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RESEARCH ARTICLE
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Characterization of flour obtained by corn agroindustry waste and its application[1]

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ABSTRACT - Corn germ fraction with pericarp is produced by degermination. Due to its high fat content, as well as its fibrous and protein composition, this residue is commonly used for oil extraction or animal feed. However, literature is scarce on this bran, especially regarding its functional properties, which would help the milling industry find alternative uses and utilize it as a food ingredient in products for human consumption. Therefore, this work aimed to characterize corn degermination flour (CDF) obtained through mechanical separation of the bran. The flour was evaluated in terms of its proximate composition, physicochemical properties, technological functional properties, and its application in a "Mexican tortilla"- type product. The flour contained moisture within the legal limits for commercial flours and had a high content of lipids and proteins. Among the evaluated technological functional properties, the high water absorption capacity, good emulsifying properties, and gel-forming capacity stand out. However, the CDF did not exhibit foaming capacity. A sensory acceptance test of the co-product's use in tortillas was conducted, yielding promising results with an acceptability index of 79%. Thus, the research presented an alternative for utilizing and adding value to an industrial corn co-product applied in a food product, in addition to characterizing its physicochemical and technological functional properties.

Keywords: Coproduct. Corn degermination. Functional properties.

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INTRODUCTION

Corn is one of the most important and widely cultivated cereals in the world, as well as one of the main commodities produced in Brazil, characterized as a strategic product for food security (Faustino *et al*., 2020). Furthermore, corn is the main macro-ingredient for animal feed production in the meat production chain, particularly in pig farming and broiler production, as major drivers of national consumption (Souza *et al*., 2018). As corn is a highly nutritious grain, especially as a source of carbohydrates, it is estimated that approximately 40% of production is destined for animal feed, 12.3% is used for industrial consumption, and 1.4% is used for human consumption (Associação Brasileira das Indústrias do Milho, 2023).

The industrialization of corn yields a series of ingredients destined for a wide variety of industrial segments, generating several byproducts. In Brazil, the main corn milling industry is dry milling, obtaining degerminated endosperm fractions to produce *canjica*, grits (for extruded foods and other applications), and corn flour (Strazzi, 2015). In this context, one of the crucial steps in industrial processing is grain degermination, as it obtains an endosperm fraction that is the main raw material and another unique fraction consisting of pericarp and germ, called corn degermination bran (CDB), considered an industrial residue or byproduct.

According to data obtained by the Brazilian Association of Corn Industries (Associação Brasileira das Indústrias do Milho, 2023), the average yield in dry corn milling is 68% cracked corn and 30% germ adhering to the pericarp. Thus, it is observed that the corn milling industry generates a large quantity of bran that can be reused for oil extraction or to obtain a fibrous concentrate, since the germ contains approximately 82.6% lipids and 26% protein, and the pericarp contains approximately 54% fiber (Deepak; Jayadeep, 2022). However, some corn milling industries use this residue exclusively as animal feed.

The use of agro-industrial waste as a way to enrich food nutritionally can be a viable alternative for industries. However, this applicability will depend on the type of material, its chemical composition, and the processing method, in order to obtain potential ingredients for traditional products on the market. The successful use of these wastes or by-products is usually limited by a lack of knowledge of both the process or technologies that can be applied and their nutritional quality, as well as by a lack of financial investment.

Regarding corn degermination bran, some studies in the literature report the viability of its use in cookie-type biscuits, snacks (Silva *et al*., 2018), and animal feed (Qi *et al*., 2022). However, this residue is little explored for human consumption, and few studies involve its characterization or the determination of the functional properties of other flours (Boucheham *et al*., 2019; Santana; Oliveira Filho; Egea, 2017), since the practicality of animal feed interferes with investments and research on this potential ingredient. Therefore, this work aimed to analyze the corn degermination residue from a milling industry in relation to its granulometric characterization and obtain a co-product such as corn degermination flour (CDF) in order to characterize it in relation to its technological, physicochemical, and functional properties and test its applicability in a tortilla-type product.

MATERIAL AND METHODS

Obtaining corn degermination flour (CDF)

Corn degermination flour (CDF) was produced using the byproduct of dry milling degermination, supplied by a corn agro-industry located in Maringá, PR, which produces approximately 50 tons per day. The byproduct is similar to conventional corn bran, obtained from the separation of milling residues, and has a fibrous profile. The material was collected in 2 kg polyethylene bags and stored at -18 °C in a domestic freezer for subsequent analysis and characterization.

The production of the desired co-product was carried out in two stages. Initially, a mechanical separation of the bran was performed using an electromagnetic sieve shaker (Bertel) to separate the coarse fractions from the finer fractions using ABNT sieves. This separation was necessary due to preliminary application tests of the raw residue, since the bran had a very bitter aftertaste, in addition to containing larger and more visible fractions of pericarp and germ. These tests consisted of using the raw residue without prior separation by sieving and grinding it in a knife mill. In this way, the co-product obtained would be a more standardized granulometrically, homogeneous (35 mesh) flour that is easy to incorporate into a food product. After separation, physicochemical characterization analyses, determination of functional technological properties, and sensory testing of the Mexican tortilla were performed.

Particle-size analysis

The particle-size distribution of the raw residue was evaluated using a set of ABNT sieves with three different mesh openings (35, 60, 100 mesh and bottom pan). A 200 g sample was weighed, and the set of sieves was arranged in ascending order of mesh opening, kept under constant magnetic agitation for 15 min. At the end, the sieves were weighed on a semi-analytical balance, and the retained fractions were determined.

Corn degermination flour (CDF) was obtained by collecting the finest fractions that passed through the 35-mesh sieve.

Centesimal composition of corn degermination flour (CDF)

For the centesimal composition of the flour, the moisture content (circulating air oven at 105 °C for 8 h), ash content (muffle furnace at 550 °C for 2 h), and protein content (Kjeldahl method using a factor of 6.25) were determined according to the methods of the Adolfo Lutz Institute (Instituto Adolfo Lutz, 2008). Lipid content was determined according to the methodology of Bligh and Dyer (1959), crude fiber was determined according to Silva (1990), and carbohydrates were determined by difference. All determinations were made in triplicate.

Physicochemical characterization of corn degermination flour (CDF)

Flour pH was determined using the methodology of the Adolfo Lutz Institute (Instituto Adolfo Lutz, 2008), performed on a benchtop pH meter (AB23PH) previously calibrated with buffer solution (pH 4 and pH 7). Water activity was determined using the AquaLab 4TE Decagon Devices® equipment, by the direct method.

CDF color was determined using the Minolta Colorimeter at three different points on the product, by reading three parameters defined by the CIELAB system, namely L-a-b. L is the lightness (L = 0 black and L = 100 white), and a and b define the chromaticity (+a red and –a green, +b yellow and –b blue). The Hue angle was estimated using Equation 1.

$$Hue = \tan^{-1}\frac{b}{a} \quad (1)$$

Technological functional properties of corn degermination flour (CDF)

Water absorption capacity (WAC) was determined according to the methodology of Godswill (2019). A mixture of 10 mL of distilled water with 2 g of CDF was prepared and magnetically stirred (Quimis, Q261-22) for 5 min and subsequently centrifuged (Hettich, Universal 320R) at 3500 rpm for 30 min. The supernatant was reserved and the remaining material was weighed. The supernatant was used for the water solubility index (WSI) analysis, being evaporated in an oven (Cienlab, CE-205) at 105 °C.

Oil absorption capacity (OAC) was determined using the same methodology as for the water absorption capacity (WAC), replacing water with commercial soybean oil. Equations 2, 3, and 4 were used to estimate the functional properties for WAC, water absorption capacity (WAC), and OAC, respectively.

$$WAC = \frac{mass\ of\ water\ absorbed\ (g)}{mass\ of\ sample\ (g)} \quad (2)$$

$$WSI = \frac{mass\ of\ evaporated\ residue\ (g)}{mass\ of\ sample\ (g)} \times 100 \quad (3)$$

$$OAC = \frac{mass\ of\ oil\ absorbed\ (g)}{mass\ of\ sample\ (g)} \quad (4)$$

The emulsifying activity (EA) and emulsion stability (ES) properties of CDF were determined according to Yasumatsu *et al*., (1972). For the determination of EA, 1.0 g of flour sample was diluted in 10 mL of water and oil in the same proportion in graduated Falcon tubes (50 mL) and centrifuged (Hettich, Universal 320R) at 3000 rpm for 5 min. Emulsion stability (ES) was calculated after heating the tubes in a water bath at 80 °C for 30 minutes and cooling them in running water for 20 min, then centrifuging them (Hettich, Universal 320R) at 3000 rpm for 5 min. Equations 5 and 6 were used to determine these parameters, respectively.

$$EA = \frac{emulsifying\ layer\ volume\ (mL)}{total\ volume\ in\ the\ tube\ (mL)} \times 100 \quad (5)$$

$$ES = \frac{remaining\ emulsifier\ layer\ (mL)}{emulsifier\ layer\ in\ the\ tube\ (mL)} \times 100 \quad (6)$$

To analyze foaming capacity (FC), according to Coffmann and Garcia (1977), it was necessary to shake a solution containing 2% (w w^{-1}) CDF in distilled water for 5 min in a domestic shaker (Walita). The total volume was transferred to a 100 mL graduated cylinder so that the percentage increase could be estimated by the difference between the initial and final volume.

The swelling index (SI) was determined using the methodology of Godswill (2019). Approximately 4 g of sample was placed in a 250 mL graduated cylinder up to the 10 mL mark, with the addition of 50 mL of water. The top of the cylinder was firmly covered and mixed, then inverted. After 2 minutes, the suspension was inverted again, left to stand for another 10 minutes, and after 15 minutes the volume was observed. The index was determined based on the ratio between the initial and final volumes.

The gel-forming capacity of CDF was determined according to the procedure carried out by Santana, Oliveira Filho and Egea (2017) with modifications. Dispersions of 5, 10, and 20% (w v^{-1}) flour in 20 mL of water were prepared and heated in a water bath for 30 min at 90 °C and subsequently cooled to 4 °C for 2 h. At the end of the process, the tubes were poured and gel formation was visually analyzed.

Application of CDF in a food product

With the aim of utilizing the flour resulting from the residue of corn degermination, the Mexican tortilla was chosen because it is a practical product, normally made with corn and wheat flour. Preliminary tests were carried out for the development of the new product; however, it was observed that a tortilla with 100% CDF did not show good texture and flavor characteristics. Therefore, it was found that a reduction in CDF and a complementary addition of wheat flour provided sensory improvements.

The tortillas were produced using 50% (w w^{-1}) of CDF, 50% (w w^{-1}) of wheat flour, 50% (w w^{-1}) of water, 4% (w w^{-1}) of soybean oil, and 2% (w w^{-1}) salt (these ingredients being used in relation to the total amount of flour). First, the flours were mixed using a stainless steel spoon, followed by the oil and salt. Finally, water was added little by little until a homogeneous dough was formed, which was then kneaded manually for about 5 minutes until the dough reached the point where it could be shaped.

The dough was shaped manually into small balls (50 g) approximately 3 cm in diameter, which were then rolled out until very thin (2 mm). This dough was cut using a circular metal mold, and the circles were placed in a preheated Teflon electric pan (Super Chef, Britânia) at 160 °C for about 7 minutes.

Sensory analysis of tortillas

Sensory analysis was conducted at the State University of Maringá (Maringá, Brazil), where 100 untrained tasters aged between 18 and 30 years were recruited. They were provided with two mini tortillas, approximately 5 cm in diameter (40 g), along with a glass of water to cleanse their taste buds and a sensory evaluation form. To verify the acceptance of the tortilla with partial substitution of wheat flour by CDF, the tasters were instructed to affectively evaluate the parameters of color, flavor, aroma, texture, and overall appearance on a scale of 1 to 9, where 1 (disliked extremely), 5 (indifferent), and 9 (liked extremely). In addition, a possible purchase intention was also evaluated using a 3-point scale where 1 (would not buy), 2 (maybe would buy), and 3 (would definitely buy). Finally, the tasters could add comments about what they liked most and least about the product. The acceptability index (AI) was calculated from the average of the scores for overall appearance in relation to the maximum score of the scale used (9).

Statistical Analysis

The particle-size analysis data were processed in Excel, and the results were analyzed using descriptive statistics through mean and standard deviation.

RESULTS AND DISCUSSION

Particle-size analysis of corn germ bran

The bran from corn degermination was initially analyzed in relation to its particle-size distribution, as shown in Figure 1.

Figure 1 shows that 63.2% of the particles in the sample were 425 µm or larger, consisting of coarse particles such as parts of the pericarp and germ. Therefore, 35.4% of the sample was represented by the finest fractions with an average diameter of 250 µm. This analysis verified that this finest fraction corresponds to the corn degermination flour used for characterization in this study, as can be seen in Figure 2 on the right side.

The separation of the coarse fractions was carried out in order to remove particles that could impart a stronger rancid taste and odor to the small, coarse germ

Figure 1 - Particle-size distribution of corn germ residue bran

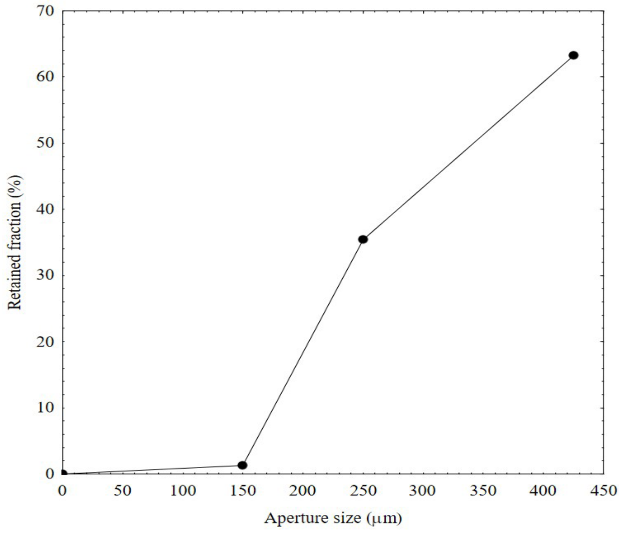

Figure 2 - Raw bran from corn degermination (left side) and flour obtained by sieving (right side)

fractions visible to the naked eye, as shown in Figure 2. Preliminary tests were performed by grinding the residue obtained from the factory to standardize the particle size. The residue was ground in a knife mill, to obtain flour passing through 35 mesh. However, the flour obtained did not show good sensory quality (very intense bitter taste) for application in food products, due to the probable rupture of lipid bodies and intensification of oxidation reactions.

Physicochemical characterization of CDF

Corn degermination flour (CDF) was characterized in relation to its centesimal composition as shown in Table 1.

It can be observed that the moisture content of the corn degermination residue flour reached 12.12%, since flours with moisture above 14% tend to form lumps, in accordance with Brazilian legislation that defines a maximum of 15% (w.b.) moisture for flours (Brasil, 2005). In order to achieve a good result for product development, it was not necessary to perform a drying operation on the flour to remove excess moisture.

Regarding the ash content, it was found that the co-product obtained had values higher than those in the literature (Silva *et al*., 2022). When comparing this with the legislation for whole wheat flour, which could have a composition similar to bran, it is observed that a maximum ash content of 2.5% is established. These values indicate the presence of mineral salts, and values above 1% indicate a greater quantity of bran adhering to the flour (Brasil, 2005). In this case, since the flour is the result of a residue from the processing of corn degermination, parts of the pericarp may be present, increasing the ash content.

Table 1 - Proximate composition and characterization of the physicochemical parameters of corn degermination flour (CDF)

Characterization	Values*
Moisture content	12.12 ± 0.16
Proteins	8.96 ± 0.09
Lipids	14.29 ± 1.29
Crude fiber	1.96 ± 0.14
Ash	2.78 ± 0.16
Carbohydrates**	59.89 ± 1.84
pH	6.73 ± 0.03
aw	0.62 ± 0.01
L	83.01 ± 0.83
a	-1.45 ± 0.15
b	32.42 ± 1.06
Hue	92.57 ± 0.33

*aw = water activity; L = lightness; a and b = chromaticity coordinates

When analyzing the main macronutrients, it was observed that the CDF had high levels of protein and lipids, compared to corn flour (Lopes; Castro, 2023; Silva *et al*., 2022). According to Naves *et al*. (2011), approximately 12% protein and 17.7% lipids were found in germ containing pericarp. The component fractions may vary according to the corn variety, sampling, as well as the moisture content present in the flour. Comparing with corn flour that undergoes the entire subsequent milling process after degermination and separation of the residue, the protein content (8.96%) of the CDF was close to the average composition of this cereal, such as 7.38% (Naves *et al*., 2011) and 9.37% (Dongmo *et al*., 2020).

Furthermore, CDF also stood out in its lipid content (14.29%), which was expected, since this co-product results from the corn degermination process, containing fragments of ground germ and parts of the pericarp. In this context, it is clear that CDF becomes an excellent ingredient to be explored for application in food products or to be extracted and/or purified for better utilization.

Regarding fiber content, only the crude fiber content was estimated, showing a low concentration. This result can be explained by the removal of the coarse fraction, which is likely responsible for the higher fiber content of the co-product. Furthermore, due to the high lipid concentration in the flour, a possible defatted CDF could have even higher concentrations.

Regarding the physicochemical characterization, it is observed that the CDF showed a neutral pH and a water activity within the acceptable limit (≤ 0.7) to prevent microbial growth. However, as verified in Table 1, the CDF showed a high concentration of lipids; therefore, care must be taken in its packaging and storage, as possible chemical and enzymatic reactions may be favored in an environment containing oxygen and light.

The instrumental color parameters observed in Table 1 show that CDF confers high lightness, since the closer to 100, the lighter the product. Furthermore, chroma b showed that the yellow hue is more intense (positive sign) than the green hue, represented by chroma a with a negative sign, confirming that the Hue angle value was in the 90° region, representing a yellowish hue. These results were expected, as according to Figure 2, the flour from corn degermination residue is yellow.

Characterization of the technological functional properties of CDF

For a better understanding of the application of a particular type of flour in food products, it is important to analyze the performance of its ingredients and its technological behavior in food systems during processing. In this context, technological characteristics are required to possibly predict and evaluate how proteins, fats, carbohydrates (starch and sugars), and/or fibers may behave in specific formulations to be developed, as well as to assess whether or not the flour can be used to replace a particular ingredient, offering improvements in product stability (Awuchi; Igwe; Echeta, 2019). Table 2 presents the main technological properties of corn degermination flour.

Properties such as water absorption, water solubility index, and swelling index are functional hydration properties, related to the polar constituents present in flour such as carbohydrates (especially polysaccharides), proteins, and other hydrophilic constituents. When analyzing the results in Table 2, it can be observed that the water absorption capacity showed a consistent value compared to those reported in the literature, for instance 2.48 g g^{-1} for semolina flour and 2.36 g g^{-1} for rice flour (Boucheham *et al*., 2019). The corn flour obtained by Boucheham *et al.* (2019) showed a higher value (5.31 g g^{-1}) than the corn byproduct used in this work, which can be explained by them being different fractions with distinct compositions. In percentage terms, CDF showed 257% water absorption, a value higher than that found in rice flour (172%) and close to that found in taro flour (245%) by Kaushal, Kumar and Sharma (2012), and lower than that estimated for millet flour (359.3%), according to a study by Akinola *et al*. (2017). The water absorption capacity of flours is associated with protein interactions, being a critical function in viscous foods and useful in products that require hydration, increasing viscosity, thickening and also preventing the product from drying out during storage, as occurs in breads and cakes (Akinola *et al*., 2017).

Regarding the water solubility index (WSI), the CDF did not show a value as high as that reported in the literature, where white bean, golden flaxseed, and brown flaxseed flours obtained values above 10%, according to Santana, Oliveira Filho, and Egea (2017). This property is related to the ability of hydrophilic constituents present in the flour to dissolve in water. Due to the high lipid concentration present in the CDF, in addition to some soluble protein fractions, components such as sugars and soluble fiber may have migrated into the solvent. According to Aditi and Arivuchudar (2018) and Suraiya, Ihsan, and Ishfaq (2016), high WSI values are related to the degree of starch degradation, releasing polysaccharide molecules, while low values may indicate that the starch granule complexed with lipids and proteins can reduce the solubility of the flour.

Table 2 - Results of the technological functional properties of corn degermination flour (CDF)

Technological functional properties	Values*
Water absorption capacity (WAC) (g_{water} g_{sample}^{-1})	2.57 ± 0.01
Water Solubility Index % (g $100g^{-1}$)	8.90 ± 1.15
Oil absorption capacity (OAC) (g_{oil} g_{sample}^{-1})	0.97 ± 0.004
Swelling index (mL g^{-1})	4.73 ± 0.33
Emulsifying activity % (mL $100mL^{-1}$)	37.66 ± 2.59
Emulsion stability % (mL $100mL^{-1}$)	104.31 ± 3.73

*Mean ± standard deviation (triplicate)

The swelling index or swelling volume is a measure of the starch's ability to absorb water, as well as other carbohydrates and proteins present, and is dependent on the flour variety, particle size, temperature, and processing methods (Awuchi; Igwe; Echeta, 2019). In the literature, there is little comparative data regarding this property, especially in relation to cereal flour. In this context, CDF flour obtained a swelling capacity similar to that of unmodified wheat flour, as reported by Kaur *et al.* (2016). This property is a quality measure for baked goods, and higher values could be achieved if the solvent used were heated, which was used to evaluate the gel-forming capacity.

Regarding oil absorption capacity (OAC), it was observed that CDF exhibited a low value compared to rice flour determined in the study by Latorres *et al*. (2014), and close to that of wheat flour reported by David *et al*. (2015). This property is related to the association of the oil used with the non-polar groups of proteins and/or the availability of lipophilic groups (Awuchi; Igwe; Echeta, 2019). Furthermore, the composition of insoluble fibers can also contribute to oil absorption capacity, which in this study was determined only for crude fiber, normally composed of cellulose, hemicellulose, and lignin. Therefore, the low concentration of crude fiber identified may have influenced the lower oil absorption capacity.

The emulsifying capacity and stability of the emulsion formed by CDF were analyzed and showed satisfactory results for application in emulsified products such as meat products, sauces, and other products. These properties may be related to the quality and quantity of soluble protein in the flours, since the flour had a considerable total protein content, which may also have contributed to its hydration properties. When comparing with the literature, it is verified that CDF showed an emulsifying activity similar to that of oat flour found by Santana, Oliveira Filho and Egea (2017).

The gel-forming capacity was also evaluated. To this end, different concentrations of flour were tested to verify if the amount added influenced the formation and strength of the gel. The results are presented in Table 3.

Table 3 shows that, as the amount of flour increased to 10% (w w^{-1}), a more consistent gel formation occurred. The gelation phenomenon results from the formation of a three-dimensional carbohydrate network, modified or not by thermal processes (Adebowale; Lawal, 2003). In comparison with other flours, Santana, Oliveira Filho and Egea (2017) observed that the gels formed were more fragile at the same concentration used in this work. Thus, it is verified that CDF could be applied to the production of products that require the formation of a cohesive and firm mass as a consequence of starch gelatinization, such as bakery products.

Sensory analysis of CDF application in tortilla

The sensory analysis of the tortilla using 50% wheat flour and 50% CDF was statistically analyzed as shown in Table 4.

In the present study, Table 4 shows that color and flavor received the highest scores compared to the other attributes. Although texture received a lower rating, the overall appearance of the tortilla was well evaluated, corresponding to an acceptability index of 79%.

The tasters were also instructed to describe the positive and negative aspects of the product. In this regard, approximately 70% of the tasters reported in the sensory analysis sheets that the texture of the tortilla was the factor that most compromised the overall appearance. As the tortillas were served several hours after preparation, they lost some of the expected crispness for this type of product. To be considered acceptable by consumers, a product must have a minimum acceptability index (AI) of 70%. Thus, with satisfactory acceptance, it is concluded that the partial substitution of wheat flour with corn degermination flour is a good alternative to conventional products that may have alterations in their formulations, without compromising the desires of the target audience and emphasizing functional characteristics such as increased protein.

Table 3 - Gel-forming capacity of corn degermination flour (CDF)

% CDF (w w^{-1})	Gel formation*
5	±
10	+
20	+

*Tests performed in triplicate: Strong gel (+); Fragile gel (±); Absence of gelation (-)

Table 4 - Results of the Sensory Analysis of the tortilla with the application of corn degermination flour (CDF)

Parameters	Evaluation scores*
Color	7.28 ± 1.48
Flavor	7.08 ± 1.68
Aroma	6.8 ± 1.63
Texture	6.36 ± 1.78
Overall appearance	7.1 ± 1.49
Acceptability index (AI)	79%

*Mean ± standard deviation

CONCLUSIONS

The residue from corn degermination processing was characterized, yielding flour with an average size of 250 µm and a composition of 8.96% protein and 14.29% lipids. The functional and technological properties of the flour were evaluated, showing suitable behavior for incorporation into cooked products that maintain a firm gel structure, such as bakery products, as well as emulsifying characteristics for meat products. The application of corn degermination flour in Mexican tortillas showed good acceptability (79%) among young people, who mostly need practical and healthy foods for their daily routine.

The research presented an alternative for developing a new product by utilizing an industrial waste product that previously had no application in human food. However, further studies regarding the protein, amino acid, and fatty acid profiles, as well as bioactive compounds, should be considered to analyze the viability of other possible applications of this co-product. Therefore, a more in-depth study could assist the corn agro-industrial sector in making better use of the generated waste and adding value to both corn degermination flour and the raw bran itself.

DATA AVAILABILITY

The entire dataset supporting the results of this study is available upon request from the corresponding author.

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