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Valorization of Sesame By-Products: Chemical and Functional Properties after Extrusion Processing

Valorización de Subproductos de Ajonjolí: Propiedades Químicas y Funcionales posteriores al Proceso de Extrusión

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ABSTRACT

Sesame (*Sesamum indicum* L.) is a seed rich in lipids, proteins, and carbohydrates, primarily used for oil extraction. The residual by-product of this process, however, could be valuable as a functional ingredient due to its high content of bioactive compounds. In this study, the chemical, physicochemical, and techno-functional characteristics of sesame by-product flour obtained by extrusion at 139 °C and 80 rpm in a single-screw extruder were evaluated. The extrusion process significantly improved the flour, increasing insoluble dietary fiber by 122.22 % and total fiber by 1.68 %. In addition, the bulk density, water activity, water solubility index, dispersibility, and emulsion stability were increased. These changes make the extruded flour suitable for the development of functional foods, such as beverages, thanks to its improved chemical and techno-functional properties. The extrusion process, therefore, proves to be an effective technology for improving flour characteristics and expanding their potential for nutraceutical applications.

KEY WORDS: Sesame by-product, extrusion, techno-functional, proximal composition, WAI, WSI.

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RESUMEN

El ajonjolí (*Sesamum indicum* L.) es una semilla rica en lípidos, proteínas y carbohidratos, utilizada principalmente para la extracción de aceite. El subproducto residual de este proceso, sin embargo, podría tener valor como ingrediente funcional debido a su alto contenido de compuestos bioactivos. En este estudio, se evaluaron las características químicas, fisicoquímicas y tecno-funcionales de la harina de subproducto de ajonjolí obtenida mediante extrusión a 139 °C y 80 rpm en un extrusor de tornillo simple. El proceso de extrusión mejoró significativamente la harina, aumentando la fibra dietética insoluble en un 122.22 % y la fibra total en un 1.68 %. Además, se incrementaron la densidad aparente, la actividad de agua, el índice de solubilidad en agua, la dispersabilidad y la estabilidad de emulsión. Estos cambios hacen que la harina extrudida sea adecuada para el desarrollo de alimentos funcionales, como bebidas, gracias a sus mejoradas propiedades químicas y tecno-funcionales. El proceso de extrusión, por lo tanto, demuestra ser una tecnología eficaz para mejorar las características de las harinas y ampliar su potencial en aplicaciones nutracéuticas.

PALABRAS CLAVE: Subproducto de ajonjolí, extrusión, tecno-funcional, composición proximal, WAI, WSI.

Introduction

Sesame (*Sesamum indicum* L.) is a seed rich in lipids (approximately 50 %), protein (17-35 %), and carbohydrates (14-24 %). Additionally, it is rich in fatty acids (oleic, linoleic, palmitic, and stearic), minerals (calcium, iron, phosphorus, and potassium), and vitamins (B1 and E) (Sharma *et al*., 2021; Quintero-Soto *et al.,* 2022). It also presents a considerable unsaponifiable fraction with the presence of compounds such as sterols, tocopherols, and lignans (sesamin and sesamolin) (Ruiz-Armenta *et al.,* 2022). The presence of some antinutritional compounds has been reported in sesame seeds such as lectins, saponins, trypsin inhibitors, tannins, and oxalates, which interfere with human metabolism, hindering the absorption of some minerals in the body. Therefore, the application of thermal technologies is necessary to reduce or eliminate these components (Quintero-Soto *et al*., 2022; Ruiz-Armenta *et al*., 2022).

Sesame seed is mainly used for oil extraction due to its good fatty acid profile, with unsaturated fatty acids predominating. However, the residual by-product generated could be utilized as an ingredient in formulating new food products because it has a high content of proteins, minerals, dietary fiber, and phenolic compounds, providing high nutritional and nutraceutical value to the resulting product (Ruiz-Armenta *et al*., 2022). It is estimated that approximately 1.53 million tons of sesame by-products are obtained annually worldwide, which are not valorized and could be candidates for the production of functional foods due to their content of bioactive compounds (Nagar *et al*., 2022; Quintero-Soto *et al*., 2022).

Extrusion is a process that belongs to green technologies since it does not generate contaminating effluents and also offers advantages such as high productivity, low operating costs, high product quality, retention of bioactive compounds, and high energy efficiency (Quintero-Soto *et al*., 2022). Extrusion is a rapid cooking process that improves the nutritional and nutraceutical quality of foods, enhances the digestibility of proteins and starch, modifies lipid content, inactivates microorganisms and enzymes, reduces antinutritional compounds, increases dietary fiber contents, and enhances the retention of phytochemical compounds (Ruiz-Armenta *et al*., 2022). It is a technological alternative for the development of foods such as ready-to-eat breakfast cereals, new ingredients, cereal-based snacks, and functional foods, among others (Patil & Kaur, 2018; Grasso, 2020). Additionally, it has the potential to produce flours with good techno-functional properties and minimal degradation of bioactive compounds, which can be used as ingredients in the development of functional foods (Gómez-Favela *et al*., 2021). Ruiz-Armenta *et al*. (2022) reported an optimization of process variables (extrusion temperature (ET)/screw speed (SS)) to obtain an extruded sesame flour with high values of water absorption index (WAI), water solubility index (WSI), *in vitro* protein digestibility (IVPD), total phenolic compounds (TPC), and antioxidant activity (AoxA). They found the best variables combination at ET= 139°C/SS=80 rpm. Quintero-Soto *et al*. (2022) developed a functional beverage using sesame by-product flour obtained under an optimized variable combination reported by Ruiz-Armenta *et al*. (2022). They reported that beverage with extruded sesame by-product flour showed a higher total phenolic compound, antioxidant activity, and enzyme inhibition (α-amylase, α-glucosidase) than beverage with non-extruded sesame by-product flour.

Only these works mentioned above have been reported to the extrusion process application on sesame by-product. Therefore, the objective of this study was to evaluate chemical, physicochemical, and techno-functional properties of extruded sesame by-product flour.

Material and methods

The raw material used was beige sesame seed (*Sesamum indicum* L.) acquired from the community of El Saladito, Elota, Sinaloa, Mexico. The seeds were cleaned to remove debris and impurities, packed in tightly sealed polyethylene bags, and stored refrigerated at 4°C until further use.

Obtaining extruded sesame by-product flour

First, the sesame seeds were passed twice through a mechanical press to remove the maximum amount of oil from the sample, as the extruder used is designed for materials with low moisture and low oil content. The obtained sesame by-product flour (500 g) was conditioned with distilled water until reaching a moisture percentage of 23 %. This moisture condition was

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determined from preliminary experiments where higher humidities did not allow for proper handling of the flours. The conditioned flour was placed in a plastic bag and refrigerated at 4 °C for 12 hours to homogenize moisture throughout the sample. Then, the sample was brought to room temperature (25 °C) to prevent it from sticking to the extruder equipment. Extrusion was carried out using a single-screw extruder model 20DN (CW Brabender Instruments, Inc., NJ, USA) with a screw diameter of 19 mm; length-diameter ratio 20:1; compression ratio 1:1, and a 4 mm outlet dice. The extrusion temperature (ET) and screw speed (SS) conditions used were ET= 139°C/SS= 80 rpm, which were previously optimized as reported by Ruiz-Armenta *et al*. (2022). The extruded product was collected in trays and left to dry at room temperature with the aid of a fan until reaching a water activity of 0.5 to prevent microbial growth. Finally, the pellets were ground to obtain extruded sesame by-product flour, which was used for the evaluation of the corresponding properties.

Chemical composition

The determination of the chemical composition of the sesame by-product flour was carried out following the AOAC (2023) methodologies for total protein (960.52), lipids (923.03), and ash (934.01). The content of soluble, insoluble, and total dietary fiber was determined using the gravimetric-enzymatic method (985.29) for total dietary fiber. The carbohydrate content was calculated by subtracting the previous components from a value of 100. The determination was performed in triplicate.

Physicochemical and techno-functional properties

pH

Determined according to the AOAC (2023) methodology. 10 g of sample was added to 100 mL of boiled and cooled deionized water and homogenized with a glass rod. The suspension was orbited (240 rpm/10 min), and the pH was subsequently measured. Evaluation was conducted in triplicate.

Bulk density

Determined according to the Mexican Official Standard NMX-FF-034/1-SCFI-2002 (NOM, 2007). 100 mL of flour was added to a graduated cylinder using a funnel; gently tapped five times to promote particle settling. The total volume was filled, the flour weight was recorded, and the bulk density was calculated from these data. Determination was performed in triplicate.

Water activity (a_w)

Determined using an Aqualab mod CX2 equipment (Decagon Devices Inc., Pullman, WA, USA) according to the procedure reported by Takeungwongtrakul *et al.* (2020). A sample of approximately 2 g was placed in the equipment's sample holder until equilibrium was reached (5-10 min). The instrument was calibrated with a sample of distilled water (a_w= 0.99-1.0). Measurements were performed in triplicate at 25 ± 1°C.

Total color difference (ΔE)

The methodology reported by Álvarez-Ossorio *et al.* (2022) was used. The color of the samples was determined using a Minolta Chroma-Meter model CR-210 colorimeter (Minolta LTD, Japan). For color evaluation, approximately 100 g of sample was placed in a 15 cm diameter Kimax Petri dish lid, and the parameters of lightness (L*), redness (a*), and yellowness (b*) were measured. A white mosaic was used as a reference (standard) with known L*, a*, and b* values (L*= 97.63, a*= -0.78, and b*= 2.85). The total color difference (ΔE) of the sample was calculated using the equation:

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

Where: ΔE = Total color difference between the standard and the sample; ΔL, Δa, and Δb = Absolute differences of the L*, a*, and b* values of the standard used and the corresponding values observed in the sample.

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

For these evaluations, the method described by Ruiz-Armenta *et al.* (2022) was used. A sample of 2.5 g of flour was suspended in 30 mL of water at 30°C in a tared 50 mL centrifuge tube. The suspension was stirred on an orbital shaker at moderate speed (250 rpm/30 min), and then centrifuged (3000 x g/30°C/10 min). The precipitate and the supernatant were used to calculate the WAI and WSI, respectively. The formed gel was weighed to calculate the WAI, expressed as grams of gel per gram of sample. The supernatant was carefully decanted into a tared container for solids determination. The WSI, expressed as grams of solids per gram of original solids, was calculated from the weight of dried solids recovered by evaporating the supernatant (110°C/12 h). Both indices were determined in triplicate.

Oil absorption index (OAI)

The oil absorption of the flours was determined using the methodology reported by Chandra *et al*. (2015) with some modifications. 0.5 g flour was added to 3 mL vegetable oil, placed in a graduated centrifuge tube, vortexed for 1 min, allowed to stand for 30 min, and finally centrifuged at 1600 x g/25°C/25 min; the volume of free oil was measured. The amount of absorbed oil was calculated using the following equation:

$$OAI = \frac{(V_1 - V_2)}{g\ of\ sample}$$

Where V_1 is the initial oil volume and V_2 is the final oil volume, expressed as mL of oil absorbed per gram of flour. The test was performed in triplicate.

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Dispersibility

Was determined according to Mora-Escobedo *et al.* (1991). One g of sample was suspended in 10 mL of deionized water in a graduated conical tube; the samples were homogenized using an Ultra Turrax homogenizer at 10,000 rpm/5 min. Phase separation was measured after 30 minutes of rest.

Foam formation and stability capacity

Foam formation and stability capacity were evaluated according to the procedures proposed by Huffman *et al.* (1975). 0.5 g of the sample was placed in a 100 mL graduated cylinder to which 50 mL of distilled water was added. The mixture was agitated using an Ultra Turrax at 12,000 rpm/1 min, and the volume of foam formed was measured. Foam formation capacity was reported as a percentage (% FFC) and calculated according to the equation:

$$\% \, FFC = \left[\frac{(Volume\ after\ stirring - Volume\ before\ stirring)}{Volume\ before\ stirring}\right](100)$$

The graduated cylinder containing the agitated suspension, for which the % FFC was evaluated, was allowed to rest for 30 minutes; at the end of this time, the volume of residual foam was measured, and foam stability, reported as a percentage (% FS), was calculated according to the equation:

$$\% \, FS = \left[\frac{Foam\ volume\ after\ rest}{Total\ foam\ volume}\right](100)$$

Emulsion activity and stability

For the determination of emulsifying activity and emulsion stability, the methodology reported by Huffman *et al.* (1975) was used. 0.7 g of the sample was weighed and added to 10 mL of distilled water and 10 mL of vegetable oil. The suspension was homogenized at 12,000 rpm for 1 minute. The homogenized suspension was centrifuged at 1300 x g/25°C for 5 minutes, and the volume of the formed emulsion was measured. Emulsion activity was evaluated by relating the emulsified layer formed to the total volume and reported as the percentage of emulsifying activity (% EA). Emulsion stability was assessed using the same tubes used in the % EA determination. The tubes were subjected to heat treatment in a water bath (80°C/30 min), cooled, and centrifuged at 1300 x g/25°C for 5 minutes, and the volume of the emulsion was measured. Emulsion stability was reported as a percentage (% ES) and expressed as the percentage of emulsion retained after heat treatment. The test was performed in triplicate.

Statistical analysis

Data analysis was performed using a one-way design, with the type of process as the study factor with two levels (extrusion-processed, unprocessed), using STATGRAPHICS Plus version

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5.1 (Statistical Graphics CorporationTM, Rockville, Maryland, USA). Fisher's test was used with a significance level of 5 % to compare the means of the chemical composition, physicochemical properties, and techno-functional properties of extruded and non-extruded flours.

Results and Discussion

Chemical characterization of extruded sesame by-product

Table 1 shows the proximate composition of sesame by-product flour and the effect of the extrusion process on these components. It can be appreciated that sesame by-products exhibited a high protein content (28.72 ± 1.86 %) and total dietary fiber (26.84 ± 0.26 %) and a low lipid content (15.13 ± 0.47 %) compared to whole grain (~50 %, data not shown). Several authors have reported the proximate composition of sesame by-product obtained using different methods. Bukya & Vijayakumar (2013) evaluated sesame paste obtained after oil extraction using the traditional oil mill method, reporting high protein (48.28 %) and carbohydrate (40.94 %) contents, and low lipid (0.82 %) and ash (9.96 %) contents. Costa do Nascimento *et al*. (2012) characterized defatted sesame paste using an expeller extruder at room temperature (25 °C), obtaining low protein contents (12.19 %) and high lipid values (38.08 %). Likewise, these authors reported ash and carbohydrate contents of 9.36 % and 40.37 %, respectively.

Table 1. Effect of extrusion process on chemical composition of sesame by-product flour

Composition (%)	USBF[1]	ESBF[2]
Total protein	28.72 ± 1.86[a]	29.74 ± 2.05[a]
Lipids	15.13 ± 0.47[a]	15.15 ± 0.83[a]
Ashes	10.14 ± 0.09[a]	10.27 ± 0.05[a]
Soluble dietary fiber	0.63 ± 0.03[b]	1.40 ± 0.01[a]
Insoluble dietary fiber	26.21 ± 0.23[a]	25.89 ± 0.31[b]
Total dietary fiber	26.84 ± 0.26[b]	27.29 ± 0.32[a]
Carbohydrates	19.17 ± 1.67[a]	17.55 ± 1.41[a]

[1]USBF = Unprocessed sesame by-product flour; [2]ESBF = Extruded sesame by-product flour. [a-b] Different letters in the same row indicates significant differences ($p < 0.05$) between the means according to Fisher's test.

Source: Own elaboration

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Banerjee *et al*. (2022) evaluated the proximate composition of defatted sesame flour using Soxhlet extraction with hexane, finding higher values of protein (52.62 %) and total carbohydrates (35.32 %), as well as lower lipid (3.84 %) and ash (8.22 %) contents compared to the results obtained in this work. Melo *et al.* (2021) extracted sesame oil by cold pressing and obtained similar values in protein content (30.74 %), ash (7.37 %), soluble, insoluble, and total dietary fiber (3.43 %, 22.53 %, and 25.96 %, respectively), to those found in this study. However, these authors reported a lipid content of 33.02 %, whereas in this study, values of ~15 % were found. On the other hand, Elsorady (2020) characterized sesame paste obtained as a by-product of the oil industry, finding a lipid content of 11.59 %, proteins of 45.63 %, 6.79 % ash, and 35.98 % carbohydrates. The differences in lipid content reported by different authors are primarily due to the type of defatting used on the seed.

Proteins have a profound effect on the physical and nutritional properties of extruded products. As can be seen in Table 1, extrusion had no significant effect on protein content; however, the main effect of the extrusion process on proteins is based on changes in protein quality, involving alterations in amino acid profile, protein digestibility, and functionality, which includes texturization and solubility. Protein quality is influenced by multiple factors such as source, food matrix, bioavailability, and processing conditions. It has been reported that the extrusion process minimally alters total protein content but significantly affects the availability of essential amino acids and protein digestibility (Gulati *et al*., 2020). One of the main changes occurs in the available lysine content, which is involved in the Maillard reaction upon contact with reducing sugars present in the material, resulting in its decrease in the processed product, and can be indirectly evaluated by observing darkening in the processed sample, indicating lysine loss. On the other hand, processing parameters influence lysine loss or retention; for example, extrusion temperature is negatively correlated with lysine loss, while feed moisture content is positively correlated. Additionally, high screw speeds and reduction of die outlet diameter can help retain lysine during the extrusion process. Other amino acids affected by the extrusion process include threonine, valine, leucine, and isoleucine, which are degraded due to barrel temperature effects (Dalbhagat *et al*., 2019; Gulati *et al*., 2020). The temperature and shear force to which foods are subjected during the extrusion process improve protein availability through different pathways, such as deactivation of antinutritional compounds like trypsin inhibitors, lectins, tannins, and phytates due to temperatures and residence times within the extruder barrel, which aid in improving protein digestibility. Furthermore, shear force helps open protein structures during extrusion, thereby enhancing their digestibility by increasing their exposure to digestive enzymes (Arribas *et al*., 2017; Dalbhagat *et al*., 2019).

Significant changes in dietary fiber content were also observed due to the extrusion process. Extruded sesame by-product flour showed a significant increase (p < 0.05) of 122.22 % and 1.68 % in soluble dietary fiber and total dietary fiber, respectively. Meanwhile, insoluble dietary fiber exhibited a significant decrease (p < 0.05) of 1.22 % (Table 1). Dietary fiber plays a crucial role in the human diet, preventing the occurrence of constipation, colon cancer, diabetes, etc., and its incorporation into different food formulations benefits weight control (Arribas *et al*., 2017). Dietary fiber is composed of edible carbohydrate polymers consisting of ten or more monomeric units that cannot be hydrolyzed by endogenous enzymes in the small

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intestine. During the extrusion process, dietary fiber can be significantly modified by altering its total content and solubility (Alam *et al*., 2016; Gulati *et al*., 2020). This process contributes to changes in the physicochemical properties of insoluble dietary fiber by increasing soluble dietary fiber content through cutting forces. The combination of high temperatures and cutting forces in the extrusion process leads to a redistribution of insoluble fiber into soluble fiber. Additionally, there is a breakdown of mineral complexes, releasing them and increasing the bioavailability of Fe, Cu, Zn, and Mn (Alam *et al*., 2016; Kamau *et al*., 2020). A combination of low moisture and high temperatures favors the conversion of insoluble fiber into soluble fiber, while the use of low screw speeds facilitates fiber transformation by providing greater compression and friction on the material (Kamau *et al*., 2020). However, long residence times lead to the formation of resistant starch due to the presence of starch-lipid complexes within the food matrix, thereby increasing soluble dietary fiber. This increase in resistant starch during extrusion can occur through two pathways: 1) the formation of new non-hydroglucosidic bonds, which are resistant to digestive enzymes, as a result of transglucosidation by breaking 1,4 carbon-oxygen bonds, and 2) the formation of retrograded amylose, which is more resistant than non-retrograded amylose to starch-hydrolyzing enzymes (Kamau *et al*., 2020).

On the other hand, the content of lipids, ashes, and total carbohydrates did not show significant changes ($p < 0.05$) after the extrusion process (Table 1). The extrusion process results in a loss of starch crystalline structure accompanied by a complete or partial release of amylose, altering its viscosity. These changes depend on the combination of extrusion variables such as moisture, extrusion temperature, residence time of the material inside the barrel, and cutting force. The extrusion process also increases starch digestibility by opening starch granules and improving gelatinization, making them more susceptible to hydrolytic enzymes. Cutting forces during extrusion partially hydrolyze amylose and amylopectin, generating maltodextrins. The increase in starch digestibility due to extrusion could be attributed to partial starch gelatinization, polymer fragmentation, and starch depolymerization, allowing greater access for digestive enzymes. However, starch-lipid complexes are also formed during extrusion, which exhibit greater resistance to enzymatic digestion (Wang *et al*., 2017; 2018; Kamau *et al*., 2020).

The transformations of starch during the extrusion process include loss of crystalline structure, destruction of granular structure, breaking of glycosidic bonds, and formation of new molecular interactions. These changes in starch depend on the moisture conditions, pressure, extrusion temperature, and shear forces during processing. At low temperatures and high moisture content, the starch granule remains nearly unchanged, resulting in a low expansion ratio in extruded products, whereas increasing the extrusion temperature (>150°C) causes the starch granules to lose their original structure (Alam *et al*., 2016). The starch structure of legumes plays a crucial role in its digestion. High extrusion temperatures alter the digestibility characteristics of starch materials, with lower starch digestibility values at temperatures of 100°C compared to temperatures of 125°C and 150°C (Rathod & Annapure, 2017).

During extrusion, lipid content plays a significant role in lubricating the material within the extruder barrel, thereby modifying specific mechanical energy (SME) and the properties of the extruded products. When samples have low lipid levels (< 5 %), the crystallinity of the extruded

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product decreases, and the SME stabilizes, whereas high lipid levels decrease the SME and extrudate expansion (Dalbhagat *et al*., 2019). Additionally, lipids can physically complex with starch, making lipids less extractable with non-polar solvents, leading to a decrease in lipid content in the proximate analysis of extruded samples. The formation of starch-lipid complexes is favored at high extrusion temperatures and high moisture contents. However, products obtained under these conditions exhibit undesirable characteristics such as high density, hardness, and oil loss in the extruder barrel (Alam *et al*., 2016; Arribas *et al*., 2017; Gulati *et al*., 2020). The formation of starch-lipid complexes is undesirable from a physical standpoint but desirable from a nutritional perspective, as lipid oxidative stability increases when they bind to starch, thereby retaining a significant amount of essential fatty acids during extrusion. Additionally, some of this starch converts into dietary fiber as it becomes resistant to digestion (Gulati *et al*., 2020). Extruding foods with a high lipid content (more than 5-6 %) complicates extruder performance by reducing slip within the cylinder, ultimately decreasing torque (Dalbhagat *et al*., 2019).

On the other hand, the bioavailability of minerals in plant-based foods is influenced by two main factors: 1) the chemical form in which the element is present (mineral, free ion, oxidation state), and 2) the association between the element and other components of the flour such as phytic acid, dietary fiber, and proteins. Minerals are thermally stable during the processing of various food matrices (Dalbhagat *et al*., 2019). The extrusion process can positively or negatively affect the bioavailability of minerals by removing antinutritional factors present in the materials such as phytates and tannins, which form complexes with minerals, hindering their absorption (Dalbhagat *et al*., 2019; Gulati *et al*., 2020). The impact of extrusion conditions on phytic acid, phenolic compounds, dietary fiber, and proteins is the primary driver of changes in mineral bioavailability. Phytic acid forms insoluble complexes by binding Fe and Zn, preventing their absorption in the digestive tract. Extrusion dephosphorylates phytic acid, releasing chelated metals. Like phytic acid, phenolics bind essential minerals to their anionic groups, reducing their bioavailability. During extrusion, phenolic compounds are degraded or polymerized, reducing their chelating properties (Gulati *et al*., 2020). The extrusion process can enhance mineral absorption by inactivating absorption inhibitors at high temperatures, hydrolyzing phytates, and increasing mineral availability, whereas at low temperatures, it reduces phytate content in wheat but has no effect on legumes (Kamau *et al*., 2020). Likewise, components of dietary fiber (lignin and polysaccharides of the cell wall) form complexes with Mg and Ca. The extrusion process modifies cell wall polysaccharides, enhancing mineral absorption; however, the release of insoluble anionic polysaccharides in the process could reduce mineral bio-accessibility as these complexes could be broken down in the large intestine by microbiota bacteria. Process conditions such as low moisture content and high extrusion temperatures facilitate the degradation of phytates, phenolics, dietary fiber, and proteins, improving mineral absorption and creating non-thermal modifications based on shear during processing. High shear during extrusion can reduce ferric iron to its ferrous form, which is more soluble and absorbed more easily (Gulati *et al*., 2020).

Physicochemical characterization of extruded sesame by-product

Table 2 displays the effect of the extrusion process on pH, bulk density, water activity (a_w), and color parameters of sesame by-product flour. It can be observed that the extrusion process

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led to a slight but significant decrease ($p < 0.05$) in the pH value and lightness (L*) of the flour, while there was a significant increase ($p < 0.05$) in bulk density, water activity (a_w), a* and b* color parameters, and total color difference (ΔE). The decrease in pH in the sesame by-product flour after being processed by extrusion could be attributed to other chemical changes, such as lipid modification, protein denaturation, Maillard reaction, and others (Escobedo-Avellaneda *et al.,* 2025). The physical properties of foods reflect the interaction among components, structure, physicochemical properties, and the nature of the food matrix environment (Bukya & Vijayakumar, 2013).

Table 2. Effect of extrusion process on physicochemical properties of sesame by-product flour

Parameter	USBF[1]	ESBF[2]
pH	6.51 ± 0.01[a]	6.4 ± 0.05[b]
Bulk density (g/mL)	0.53 ± 0.03[b]	0.63 ± 0.01[a]
Water activity (a_w)	0.496±0.008[b]	0.552±0.01[a]
Color		
L*	59.38±0.74[a]	54.58±0.67[b]
a*	2.88±0.05[b]	3.38±0.12[a]
b*	16.22±0.15[b]	17.94±0.13[a]
Total color difference (ΔE)	37.08±0.67[b]	43.11±0.95[a]

[1]USBF = Unprocessed sesame by-product flour; [2]ESBF = Extruded sesame by-product flour. [a-b] Different letters in the same row indicate significant differences ($p < 0.05$) between the means according to Fisher's test.

Source: Own elaboration.

Bulk density of a flour is the density measured without the influence of any compressive force and reported in g per cm^3. Bulk density depends on the combined effects of interrelated factors such as the intensity of attraction forces between particles, particle size, and the number of contact points (Bukya & Vijayakumar, 2013). High bulk density values in flours suggest their suitability for use in preparing food products, whereas low bulk density would be advantageous for complementary food formulation (Chandra *et al*., 2015).

Furthermore, color plays a crucial role in the acceptance of extruded products by consumers. This parameter is affected by the material composition and process conditions used (Dalbhagat *et al*., 2019). Color determination is based on the tristimulus effect, where L* measures brightness and varies from 100 for a perfect white to zero for black. Chromaticity dimensions are given by the a* and b* parameters. A positive value of a* is associated with red color and negative with green, while a positive value of b* is associated with yellow and negative with blue (Álvarez-Ossorio *et al*., 2022).

During the extrusion process, some non-enzymatic browning reactions such as the Maillard reaction and caramelization occur, as well as pigment degradation causing a change in coloration by decreasing or increasing lightness (L*), reddening (a*), and yellowness (b*) (Becker *et al*., 2014; Mehraj *et al*., 2018; Dalbhagat *et al*. 2019). The decrease in lightness (L*) values in the extruded flour indicates an increase in flour opacity, while an increase in the value of ΔE in the extruded sesame by-product flour indicates darkening of the flour due to the extrusion process. The value of ΔE depends on both temperature and material moisture content, meaning that high-temperature treatments will significantly affect this parameter of the product, while high humidities will result in lesser color change (Dalbhagat *et al*. 2019).

Techno-functional characterization of extruded sesame by-product

Techno-functional properties are decisive characteristics for the launch of a new food product (Álvarez-Ossorio *et al*., 2022). Table 3 presents the effect of the extrusion process on the techno-functional properties of sesame by-product flour. It can be observed that the extrusion process did not generate significant changes ($p > 0.05$) in the water absorption index (WAI), oil absorption index (OAI), and foam capacity and stability. However, it did show a significant increase in water solubility index (WSI), dispersibility, and emulsion capacity and stability.

The water absorption index (WAI) is a measure of starch's water retention capacity after swelling due to excess water and is related to the hydrophobicity and gelation capacity of biomacromolecules such as starch and proteins. It determines the volume occupied by starch after swelling and indicates its integrity in aqueous dispersions (Ye *et al*., 2018; Dalbhagat *et al*., 2019). Low WAI values can be due to a lower availability of polar amino acids in the flours (Chandra *et al*., 2015) and to the decrease in molecule size due to starch degradation (Dalbhagat *et al*., 2019). The use of high moisture content induces a plasticizing effect on the material, delaying mechanical cutting and reducing starch gelatinization and degradation, thereby increasing WAI. Conversely, high temperatures and low moisture contents decrease WAI due to the prevalence of dextrinization over starch gelatinization (Sharma *et al*., 2017; Dalbhagat *et al*., 2019).

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Table 3. Effect of extrusion process on techno-functional properties of sesame by-product flour

Parameter	USBF[1]	ESBF[2]
Water absorption index (WAI) (g gel/g)	4.63 ± 0.04[a]	4.25 ± 0.40[a]
Water solubility index (WSI) (%)	9.68 ± 0.58[b]	12.49 ± 1.21[a]
Oil absorption index (OAI) (mL of oil/ g)	1.43 ± 0.12[a]	1.30 ± 0.11[a]
Dispersibility (%)	40.83 ± 1.44[b]	74.20 ± 2.47[a]
Foam formation capacity (FFC) (%)	1.03 ± 0.05[a]	1.00 ± 0.01[a]
Foam stability (FS) (%)	96.96 ± 5.24[a]	99.34 ± 1.13[a]
Emulsion activity (EA) (%)	28.56 ± 0.84[b]	39.81 ± 2.98[a]
Emulsion stability (ES) (%)	54.52 ± 5.06[b]	63.88 ± 2.40[a]

[1]USBF = Unprocessed sesame by-product flour; [2]ESBF = Extruded sesame by-product flour. a-b Different letters in the same row indicate significant differences ($p < 0.05$) between the means according to Fisher’s test.

Source: Own elaboration.

On the other hand, WSI is a measurement of starch degradation during the extrusion process increasing soluble polysaccharides (Dalbhagat *et al*., 2019) and is an indicator of the solubility of molecules present in the material (Udachan *et al*., 2017); WSI can be affected by the formation of amylose-lipid and protein-starch complexes during thermal processes (Du *et al*., 2014). WSI is associated with starch degradation during thermal processing, involving fusion, gelatinization, dextrinization, and starch solubility (Natabirwa *et al*., 2018; Dalbhagat *et al*., 2019). During the extrusion process, there is macromolecular degradation of both amylose and amylopectin, leading to the formation of low molecular weight molecules, resulting in increased water solubility of the extruded product (Dalbhagat *et al*., 2019; Ruiz-Armenta *et al*., 2022). An increase in extrusion temperature and screw speed causes an increase in pressure and shear force within the extruder, intensifying starch gelatinization and depolymerization, resulting in an increase in WSI (Ali *et al*., 2017).

While the OAI is related to flavor retention, enhancing emulsion properties, and the shelf life of functional ingredients (Álvarez-Ossorio *et al*., 2022). It is also associated with the hydrophobic groups of extruded products and the ability to maintain a certain amount of oil in their structure. In the extrusion process, starch granules gelatinize, and the hydrophilic/hydrophobic balance changes, thus altering oil absorption (Becker *et al*., 2014). Chandra *et al*. (2015) reported that proteins are the main component of flours that affect the oil absorption index due to the presence

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of hydrophobic and hydrophilic parts in their composition, where the non-polar amino acid side chains can form hydrophobic interactions with the hydrocarbon chains of lipids.

Dispersibility increased significantly ($p < 0.05$) after extrusion, by 81.7 %. Dispersibility depends on particle size, porosity, material density, and the chemical composition of the particle surface (Pismag *et al.,* 2024). It has been reported that extrusion temperature and moisture content of the sample influence the dispersibility of the sample (Nahemiah *et al.,* 2016). Some authors have reported an increase in dispersibility in materials extruded at high temperatures in rice-cowpea mixture (Nahemiah *et al.,* 2016), rice-soybean mixture (Nahemiah *et al.,* 2017), and millet-soybean mixture (Fernandes *et al.,* 2003) with temperatures of 100-180°C, 84-140°C, and 95-150°C, respectively. The temperature used in this work is within the reported ranges.

The emulsion property of an ingredient depends on the technique used for emulsification, the type of oil, and the methodology for determining emulsion capacity (Álvarez-Ossorio *et al*., 2022). Emulsion activity reflects the ability and capacity of proteins to assist in emulsion formation, relating to the protein's ability to absorb the interfacial area of oil and water in an emulsion (Du *et al*., 2014; Chandra *et al*., 2015); meanwhile, emulsion stability typically reflects the ability of proteins to strengthen an emulsion, providing resistance to stress and changes, and is also related to the resistance of the interfacial area over a certain period of time. Differences in emulsion activity and stability are related to protein content and other components, such as lipids and sterols. Interactions between protein-water occur in polar amino acid regions of protein molecules, and most proteins contain diverse polar side chains with peptides in the main chains, making them hydrophobic, affecting their solubility and emulsion properties (Du *et al*., 2014). Increased emulsion activity (EA), emulsion stability (ES), and fat binding during processing are primary functional properties of proteins in foods such as ground meat products, salad dressings, ice cream, and mayonnaise (Chandra *et al*., 2015).

On the other hand, foam formation and stability depend generally on the interfacial film formed by proteins, which keeps air bubbles suspended and retards the rate of coalescence. Foaming properties depend on proteins and other components like carbohydrates present in the material (Du *et al*., 2014; Chandra *et al*., 2015). Chandra *et al*. (2015) reported that flours with high foam formation capacity could form long air bubbles surrounded by a thinner and less flexible protein film, and these bubbles could collapse easily and consequently lose foam stability.

Conclusions

In this study, the extrusion process carried out under the optimized processing conditions maintained the nutritional quality of sesame by-product flour, as well as some techno-functional characteristics that serve as indicators to define the types of food products that could be developed using this material. The extrusion process improved the contents of soluble and total dietary fiber, as well as the water solubility index, dispersibility, activity, and emulsion stability of sesame by-product flour. These results suggest that it could be used in the production of a functional beverage and/or an emulsified product. It is necessary to develop and evaluate these types of products as an alternative for use in commercial products with low nutritional content, in addition to adding value to the sesame byproduct generated in the oil industry.

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Credit authorship contribution statement

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"All authors have read and agreed to the published version of the manuscript".

Declaration of Competing Interest

The authors declare no competing interests. The manuscript has not been submitted for publication in other media with substantial circulation. All previously published works cited in the manuscript have been duly acknowledged. All authors have made substantial contributions to the manuscript and approved the final submission. No conflicts of interest exist between the authors and the reviewers who assessed this manuscript. The preparation of this manuscript strictly adhered to the journal formatting provided in the authors' guide.

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