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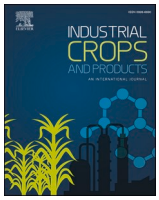

Dry fractionation of solvent-extracted castor meal

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ABSTRACT

Castor meal is the main by-product from the industrial process of castor oil extraction. It is a nitrogen-rich material with anti-nematode effect, which has been predominantly used as organic fertilizer. Castor meal was dry fractionated by sieving and air classification, and nutritional and physicochemical properties were measured in each fraction. Near Infrared Spectroscopy was tested for accuracy to estimate the nutritional composition of the fractions. Sieving and air classification promoted significant separation of the inputs that compose castor meal. The coarsest fraction (particles > 2.0 mm) was predominantly composed of fruit husk fragments, and it had low crude protein, (16.5–24.7 %), high acid detergent fiber content - ADF (40.3–46.5 %), and high potassium content (13.8–14.2 g/kg). The finest fractions (particles < 0.5 mm) were predominantly composed of seed kernel fragments, and they had high protein content (39.6–49.6 %), low ADF (12.8–25.7 %), and high phosphorus content (14.7–20.2 g/kg). The highest ADF (59.2 %) was found in the fraction predominantly composed of seed coat fragments (light particles, 1.4–2.0 mm). The lowest water holding capacity was found in the fractions with predominance of seed coat fragments, and the highest in the fractions composed of fruit husks or seed kernel. Clay is an input employed in the industrial process that goes into castor meal and influences its composition, especially the ashes content. Near Infrared Spectroscopy was very accurate to estimate the content of protein, lignin, and fibers in the dry fractions of castor meal.

1. Introduction

The oil extracted from castor seed (*Ricinus communis*) is a high-value material used for the fabrication of a long list of products in the fine chemicals industry. The meal resulting from castor oil extraction is a nitrogen-rich powder with anti-nematode effects, which has been predominantly used as organic fertilizer (Galbieri et al., 2024; Severino et al., 2021; Sousa et al., 2022), and which is toxic because of the presence of ricin. The castor oil industry aims to take advantage of its high protein content to add value to this by-product by using it as an ingredient in ruminant feed. The possibility of using castor meal as a feed ingredient was strengthened after several studies demonstrated that: (i) the regular process of solvent extraction inactivates the toxic protein ricin to a level that is tolerable by ruminants, (ii) microbes in the rumen are able to digest the residual ricin in the meal, and (iii) experiments demonstrated that ruminants have normal growth and health conditions feeding on detoxified castor meal (Diniz et al., 2011; Oliveira et al., 2010, 2015a, 2015b; Rocha et al., 2022; Silva et al., 2015).

The value of castor meal would be optimized if the material was fractionated, and each fraction was characterized and optimized for a specific use. For instance, nutritional qualities are required for a feed ingredient, agricultural functions are important in an organic fertilizer, and physical characteristics define the value of the fraction to be burnt for heat or employed as a construction material. A previous study demonstrated that castor meal could be separated by sieving in fractions with contrasting nitrogen content (Severino et al., 2021). Then, it was hypothesized that the value of the meal could be optimized if it could be separated in a high-protein fraction to be potentially marketed as feed ingredient and high-fiber fractions to be directed for other ends.

There are many methods employed for the fractionation of protein-rich seed flours, and they can be grouped as wet and dry methods. Wet methods can produce isolates with protein content in excess of 80 %; however, they are too intensive in water, energy, chemical inputs, and effluents to be treated (Rivera et al., 2024). Nevertheless, wet methods are the predominant choice in industrial-scale for protein concentration in oilseed meals (Arrutia et al., 2020). Whenever possible,

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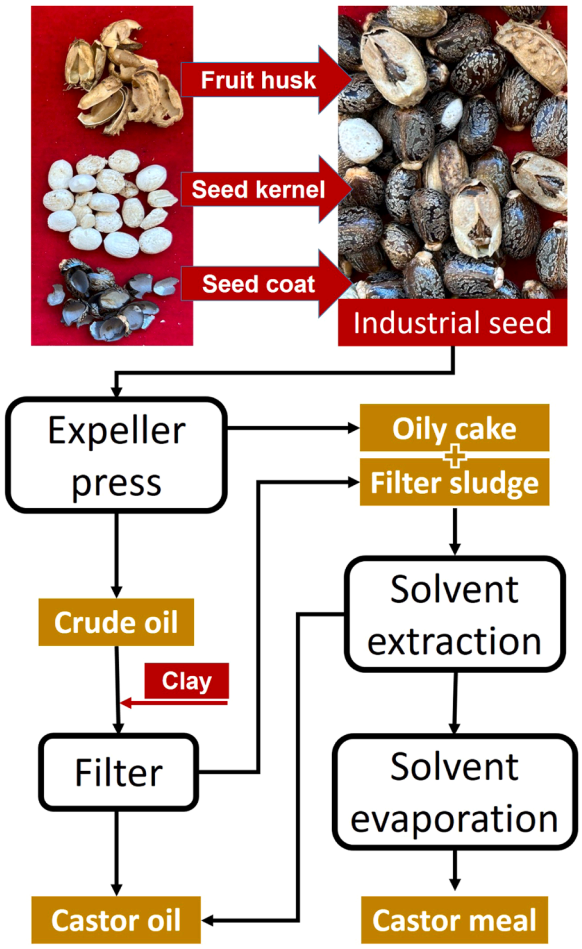

Fig. 1. Diagram of the industrial process of castor oil extraction highlighting the main four inputs, steps, and two outputs.

dry methods are preferred by the industry because they are more energy-efficient and do not require chemical inputs, although they have limited capacity to reach high protein content (Hansen et al., 2017; Rivera et al., 2024; Vidosavljević et al., 2022). Dry fractionation can be performed by methods such as milling, sieving, electrostatic separation, and air classification. Separation by sieving depends mainly on the particle size, while the air classification relies on the combination of the particle size, density, and tridimensional configuration (aerodynamic) (Rivera et al., 2024; Fernando, 2021). The electrostatic separation relies on the difference in tribo-electric properties among the particles in the meal (Arrutia et al., 2020; Laguna et al., 2018).

If the high-protein fraction is to be used as ingredient in ruminant feed, it needs to be characterized for key nutritional characteristics such as protein, fiber, ashes, and lignin (Gaffey et al., 2023). For using some fractions as organic fertilizer, they need to be characterized for the nutrients content. The fractions also need to be characterized for potential uses that depend on properties like heat of combustion and water holding capacity (Bala-Litwiniak and Musial, 2024).

The castor seed received by the oil extraction industry has highly variable composition, particularly on the content of fruit husks and seed coat. The raw material is produced by farmers with a broad diversity of varieties, harvesting methods, and rigor on quality control. On the one hand, the composition of the industrial castor meal produced is variable because of the irregular quality of the raw material. On the other hand, the users of the meal require that its properties should vary in a narrow range if it is to be used as input in other industrial processes. For that reason, the oil extraction industry needs to monitor the industrial process to assure the quality and uniformity. Methods based on Near Infrared Spectroscopy are widely used in the industry for monitoring the composition and quality of materials because it allows fast results, at low cost without using chemicals (Lebot et al., 2009).

This study tested the following hypotheses: (i) that castor meal can be dry fractionated by sieving followed by air classification; (ii) that the inputs in the process of oil extraction (fruit husk, seed coat, seed kernel, and clay) can be quantified in the fractions, and (iii) that Near Infrared Spectroscopy is accurate to estimate the composition of castor meal dry fractions.

2. Material and methods

2.1. Industrial process of castor oil extraction and collection of samples

This study was made with samples collected in an industry of castor oil extraction (A. Azevedo Óleos Ltda., Itupeva-SP, Brazil). The main components of the raw material received by the industry consists of the clean seed with a variable content of fruit husks as impurity, which were considered because they largely influence the meal's composition (Fig. 1). For the objective of this study, the seed was considered to be composed of the seed coat and the seed kernel, which corresponds to the endosperm and embryo or the "white structures" inside the seed (Severino et al., 2015). There are other impurities with the seed (sand, woody materials, plastic and metal objects) that are not discussed because they are removed before the process and do influence the castor meal composition. The seed goes through cleaning operations, is warmed to about 90 °C by both dry heat and steam injection, and enters the expeller press. The expeller press applies pressure and shear to extract the oil from the seed, resulting in the crude oil and the oily cake. At this step, the cake still contains a significant amount of oil (ca. 10 %) (Avramovic et al., 2024; Arrutia et al., 2020; Liu et al., 2016).

For the filtering step, the crude oil is mixed with clay (Tonsil®), which is an acid-activated bentonite that removes pigments

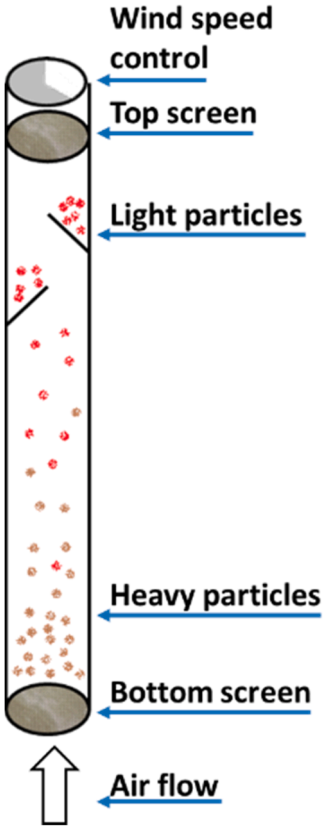

Fig. 2. Diagram of the wind separator used for air classification of castor meal.

(chlorophyll, carotenoids) and retains fine particles suspended in the oil (El-Hamidi and Zaher, 2016). The filtering operation produces a sludge containing clay, impurities removed from the oil, and a significant content of castor oil. The oil-rich sludge is mixed with the oily cake coming from the expeller press and goes through the solvent extraction with hexane to recover the remaining castor oil (Fig. 1). After being washed with the solvent, the wet meal is exposed to approximately 105 °C for 25 minutes to evaporate and recover the solvent. For a deeper review on the industrial process of castor oil extraction, see Avramovic et al. (2024).

This study used five samples of castor meal, weighing 0.8 kg on average, collected immediately after the step of solvent evaporation, stored on sealed plastic bags, and kept under refrigeration (8 °C) until processed in the laboratory. The samples were collected along two days of regular operation of the industry in October/2023 (3 samples) and two days in April/2024 (2 samples). There was no experimental control over the seed entering the industrial process, and some variability in its composition was expected.

2.2. Dry fractionation of castor meal

The castor meal had many agglomerates formed by the pressure applied in the expeller press. The visible agglomerates were broken up in a mortar grinder (Retsch RM 200) until there were no more agglomerates greater than 2.0 mm. Then, the samples of castor meal were passed through four sieves with openings of 2.0, 1.4, 0.5, and 0.25 mm. The sieves were stacked, the meal was placed in the top and vibrated with a sieve shaker (Bronzinox ABME 0800) for five minutes. The sieving process resulted in five dry fractions.

Each sieve-fraction was air classified using a wind separator (Deleo). The fraction was placed inside the tube, the wind flow was turned on, and the window on the top was adjusted by hand to control the wind speed aiming to separate the light particles (Fig. 2). The air classification was not continuous, but it was made in multiple batches of approximately 100 g. The fraction that remained in the bottom of the tube was assumed as "heavy particles" and the fraction transported to the top was assumed as "light particles". The intensity of separation was not controlled *a priori*, but it was measured after the air classification as the ratio between the weight of the light particles and the total weight of the fraction. The fraction with particles smaller than 0.25 mm could not be air classified because it was a thin powder that was not retained by the top screen of the equipment. The fractionation with sieving and air classification resulted in nine fractions (four particles size x two air classifications + particle size < 0.25 mm), and they were obtained from five samples of castor meal.

2.3. Inputs in the industrial process of castor oil extraction

Samples of the inputs used in the industrial process of castor oil extraction were collected in the industry in the same days that the castor meal was sampled. In the laboratory, the seed was separated by hand into fruit husks, seed coat, and seed kernel, and each input was ground to pass a 1.4 mm sieve and subjected to the same analysis of the dry fractions. The sample of seed kernel was defatted using an automated lipid extraction system (Ankom XT15) with ether as solvent (Luthria et a., 2019). One sample was taken from the clay that is added to the crude oil before filtering (Fig. 1).

2.4. Neutral and acid detergent fiber and lignin

All the analysis were made in five replications of the dry fractions and three replications of the inputs, resulting in 57 samples. The fiber in neutral and acid detergent, and lignin contents were measured according to Van Soest et al. (1991) using an Ankon 2000 Fiber Analyzer. Ankon bags were filled in duplicate with 0.5 g of the material to be analyzed and heat-sealed. As control, five empty bags were subjected to the same procedures. The bags were subjected to continuous circulation of neutral detergent for 60 minutes at 100 °C, and then rinsed in water, oven dried at 105 °C for 12 h, and weighed to estimate the neutral detergent fiber. For measuring the acid detergent fiber, the same bags were subjected to the acid detergent for 75 minutes at 100 °C, rinsed in water, oven-dried, and weighed. For measuring the lignin content, the same bags were washed with sulfuric acid for 3 hours at room temperature, rinsed in acetone, oven-dried and weighed (Cequier et al., 2019). After this step, the residual ashes were measured in each bag (as described in Section 2.5) just for correcting the lignin content. Calculations of each component were made by weight difference, considering the weight of empty bags as correction. The results were not corrected for the moisture content of the material.

2.5. Ashes, moisture, and crude protein

Moisture and ashes were measured according to the method ASTM E1755 – 01. Duplicate samples of 2 g were placed in porcelain crucibles, warmed to 105 °C for 12 hours, cooled to room temperature in a desiccator, and weighed to measure the moisture content. The same crucibles were then incinerated in a muffle at 600 °C for 12 hours, cooled in a desiccator, and weighed to estimate the ashes content. The ashes were used for measuring the content of micronutrients as described in the Section 2.7.

The Kjeldahl method was used to estimate the total nitrogen content according to Silva (2009). In short, duplicate samples of 2 g were placed in digestion glass tubes, mixed with sulfuric acid and Na_2SO_3 as catalyst, digested at 420 °C, mixed with sodium hydroxide, distillated to a solution of boric acid, and titrated with a solution of sulfuric acid. The nitrogen content was multiplied by the factor 6.25 to obtain the crude protein content. The results were not corrected for the moisture content of the material.

2.6. Heat of combustion and water holding capacity

The heat of combustion (calorific power) was measured with a Parr 6400 Calorimeter (Parr Instrument Co., Moline, IL, USA) according to the method ASTM D240 (Freedman and Bagby, 1989). Duplicated samples of 0.4 g were placed in metal caps and incinerated in the calorimeter. The samples were analyzed with natural moisture content. The samples of clay could not be incinerated because that is a non-organic material, and their heat of combustion was assumed as zero.

The water holding capacity was measured in samples of 15 g of each fraction, placed in drained plastic cups in a plastic tray. The tray was slowly filled with water until it reached ¾ of the height of the samples inside the cups. The water entered in the cup by the drains in the bottom and soaked three quarters of the sample, while the water saturated the quarter on top by capillarity. The sample was left soaking for 12 hours, and then the cups were drained for two hours. The water-saturated material was transferred to aluminum weighing boats, weighed, oven dried for 24 h at 105 °C, and weighed. The water holding capacity was calculated as the difference between the wet weight and dry weight divided by its dry weight.

2.7. Nutrients content

The content of macronutrients and micronutrients was measured according to Silva (2009) in each fraction of castor meal and inputs. The samples for measuring the content of macronutrients (P, K, S, Ca, and Mg) were digested with a mix of nitric acid (HNO_3) and perchloric acid ($HClO_4$) in the proportion of 3:1 (v:v) and heated at 200 °C for 4 hours (Silva, 2009). The measurement of minor nutrients (B, Cu, Zn, Fe, and Mn) was made in the ashes that were generated in the measurement of ashes content (Section 2.5). The ashes were digested in nitric acid (Silva, 2009). The content of each nutrient was measured in the extracts with spectrophotometry of atomic absorption (PerkinElmer, model

Fig. 3. Fractions of castor meal separated by sieving in five fractions according to the particle size. The red squares (on the left) inform the dimension of the sieve openings. The photographs in the left depict the observation with naked eye, and in the right are microscope pictures with magnifications from 1.5 to 5 x.

AAnalyst200) using the hollow cathode lamp with wavelength specific for each atom to be detected (Silva, 2009).

2.8. Comparison of characteristics of the fractions and inputs

The data on the physical, chemical, and nutritional characteristics of the fractions and the inputs in the industrial process of oil extraction were subjected to analysis of variance, and the means were compared using the test of Tukey ($p < 0.05$). The means of the fractions were not compared with the inputs. Statistical analysis were made with the software R.

2.9. Near Infrared Spectroscopy

Sample preparation and near infrared spectra acquisition were made as described by Bittner et al. (2016). The material was spread on a glass disc, without further milling, at room temperature, and at natural moisture. The reflectance spectra was acquired in the range of 2500–1000 nm, with 4/cm intervals, on a Nirflex N500 (Büchi Labortechnik AG, Flawil, Switzerland) on the samples of castor meal fractions and inputs. The data was imported to the software NIRCalc, and each spectrum was transformed by standard normal variate (SNV) and first derivative Savistsky-Golay of 9 points (dg1). The models were calibrated for crude protein, neutral and acid detergent fiber, and lignin. The models were calibrated using partial linear square discriminant analysis regression (PLS) with the transformed spectra and measured values of each characteristic. Calibration of the model was made with 43 randomly chosen spectra, and the accuracy was tested with 14 spectra. The accuracy was the coefficient of determination (R^2) of a linear regression between the measured and the predicted values in the set of test.

2.10. Content of fruit husk, seed coat, seed kernel, and clay in each fraction

The content of each input in the composition of each fraction of castor meal was estimated using multiple regression analysis. The chemical properties of the four inputs (fruit husk, seed coat, seed kernel, and clay) were assumed as the independent variables ($X_1 \ldots X_4$), and the chemical properties of each fraction was assumed as the dependent variable (Y). Initially, the analysis was made with the 17 variables measured in this study. The values of each characteristic was standardized dividing the value by its mean (fractions and inputs pooled). The standardized values of the 17 chemical properties for one given fraction were subjected to the model at the same time. One mean value for each chemical property of the input (X) was repeated for the five

Table 1
Relative weight (%) of each fraction of castor meal separated by sieving (particle size) and air classification (particles density).

Particles size	Light	Heavy	Total
> 2.0 mm	6.8 %	12.0 %	18.8 %
1.4 – 2.0 mm	9.2 %	15.4 %	24.7 %
0.5 – 1.4 mm	9.2 %	23.3 %	32.5 %
0.25 – 0.5 mm	4.7 %	6.1 %	10.8 %
> 0.25 mm	13.2 %		13.2 %

replications of the fractions. A multiple regression equation was calculated with the following model.

$$Y = a_1X_1 + a_2X_2 + a_3X_3 + a_4X_4 + b + \varepsilon$$

Y = standardized values of all characteristics in a given fraction of castor meal;
a_1 to a_4 = regression coefficients that estimate the contribution of each input
X_1 = standardized mean value of each characteristic in the seed kernel;
X_2 = standardized mean value of each characteristic in the seed coat;
X_3 = standardized mean value of each characteristic in the fruit husk;
X_4 = standardized mean value of each characteristic in the clay;
b = intercept of the equation
ε = error of the model.

The regression coefficients a_1 to a_4 were assumed as estimation of the content of the respective input into a given fraction of castor meal. After the initial estimation using 17 variables was performed, the deviations between observed and estimated values were calculated for each variable, and the five variables with the greater mean deviation were excluded from the analysis. The analysis was made again with 12 variables. Calculations were made with the software Excel.

2.11. Intensity of air classification

The intensity of air classification was calculated dividing the weight of the light fraction by the initial weight of the sample. A simple linear regression was calculated with the intensity of air classification as the independent variable, and the characteristics of the light fraction as the dependent variable. The regression analysis was considered significant when $p < 0.1$. The analysis of ashes and protein were presented graphically, and the analysis of other five properties were presented as the coefficients of regression analysis in a table. The regression line was solid when significant and dashed otherwise. Calculations were made in the software SigmaPlot.

3. Results

3.1. Physicochemical characterization of dry fractions

The dry fractionation of castor meal was easy and very effective, resulting in visually distinct materials (Fig. 3). The fraction with the largest particles (> 2.0 mm) corresponded, on average, to 18.8 % of the castor meal weight, the fraction with particles between 1.4 and 2.0 mm weighed 24.7 %, and the next fraction (0.5 – 1.4 mm) weighed 32.5 % of the initial sample of castor meal (Table 1). The weight of the two finest fractions were 10.8 % (particles 0.25–0.5 mm) and 13.2 % (particles < 0.25 mm) of the castor meal. The weight proportion between light and heavy particles is not an intrinsic characteristic of the meal, but it just reflects the intensity of the air classification applied in this study.

The fractions of castor meal were highly divergent in chemical, physical, and nutritional characteristics (Table 2). The fraction with large particles (> 2.0 mm) was low in protein and high in both neutral and acid detergent fibers. The lignin content was considerably higher (from 28.1 % to 44.2 %) in the fractions with particles between 0.5 and 1.4 mm, while it was the lowest in the particles smaller than 0.5 mm (below 15.4 %).

The inputs were also very divergent in all the characteristics. The fruit husk had low protein (9.0 %), medium ashes content (7.8 %), a high neutral detergent fiber (56.3 %) associated with a low acid detergent fiber (38.1 %), low lignin content (5.7 %), heat of combustion of 4.37 kcal/g, and the highest water holding capacity (510.3 %). The seed coat was very poor in protein (6.0 %) and had low ashes content (3.6 %), while it had high content of both neutral detergent fiber (81.0 %) and acid detergent fiber (62.8 %), and the highest lignin content among all the inputs (62.8 %). The seed coat had also heat of combustion of 4.82 kcal/g, and the lowest water holding capacity (219.7 %). The seed kernel was the richest in protein (54.8 %), with ashes content of 6.7 %, a low content of neutral detergent fiber (34.9 %) associated with an even lower acid detergent fiber (6.8 %), and very low lignin content (0.46 %). The seed kernel had also the highest heat of combustion (5.54 kcal/g) and water holding capacity of 430.7 %.

Table 2
Physical, chemical, and nutritional characteristics of nine dry fractions of castor meal separated by sieving and air classification and of the four inputs in the industrial process of castor oil extraction.

		Moisture content (%)	Crude protein (%)	Ashes (%)	Neutral detergent fiber (%)	Acid detergent fiber (%)	Lignin (%)	Heat of combustion (kcal/g)	Water holding capacity (%)
Fractions separated by sieving and air classification									
Sieve opening	Density								
> 2.0 mm	Light	6.4^{a}	16.5^{a}	5.9^{a}	62.5^{c}	46.5^{c}	20.5^{cde}	4.30^{cde}	382.2^{cd}
	Heavy	6.7^{a}	24.7^{ab}	10.5^{b}	52.7^{bc}	40.3^{c}	18.9^{cd}	4.11^{b}	326.8^{bc}
1.4 – 2.0 mm	Light	7.2^{a}	15.9^{a}	5.7^{a}	68.7^{c}	59.2^{d}	44.2^{g}	4.52^{f}	242.6^{a}
	Heavy	7.3^{a}	28.2^{b}	10.4^{b}	51.0^{bc}	41.6^{c}	28.1^{de}	4.20^{bcde}	266.5^{ab}
0.5 – 1.4 mm	Light	7.7^{a}	31.2^{bc}	7.2^{a}	50.1^{b}	40.6^{c}	31.1^{ef}	4.35^{def}	314.3^{abc}
	Heavy	7.3^{a}	25.7^{ab}	8.3^{ab}	60.3^{bc}	51.2^{cd}	41.3^{fg}	4.37^{ef}	243.8^{a}
0.25 – 0.5 mm	Light	8.1^{a}	49.0^{d}	10.1^{b}	24.6^{a}	13.5^{a}	5.3^{ab}	4.13^{bc}	408.9^{d}
	Heavy	7.5^{a}	39.6^{cd}	10.3^{b}	35.0^{a}	25.7^{b}	15.4^{bc}	4.19^{bcd}	328.7^{bcd}
< 0.25 mm	-	7.9^{a}	49.6^{d}	15.0^{c}	32.3^{a}	12.8^{a}	3.0^{a}	3.92^{a}	363.3^{cd}
Inputs in the industrial process of castor oil extraction									
Fruit husk	-	6.4^{ab}	9.0^{c}	7.8^{c}	56.3^{c}	38.1^{b}	5.7^{b}	4.37^{b}	510.3^{b}
Seed coat	-	7.6^{b}	6.0^{b}	3.6^{a}	81.0^{d}	74.0^{c}	62.8^{c}	4.82^{c}	219.7^{a}
Seed kernel	-	4.0^{a}	54.8^{d}	6.7^{b}	34.9^{b}	6.8^{a}	0.46^{a}	5.54^{d}	430.7^{b}
Clay	-	7.5^{b}	0.6^{a}	85.7^{d}	7.9^{a}	6.6^{a}	0.49^{a}	0^{a}	218.5^{a}

The means followed by different superscript letters in the column are significantly different (test of Tukey, $p < 0.05$). The means of the fractions were not compared with the means of the inputs.

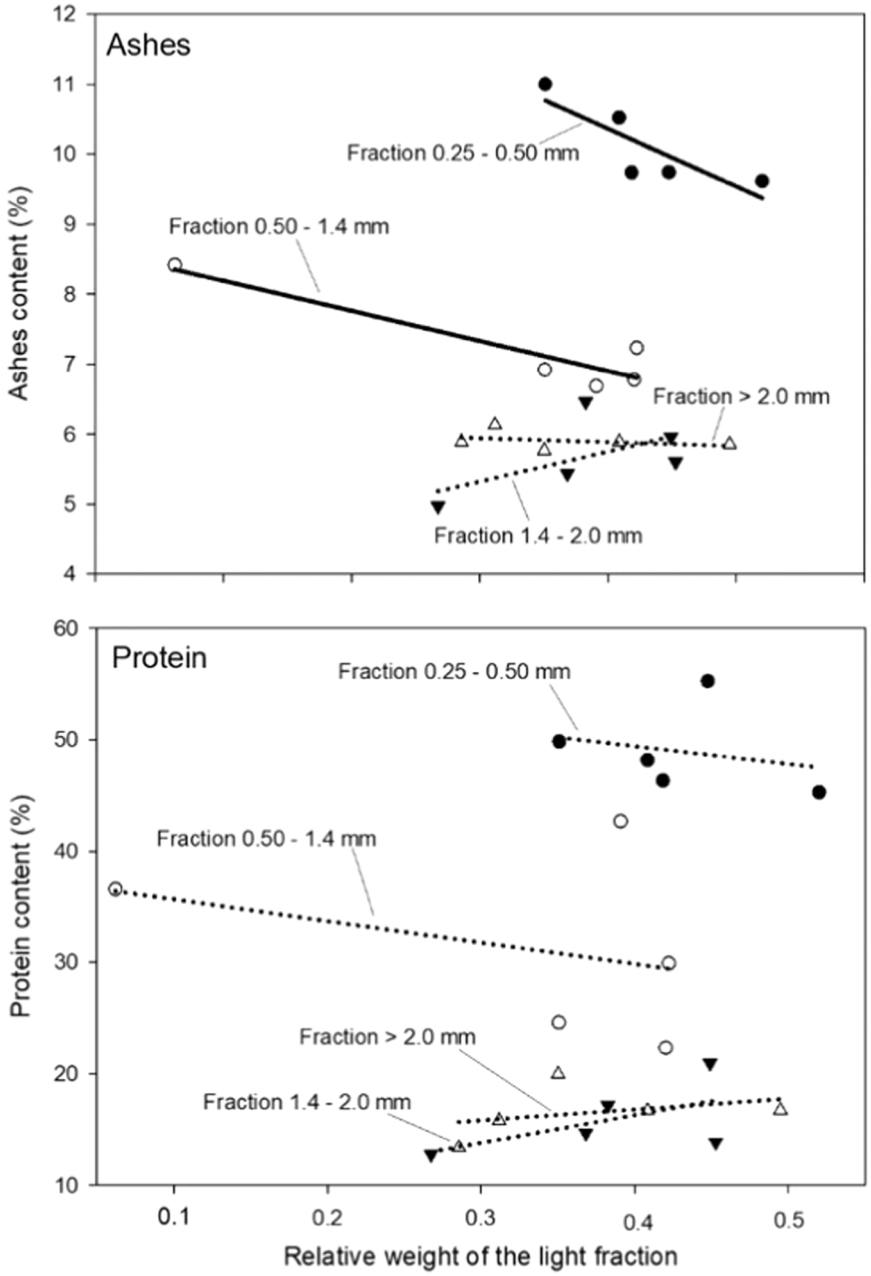

Fig. 4. Effect of the intensity of air classification on the content of ashes and protein of the light particles in four dry fractions of castor meal separated according to the particles size. Solid lines are a significant slope ($p < 0.1$).

It is not usual measuring nutritional characteristics in clay, but it was necessary because clay is an important component of castor meal, and it plays a role if castor meal is used as feed ingredient. It was found that the clay had almost no protein (0.6 %), while the ashes (85.7 %) were the most relevant component. Clay's fiber content was very low in both neutral (7.9 %) and acid detergent (6.6 %), while the lignin content was near zero (0.49 %), the heat of combustion could not be measured because the material is not combustible, and it was able to hold water in 218.5 % of its dry weight.

The air classification was effective to separate some components of the fractions that had been previously fractionated by sieving. For instance, in the two fractions with particles larger than 1.4 mm, protein-rich fragments remained in the heavy fraction, while the opposite effect occurred in the fine fractions, in which the wind carried the protein-rich particles to the light fraction (Table 2). It was observed that the intensity of air classification resulted in proportional change in some characteristics. In the sieve-separated fractions of 0.25 – 0.5 and 0.5 – 1.4 mm, the more intense was the air classification, the lower was the ashes content in the light fraction (Fig. 4). Regarding protein, the effect of air classification intensity was not significant, and it needs to be studied with a larger number of samples. Air classification was also found significant for influencing the content of both neutral and acid detergent fiber, lignin, and the water holding capacity of some sieved-classified fractions (Table 6).

The fractions with the lowest water holding capacity were those with predominance of seed coat fragments (242.6 % in the light fraction of particles 1.4 – 2.0 mm), and the highest water holding capacity was observed in the fractions with predominance of fruit husks (382.2 % in the light fraction of particles > 2.0 mm) or seed kernel (408.9 % in the light fraction of particles 0.25 – 0.5 mm).

Table 3
Content of macronutrients of nine dry fractions of castor meal separated by sieving and air classification and of the four inputs in the industrial process of castor oil extraction.

Sieve opening	Density	P	K	Ca	Mg	S
		g/kg				
Fractions separated by sieving and air classification						
> 2.0 mm	Light	6.0^{a}	14.2^{de}	5.7^{a}	3.1^{a}	2.5^{a}
	Heavy	9.3^{ab}	13.8^{cde}	6.6^{ab}	4.6^{ab}	3.3^{ab}
1.4 – 2.0 mm	Light	5.9^{a}	8.1^{a}	9.2^{d}	3.4^{a}	2.6^{a}
	Heavy	10.2^{abc}	10.4^{ab}	7.7^{bc}	5.4^{bc}	4.2^{bc}
0.5 – 1.4 mm	Light	11.6^{bc}	10.8^{abc}	8.6 ^{cd}	5.3^{bc}	4.1^{bc}
	Heavy	9.2^{ab}	8.8^{ab}	9.5^{d}	4.7^{ab}	3.6^{ab}
0.25 – 0.5 mm	Light	20.2^{d}	15.6^{e}	5.6^{a}	9.1^{d}	6.9^{d}
	Heavy	14.7^{c}	11.6^{bcd}	7.2^{bc}	6.6^{c}	5.3^{c}
< 0.25 mm	-	19.3^{d}	15.4^{e}	5.7^{a}	9.0^{d}	7.0^{d}
Inputs in the industrial process of castor oil extraction						
Fruit husk	-	1.62^{d}	30.2^{d}	2.9^{b}	1.6^{b}	1.5^{c}
Seed coat	-	0.72^{c}	5.0^{a}	8.2^{d}	1.2^{a}	0.8^{b}
Seed kernel	-	0.51^{b}	7.6^{b}	1.9^{a}	5.9^{c}	1.0^{b}
Clay	-	0.03^{a}	9.3^{c}	4.0^{c}	8.7^{d}	0.3^{a}

The means followed by different superscript letters in the column are significantly different (test of Tukey, $p < 0.05$). The means of the fractions were not compared with the means of the inputs.

Table 4
Content of micronutrients of nine dry fractions of castor meal separated by sieving and air classification and of the four inputs in the industrial process of castor oil extraction.

Sieve opening	Density	B	Cu	Zn	Fe	Mn
		mg/kg				
Fractions separated by sieving and air classification						
> 2.0 mm	Light	8.7^{a}	11.6^{a}	55.2^{a}	192.0^{a}	36.7^{a}
	Heavy	10.4^{ab}	15.6^{ab}	82.6^{abc}	164.5^{a}	57.4^{a}
1.4 – 2.0 mm	Light	10.1^{ab}	13.1a	62.7^{ab}	156.7^{a}	48.7^{a}
	Heavy	8.6^{a}	21.0^{ab}	109.5 ^{cd}	192.8^{a}	62.2^{ab}
0.5 – 1.4 mm	Light	10.4^{ab}	20.9^{ab}	123.2^{de}	183.5^{a}	58.5^{a}
	Heavy	10.3^{ab}	16.0^{ab}	88.3^{bc}	184.3^{a}	64.2^{ab}
0.25 – 0.5 mm	Light	10.4^{ab}	26.8^{b}	186.0 ^{g}	206.4^{a}	65.3^{ab}
	Heavy	10.8^{ab}	22.8^{ab}	151.1^{ef}	211.0^{a}	72.8^{ab}
< 0.25 mm	-	12.0^{b}	23.5^{ab}	161.1^{fg}	162.0^{a}	103.1^{b}
Inputs in the industrial process of castor oil extraction						
Fruit husk	-	5.7^{b}	4.1^{a}	20.6^{a}	137.5^{b}	13.8^{a}
Seed coat	-	5.8^{b}	4.5^{a}	16.9^{a}	44.6^{a}	21.7^{b}
Seed kernel	-	2.0^{a}	22.6^{b}	171.0^{c}	107.8^{ab}	18.7^{ab}
Clay	-	2.0^{a}	3.2^{a}	50.1^{b}	126.4^{b}	145.7^{c}

The means followed by different superscript letters in the column are significantly different (test of Tukey, $p < 0.05$). The means of the fractions were not compared with the means of the inputs.

3.2. Nutrients in the fractions and inputs

The composition of macronutrients was highly divergent among fractions and inputs of castor meal (Table 3). The phosphorus was found in higher content in the fractions with smaller particles, as 20.2 g/kg in the fraction 0.25 – 0.5 mm (light). Potassium content was high in the fruit husk (30.2 g/kg) and in clay (9.3 g/kg) and consequently in the fractions with predominance of these inputs, such as > 2.0 mm and < 0.5 mm. Seed coat was the input with the highest calcium content (8.2 g/kg), and this characteristic was reflected in the fractions with particles between 0.5 and 1.4 mm. Clay had a relevant content of magnesium (8.7 g/kg), followed by the seed kernel (5.9 g/kg), contrasting with the low content in the seed coat (1.2 g/kg). For that reason, Mg was the highest in the fine fractions. Sulfur content was the highest (7.0 g/kg) in the finest fraction and the lowest (2.5 g/kg) in the coarsest fraction (> 2.0 mm, light).

Boron content varied among the fractions between 8.6 g/kg (1.4 – 2.0 mm, heavy) and 12.0 g/kg (< 0.25 mm) (Table 4). The content of the micronutrients copper (22.6 g/kg) and zinc (171.0 g/kg) in the seed kernel presented relevant contrast with the low content found in the fruit husk (4.1 and 20.6 g/kg, respectively). In consequence, the highest Cu and Zn contents were found in the fractions with fine particles. The manganese content measured in the clay was about 8 times the average of this micronutrient among the three seed components. As a consequence, the fraction < 0.25 mm, which had the highest content of clay, confirmed the highest Mn content among the fractions.

Table 5
Estimation of the contribution of each input from the process of castor oil extraction into the chemical and physical characteristics of each fraction of castor meal.

Sieve opening	Density	Seed kernel	Seed coat	Fruit husk	Clay	R^2
> 2.0 mm	Light	−0.03	0.25	0.27	−0.04	0.875
	Heavy	−0.09	0.05	0.09	−0.03	0.529
1.4 – 2.0 mm	Light	−0.15	0.51	−0.09	−0.05	0.947
	Heavy	−0.06	0.07	−0.13	−0.04	0.422
0.5 – 1.4 mm	Light	−0.05	0.02	−0.13	−0.09	0.456
	Heavy	−0.14	0.28	−0.18	−0.06	0.808
0.25 – 0.5 mm	Light	0.03	−0.75	−0.11	−0.13	0.935
	Heavy	−0.03	−0.33	−0.18	−0.09	0.661
< 0.25 mm	-	−0.07	−0.72	−0.14	−0.08	0.909

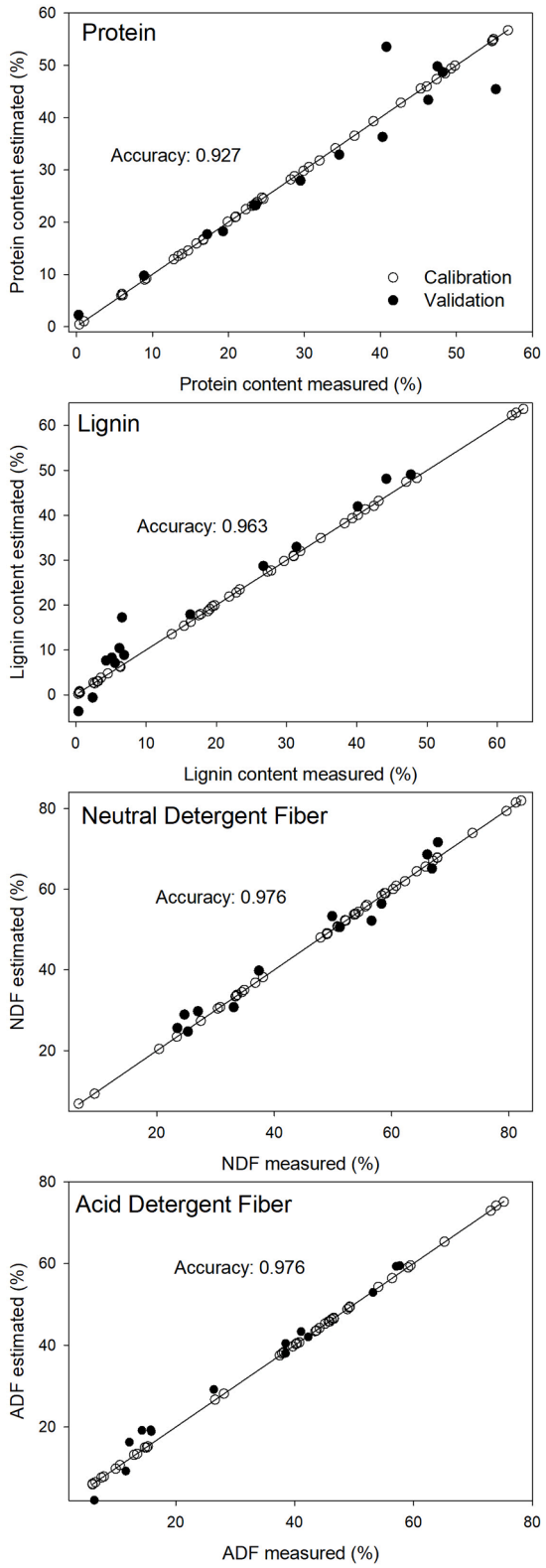

Fig. 5. Measured content of protein, lignin, neutral detergent fiber, and acid detergent fiber compared with the values estimated using NIR spectra for calibration (◯) and validation (●) of the model.

3.3. Fate of inputs among the dry fractions

The initial multiple regression analysis was made with 17 variables, and the five of them with the highest mean deviation were excluded (copper, iron, lignin, manganese, and phosphorus). The 12-variables multiple regression analysis was coherent to associate each dry fraction with a given input according to the chemical, physical, and nutritional characteristics. In the fraction > 2.0 mm, there was a predominance of fruit husks (light = 0.27, heavy = 0.09) and the lowest presence of seed kernel (light = −0.03, heavy = −0.09) (Table 5). The regression analysis corroborated that when the coarse fraction was air classified, the fragments of fruit husk were transported and concentrated in the light fraction. The predominance of fruit husks in the coarse fraction is also evident at visual inspection (Fig. 3). The characteristics of the seed coat were detected predominantly in the fraction 1.4 – 2.0 mm, and the air classification concentrated these fragments in the light particles (0.51) compared with the heavy fraction (0.07). The three fractions with particles smaller than 0.5 mm were strongly associated with the characteristics of the seed kernel (-0.07–0.03), while they were very divergent from the characteristics of fruit husk and seed coat (-0.72 to −0.14).

Clay was always a minor component in the fractions, and its influence was estimated with negative values in all the fractions (-0.13 to −0.03). The less negative association of clay characteristics occurred with the fraction > 2.0 mm (heavy particles = −0.03 and light particles = −0.04). A partial analysis suggests that the clay was concentrated in the fraction < 0.25 mm considering its significantly higher ashes content (15.0 %) and its reduced heat of combustion (3.92 kcal/g), which are characteristics strongly associated with the clay (Table 2).

3.4. Prediction of composition with NIR spectroscopy

The content of protein, lignin, and neutral and acid detergent fiber was predicted using NIR spectra with accuracies varying from 0.927 to 0.976 (Fig. 5). Although the models were calibrated with only 43 samples, this high accuracy was evidence that the main nutritional components of castor meal can be estimated using NIR Spectroscopy. A potential application of NIR is for real time monitoring of the dry fractionation process in the industry of castor oil extraction. Based on quick, inexpensive, and reliable measurements, the fractionation process can be continually adjusted aiming to optimize the separation of target components.

4. Discussion

4.1. Dry fractionation is efficient for castor meal

One feature of castor seed is that the morphological structure that contains the oil and the protein (seed kernel) is not bound to the fibrous parts (the seed coat and the fruit husk). For that reason, although the inputs are mixed along the industrial process of oil extraction, they can be easily separated afterwards. The dry fractionation of solvent-extracted castor meal is especially easier because of the low oil content. It is frequent the oil content reducing the dry fractionation efficiency in other oilseed meals (Arrutia et al., 2020; Hansen et al., 2017), and it is likely that the same would occur if the oily castor cake was subjected to fractionation.

The observation of the particles of each fraction in the microscope (Fig. 3) corroborates their chemical composition. The coarse fraction (> 2.0 mm) is predominantly composed of fragments of fruit husk, but there are some agglomerates that were not properly broken up in the milling operation because they were protected inside the pieces of husk. As the remaining agglomerates have the same mean composition of the castor meal before fractionation, they are the reason why the coarse fraction has a protein content greater than in the fruit husk. In the air classification, the agglomerates are heavier than the particles of fruit husk, and they remain in the heavy fraction, increasing its protein content (Table 2). The milling operation is crucial for the dry fractionation of all oilseed meals (Arrutia et al., 2020; Hansen et al., 2017; Sredanović et al., 2011; Vidosavljević et al., 2022), and it should be the

key step for improving the segregation efficiency of castor meal.

Seed coat fragments (black or brown color) are visually the most frequent component in the fraction 1.4 – 2.0 mm (Fig. 3), corroborating the estimation based on the physicochemical characteristics of the inputs (Table 5). Some agglomerates are also present in this fraction, and hypothetically, the fractionation could be further optimized if the agglomerates were completely broken up. It should be also noted that the fruit husk and the seed coat originate fragments with different sizes, and for that reason, they are easily segregated by sieving.

The fraction 0.5 – 1.4 mm also has predominance of seed coat fragments, with a few pieces of seed kernel. The picture illustrates how the white fragments of seed kernel are not bound to the brown fragments of seed coat (Fig. 3). This characteristic suggests that the process of fractionation can be optimized to segregate more efficiently those inputs. The two finest fractions (< 0.5 mm) are predominantly composed of seed kernel fragments with some occurrence of small seed coat pieces. The fractionation by sieving failed to separate those fragments, and air classification had a limited effect.

The enrichment reaching 49 % of crude protein by dry fractionation of castor meal compares to the concentration observed in the major oilseed meals (Arrutia et al., 2020; Sredanović et al., 2011). Unlike castor meal, the dry fractionation of rapeseed meal was effective for reducing fiber but not much in increasing its protein content (Hansen et al., 2017). In sunflower, the initial crude protein of 36 % was increased to 48 % with double milling operations (Vidosavljević et al., 2022). Electrostatic separation can be also considered for classification of the protein-rich particles, as demonstrated for rapeseed and sunflower meals that were milled to very fine particles (Laguna et al., 2018).

The particles with high content of fiber and lignin are fragments of fruit husk and seed coat, while the high-protein particles are fragments of the seed kernel. As those inputs are segregated, it is common to find a negative correlation between crude protein and fiber or lignin content among dry fractions of oilseed meals (Hansen et al., 2017; Laguna et al., 2018; Sredanović et al., 2011; Vidosavljević et al., 2022).

4.2. Different potential uses for each fraction of castor meal

Castor meal can be explored in the concept of biorefinery, in which each component can be isolated and characterized for its best use and value. The most obvious use for the protein-rich fraction of castor meal is to be an ingredient in ruminant ration. That is the natural option because there is large demand for this product, and that is the most common use for most of the protein obtained from oilseed meals worldwide (Arrutia et al., 2020; Diniz et al., 2011; Fernando, 2021; Hansen et al., 2017; Laguna et al., 2018; Vidosavljević et al., 2022).

As for the other fiber-rich fractions, they should be considered for other potential uses, which will depend on their specific chemical and physical characteristics. Burning the low-protein fraction for heat production is an interesting option, considering that the industry could replace its own demand for wood and other biomasses burned in boilers. Some key properties in a material to be burned are the heat of combustion and the ashes content (Bala-Litwiniak and Musial, 2024). The coarse fractions of castor meal (particles > 0.5 mm) have adequate calorific power, in the range of 4.11–4.37 kcal/g (Table 2), but their high ashes, potassium, and protein content may be a concern. The elevated ashes content in the coarse fractions is likely to be derived from contamination with clay, because they are predominantly composed of fruit husk and seed coat, which have lower ashes content. Nitrogen (protein) is unwanted in the fraction to be burnt because it generates toxic compounds (nitrogen oxides, NOx) in the exhaust gas (Bala-Litwiniak and Musial, 2024). In summary, the use of coarse fractions for burning will benefit from an optimized dry fractionation that remove most of the ashes and nitrogen-rich particles.

Organic fertilizer is the current main use of castor meal, and that continues as an interesting option for the fiber-rich fractions. Although these fractions have reduced nitrogen content, they are still rich in many other nutrients, including some residual nitrogen. A previous study demonstrated that the nitrogen found in the coarse fractions is readily available when applied as fertilizer (Severino et al., 2021).

4.3. Variability in the fraction's characteristics and monitoring with NIRS

A requirement for the optimization of industrial processes is stability on the key characteristics of the inputs. The raw material used for castor oil extraction varies in quality because castor seed is produced on a variety of farmers, environments, and production systems. Regarding the properties evaluated in this study, fruit husks are the most variable and influencing quality parameter in the raw material. Fruit husk does not contain any castor oil, and it has very low protein content; however, the husk increases the freight costs for being transported long distances, inflates the requirement for storing space as it has low density, and reduces the efficiency of oil extraction because it absorbs oil. The content of fruit husk depends on the cleaning operation that is performed on the farm, and it can be improved with careful seed cleaning operation. Hypothetically, the weight of the fraction with particles > 2.0 mm would approach zero if the seed used by the industry were free of fruit husks.

The dry fractions with particles between 0.4 and 1.4 mm are predominantly composed of seed coat, and they would be influenced by the seed composition. There is wide variability among castor varieties on the seed coat thickness and density, and in the weight of the seed coat to seed kernel (Severino et al., 2015, Severino, 2024). Thin castor seed coat is associated with characteristics desired by farmers (faster germination) and by the industry (higher oil content). This study demonstrated that reduced fiber and lignin content in castor meal is another reason for the search of castor varieties with thinner seed coat.

NIR Spectroscopy was confirmed to be effective to estimate the composition of castor meal. The most important components to be monitored were protein and fiber. It is expected that in the industrial process the variability on protein content is limited because this is a essential seed component. The highest variability is expected to occur in the fiber content because it depends on the seed quality, particularly on the contamination with fruit husk and on the content of empty seeds. This study did not investigate what adjustment in the industrial process are associated with the protein content. It just demonstrates that this characteristic can be monitored with NIR spectroscopy, and that dry fractionation methods are effective to separate those components.

4.4. Further studies on castor meal fractions

The dry fractionation of castor meal opens many new options on how this byproduct can be elaborated for other uses. The effective nutritional value of each fraction of castor meal should not consider only the crude protein, but it should be evaluated for protein digestibility (Mahesh et al., 2017). It is likely that the high acid detergent fiber and lignin found in the seed coat and in the fractions between 0.5 and 2.0 mm are associated with forms of protein with low digestibility. The amino acids profile should be characterized as an important requirement regarding protein quality. Although ruminants in general do not require a well-balanced amino acids profile, this property is relevant for high performance animals (Mahesh et al., 2017) or if the ingredient is considered for monogastric feed. Prior analysis made in the whole castor meal found low content of glycine, lysine, and methionine and extremely low on tryptophan (Annongu and Joseph, 2008; Igwe et al., 2012), and this profile needs to be measured specifically on the protein-rich fractions. Other anti-nutritional factors such as oxalates, phytates, tannins, phenolic compounds, and trypsin inhibitors should also be investigated (Arrutia et al., 2020; Enujiugha and Ayodele-Oni, 2003). A ricin concentration effect should also be checked, as the toxic protein from the seed kernel is concentrated in the finest fraction.

The methods employed to measure fiber content are not appropriate to analyze a non-organic material such as clay. Considering the broad

Table 6
Effect of the intensity of air classification (x) on the content of neutral detergent fiber, acid detergent fiber, lignin, heat of combustion, and water holding capacity (y), in the fraction with light particles, estimated by linear regression analysis.

Property	Regression equation	R^2
Fractions with particles > 2.0 mm		
Neutral detergent fiber (%)	$y = -9.3^{ns} x + 65.9$	0.043
Acid detergent fiber (%)	$y = 3.0^{ns} x + 45.4$	0.023
Lignin (%)	$y = 50.9^{*} x + 1.6$	0.710
Heat of combustion (kcal/g)	$y = 0.28^{ns} x + 4.20$	0.164
Water holding capacity (%)	$y = -0.50^{ns} x + 4.01$	0.056
Fractions with particles between 2.0 and 1.4 mm		
Neutral detergent fiber (%)	$y = -30.4^{ns} x + 80.3$	0.602
Acid detergent fiber (%)	$y = -43.3^{*} x + 75.8$	0.849
Lignin (%)	$y = -44.9^{*} x + 61.5$	0.879
Heat of combustion (kcal/g)	$y = -0.22^{ns} x + 4.61$	0.296
Water holding capacity (%)	$y = -0.73^{ns} x + 2.70$	0.199
Fractions with particles between 1.4 and 0.5 mm		
Neutral detergent fiber (%)	$y = 52.7^{*} x + 32.8$	0.665
Acid detergent fiber (%)	$y = 48.5^{ns} x + 24.6$	0.618
Lignin (%)	$y = 55.1^{*} x + 13.0$	0.704
Heat of combustion (kcal/g)	$y = 0.57^{ns} x + 4.16$	0.595
Water holding capacity (%)	$y = -3.9^{*} x + 4.4$	0.866
Fractions with particles between 0.5 and 0.25 mm		
Neutral detergent fiber (%)	$y = 38.5^{*} x + 8.1$	0.688
Acid detergent fiber (%)	$y = 31.7^{*} x - 0.16$	0.663
Lignin (%)	$y = 23.2^{ns} x - 4.7$	0.621
Heat of combustion (kcal/g)	$y = 0.33^{ns} x + 3.99$	0.600
Water holding capacity (%)	$y = -3.5^{ns} x + 5.6$	0.208

* The coefficients followed by * or ^{ns} are significant at $p < 0.1$ or non-significant, respectively.

definition from animal nutrition that fibers are a source of energy with restrictions to be digested, the fiber content of clay should be near 100 %, but it was measured instead as 7.9 % in neutral detergent and 6.6 % in acid detergent (Table 2). This misguided result occurs because the clay's particles are finer than the openings in the bag or filter used to measure the fiber (Van Soest et al., 1991), and the clay is washed by the detergent and accounted as a non-fiber material. As clay is present in the fractions of castor meal, further studies are needed to understand how the measurements of fiber content are influenced and what adjustments should be made on the interpretation of such results.(Table 6).

Organic fertilizer is the most important current use for castor meal, and the dry fractions continue to be considered for that purpose, especially if specific properties are identified in the fractions. The effect of the whole castor meal is well documented against parasite nematodes in plants and gastrointestinal in small ruminants (Galbieri et al., 2024; Meneses et al., 2022), and they should be further evaluated among the fractions. Properties such as the capacity to alleviate salt stress (Sousa et al., 2022) deserve further studies in the fractions to be used as organic fertilizer. Many bioproducts, secondary metabolites and extractives from fractions of castor meal can be explored for a variety of uses (Avramovic et al., 2024).

5. Conclusions

The process of dry fractionation of castor meal resulted in fractions highly divergent on protein, fiber, ashes and other chemical, physical, and nutritional characteristics. Air classification promoted an additional segregation to the prior fractionation by sieving. The fragments of fruit husks were concentrated in the coarse fraction (particles > 2.0 mm), the fragments of seed coat were predominant among the particles between 0.5 and 2.0 mm, and the fragments of the seed kernel were separated in the finest fraction (particles < 0.5 mm). Near Infrared Spectroscopy was very efficient to estimate the content of protein, lignin, and fibers in the dry fractions of castor meal.

CRediT authorship contribution statement

Liv S. Severino: Writing – review & editing, Writing – original draft, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Fabio P. Silva:** Methodology, Formal analysis, Data curation. **Maria E.C. Esquibel:** Investigation, Formal analysis. **Andressa S. Rocha:** Methodology, Investigation.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Liv Soares Severino reports financial support and equipment, drugs, or supplies were provided by Azevedo Óleos Vegetais Ltda. The corresponding author was Associate Editor of this journal. This relation was terminated one year before submission of this manuscript. If there are other authors, they 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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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.indcrop.2025.120831.

Data availability

The raw data is publically available in a repository.

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