5.6
20	5.4	5.4	5.9	5.0	5.0	5.2	5.5	5.0
21	0.0	5.5	0.2	0.2	5.9	5.4	5.5	5.4
22	5.1	5.1	4.8	5.2	5.0	5.1	5.5	5.4
23	5.9	5.1	5.7	5.0	0.0 6 1	5.8	5.5 5.6	5.8
24 25	5.2	5.7	0.1	0.0 5.9	0.1	5.9	5.0	5.9
25	5.0	5.7	5.5	5.8	0.2 5.0	5.4	6.0 5.4	0.3
20	5.5	5.0	5.5	5.4	5.9	5.7	5.4	5.9
27	5.5	4.7	5.2	5.3	5.2	5.1	4.8	5.3
28	4.6	4.4	4.3	5.0	5.0	5.1	4.4	4.8
29	5.3	5.4	5.4	5.5	5.8	5.0	5.6	5.7
30	4.9	4.9	6.0	5.8	5.7	5.9	5.4	5.3
31	5.5	5.0	5.9	5.5	5.4	5.7	5.0	5.6
32	5.7	4.7	6.2	5.5	5.6	5.9	5.3	5.8
33	5.9	5.6	6.1	5.7	6.0	5.9	5.7	6.1
34	6.0	6.2	5.3	6.0	5.1	5.4	6.2	6.2
35	6.4	6.2	5.7	5.7	5.4	5.6	6.3	6.3
36	6.2	6.0	6.0	6.4	6.1	5.9	6.5	6.3
37	6.2	5.6	5.8	5.3	5.3	5.7	6.0	6.4
38	6.1	5.6	5.7	5.7	6.0	5.6	6.0	6.3
39	6.8	6.2	6.4	6.1	6.3	6.3	6.4	6.8
40	6.2	5.6	5.5	5.4	6.0	5.6	5.6	6.2
41	6.4	5.3	5.9	5.9	5.6	5.7	5.7	5.7
42	5.8	5.7	5.4	5.9	6.0	5.6	6.0	6.2
43	6.4	5.7	6.0	6.0	5.5	5.8	6.1	6.1
44	6.2	5.1	5.7	5.8	5.5	5.2	5.5	5.9
45	5.1	4.9	5.2	5.2	5.6	5.2	5.1	5.3
46	5.4	5.7	5.4	5.2	5.5	5.7	5.5	5.6
47	5.1	5.1	5.2	5.6	5.6	5.3	5.6	6.0
48	5.3	5.1	5.2	5.3	5.3	5.3	5.1	5.5
49	5.2	4.8	5.3	4.9	5.5	5.4	4.7	5.3
50	5.2	5.0	5.7	5.2	5.8	5.3	5.5	5.5

## 2.4. Numerical and graphical optimization of formulated value-added sesame seed candy

In this work, the numerical and graphical optimization approaches, exploiting desirability function technique, were utilized to generate the optimal formulation with the anticipated responses. Desired limits of response variables, optimization goals, and their objectives functions were decided, optimization constraints were clearly defined and individual response is scaled to objective function called "individual desirability index". The summary of the optimization constraints employed for the formulated noodles are presented in Table 4.

#### 3. Results and Discussion

# **3.1.** Optimization of formulated value-added sesame seed candy

Since many variables were being investigated (ingredients and process parameters) and many responses were involved in the study, numerical optimization approach, exploiting the desirability function technique; was utilized to generate optimal formulation with the anticipated responses. Based on the criteria constraints (Table 4), fifty-two desirability formulation solutions (component proportions and process parameter values) were found as summarized in Table 5; with the quality properties of the optimal formulation presented in Table 6.

Table 4. Optimization constraints for the formulated value-added sesame seed candy.

Name	Goal	Lower	Upper	Lower	Upper	Importance
		Limit	Limit	Weight	Weight	-
Sesame Seed	target = 50	40	70	1	10	3
Ginger	in range	10	30	1	1	3
Cinnamon	target = 30	10	30	1	10	5
Honey	target = 30	10	30	5	10	5
Roasting Temperature	target = 100	100	150	1	1	3
Roasting Time	target = 10	10	30	1	1	3
Moisture Content	minimize	4.2	10.6	1	1	3
Dietary fiber	in range	19.33	23.61	1	1	3
Protein	target = 42	18.16	43.5	5	10	5
Carbohydrate	minimize	3	26.46	1	1	3
Fat Content	minimize	16.95	26.28	1	1	3
Calcium	maximize	206	291	1	1	5
Vitamin C	target = 0.07	0	0.07	1	10	3
Energy value	target = 395	304.78	399.97	1	10	5
Hardness	in range	4.5667	7.1	1	1	3
Taste	in range	4.4	6.6667	1	1	3
Colour	in range	4.3333	6.8667	1	1	3
Chewiness	in range	4.8667	6.8667	1	1	3
Gumminess	in range	4.8	6.7	1	1	3
Crispness	maximize	4.5517	6.5517	1	1	3
Flavour	maximize	4.4	6.8333	1	1	3
Overall Acceptability	maximize	4.7667	7.2333	1	1	3

Table 5. Optimal formulation conditions for the formulated value-added sesame seed candy.

No	Sesame Seed	Ginger	Cinnamon	Honey	Roasting Temperature	Roasting Time	Desirability	
1	43.534	10.000	16.466	30.000	109.611	27.101	0.506	Selected
2	43.536	10.000	16.464	30.000	109.782	27.098	0.506	Serected
3	43.539	10.000	16.461	30.000	109.390	27.132	0.506	
25	49.182	10.000	12.193	28.625	147.618	28.424	0.400	
26	40.834	10.000	30.000	19.166	100.000	27.876	0.351	
27	49.985	16.493	12.254	21.268	100.000	23.555	0.320	

Table 6. Quality properties of the optimally formulated value-added sesame seed candy.

No	Moisture	Dietary	Protein	Carbohydrat	Fat	Calcium	Vitamin	Energy	Desirability	
	content	fiber		e	content		С	value		
1	7.949	20.761	37.886	10.396	17.198	249.705	0.029	353.541	0.506	Selected
2	7.948	20.761	37.872	10.403	17.190	249.799	0.029	353.616	0.506	
3	7.953	20.760	37.920	10.377	17.198	249.653	0.029	353.429	0.506	
25	7.831	20.403	35.281	12.154	18.723	269.471	0.036	363.079	0.400	
26	7.750	22.238	34.934	11.325	19.703	252.115	0.061	363.457	0.351	
27	7.739	20.476	33.691	13.949	21.300	232.852	0.036	368.581	0.320	
Cont	inue									

No	Hardness	Taste	Colour	Chewiness	Gumminess	Crispness	Flavour	Overall Acceptability	Desirability	
1	5.783	5.999	5.743	5.873	5.921	5.600	6.108	6.195	0.506	Selected
2	5.779	6.005	5.747	5.875	5.921	5.603	6.110	6.197	0.506	
3	5.786	5.994	5.738	5.870	5.921	5.598	6.106	6.192	0.506	
25	6.681	6.520	6.305	6.184	5.632	5.910	6.509	6.914	0.400	
26	6.549	5.559	5.528	5.896	6.090	5.739	6.142	6.005	0.351	
27	6.501	4.942	5.757	5.651	6.385	5.408	5.613	5.654	0.320	

Solution 1 (with the comment "selected") is the one that best meet the specified criteria. The factor settings (i.e. ingredients proportions and processing parameters values) are those that result in the highest desirability scores and indicates the island of the design space/response surface with the best acceptable outcome. So, in this work we have fifty-two islands (local optima), with island 1 (solution 1) being the best. Figure 1 shows the numerical optimization solution desirability bar graph for the optimal value-added sesame seed candy while the graphical optimization solution overlay contour and overlay mix-process plots are presented as Figures 2 and 3.

The graphical optimization solution overlay contour and overlay mix-process plots give the summary or details of the optimal conditions and quality properties of the optimal valueadded sesame seed candy. The contours are plotted at the limits specified by the "set criteria". The bright yellow defines the acceptable factor settings while the grey defines the unacceptable factor settings. If intervals are included in the criteria, then a blend of the acceptable and unacceptable colors is used to show where the interval limits are unacceptable.

The composition of sesame seeds comprises 45-65% oil, a noteworthy source of plant-based protein with content ranging from 19 to 35% per 100 g of seeds, 14 to 20% carbohydrates, and 15 to 20% hull material. Studies conducted by other researchers have shown that sesame seeds and sesame oil are richer in phytochemicals and have higher nutritional value than other parts of the sesame plant. Sesamin, sesamol and other chemical components have a variety of pharmacological effects and are of great benefit to human health, and can be used in the treatment of diseases such as anti-inflammatory, antioxidant, anti-cancer, antimelanogenic, auditory protection, anti-

cholesterol, and anti-aging, and have a protective effect on the heart, liver, and kidneys (Ahmed et al., 2020; Wei et al., 2022; Yüzer & Gençcelep, 2023; Mostashari et al., 2024). Studies have shown that candies can be used to increase the nutritional status of consumer by incorporating nutrients such as protein and fiber from plant source, which have health benefits. Candies increases consumers' satiety and pleasure (Wang & Fang, 2010). The quality properties of the optimal candy gotten from this research were 7.95% moisture content, 20.76% dietary fiber, 37.89% protein, 10.40% carbohydrate, 17.20% fat content, 249.71 mg calcium, 0.029 mg/100g vitamin C, 353.54 kcal energy value, From the result of the study, the optimal value-added sesame seed candy was higher quality in comparison with the traditional sesame seed candy.



## Desirability







Overlay Plot

Figure 2. The graphical optimization solution overlay contour plot for the optimal value-added sesame seed candy.



**Overlay Plot** 

Figure 3. The graphical optimization solution overlay mix-process plot for the optimal value-added sesame seed candy

#### 4. Conclusions

This study presents a novel procedure that optimizes products/processes with multiple responses. Optimization of value-added sesame seed candy production was accomplished by response surface methodology. The combinations of 43.5% s sesame seed, 10% ginger, 16.47% cinnamon, and 30% honey, with 110 °C roasting temperature and 27 min mixing time, gave the highest desirability index of 0.506. The optimum ranges as well as the quality properties of the optimal product indicated that the developed value-added sesame seed candy was of higher quality, when compared with the traditional sesame seed candy. The developed optimal product is nutritionally superior with higher consumer acceptability and sensory characteristics comparable to commercial traditional sesame seed candy.

#### **Conflict of Interests**

The authors declare that they have no competing interests.

#### Funding

This research is self-sponsored and did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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