



# Fiber Rich Co-Products from Carioca Bean Protein Fractionation: Characterization and Ball Milling Treatment

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## Abstract

This work aimed to explore the potential uses of co-products from the concentration of carioca beans protein and to apply ball mill treatment to produce high-viscosity suspensions. Two co-products were evaluated: hulls obtained by industrial dry fractionation process (HDF) (49.8% of fiber) and fibrous biomass from wet fractionation (FBWF) (33.9% of fibers). The drying of FBWF reduced the moisture content from  $84.8 \pm 0.4\%$  to  $11.8 \pm 0.1\%$  (5 h/60°C), and the drying curve well-adjusted to Logarithmic model. Ball mill treatment was performed at 400 rpm for 6 h at 25 °C using zirconium spheres. In both co-products, insoluble fibers were predominant, and among them, the HDF sample showed a higher amount of soluble fibers. The longer the milling time, the greater the increase in viscosity and the reduction in particle size of the suspension. FBWF exhibited stable viscosity during heating, whereas HDF viscosity decreased as it was heated. In both fractions, the treatment promotes changes in its interactions with water, due to starch damage in the FBWF and fibers size decrease in the HDF. Therefore, co-products studied in this work can be used in the food industry as a source of fiber and, when processed in a ball mill, as a thickening agent.

**Keywords** Waste recovery · Hulls · Protein extraction residues · Dietary fiber

## Introduction

Beans are a widely consumed staple food across the globe, particularly in tropical and developing countries, offering a rich source of energy, nutrients, and dietary fiber [1].

Carioca bean is a pulse widely cultivated and consumed in Brazil, being considered a traditional food for the Brazilian population and of great economic, social and nutritional importance [2]. Protein concentrates from carioca beans offer potential for the food industry because of their low cost and promising techno-functional properties, particularly in foaming and emulsifying capacity [3]. To extract proteins from various pulses, wet fractionation is a commonly used method. This process involves solubilizing the protein at an alkaline pH and then precipitating it at an acidic pH. However, this approach generates solid residues, including hulls and insoluble fractions that are rich in fiber and starch [4]. Another emerging technique is dry fractionation, that involves milling to separate starch granules from smaller protein-rich particles, and air classification to separate one fraction from another based on differences in density and size [5]. These method produce residues, such as hulls and a starch-rich fraction [6].

With growing consumer awareness of health and environmental concerns, food manufacturers are integrating sustainable ingredients, such as dietary fibers derived from

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agricultural waste [7]. The co-products of carioca bean protein extraction are rich in fiber and phenolic compounds that can be used to enrich food products. In general, common beans are an excellent source of dietary fiber, containing two to three times more fiber per 100 g of edible portion compared to other staple foods [8]. Besides, the presence of (poly)phenols, including flavonoids, phenolic acids, and tannins play a crucial role in supporting health [9]. Dietary fiber, composed of non-digestible plant polysaccharides, promotes gut health by softening stool, shortening intestinal transit through insoluble fibers, and supporting bacterial biomass and short-chain fatty acid production via soluble fiber fermentation [1, 10]. The non-extractable polyphenols, which are bound to the plant cell wall matrix can provide prolonged bioactivity compared to extractable polyphenols. These conjugates can enhance the nutritional value of food products and be tailored for consumers at high risk of health issues, such as type II diabetes, obesity, or cardiovascular disease, as well as for personalized nutrition strategies [11]. These compounds are found mainly in the hulls, a co-product of protein extraction, that represents from 7 to 13 g/100 g bean seed weight [12].

Despite the great potential to health, insoluble fiber rich residues application in food can be challenging due to their rough texture, limited solubility, and potential impact on sensory properties [13, 14]. To solve this problem, defibrillation techniques, such as milling, can be used to obtain suspensions with a gel like behavior [15] and increased swelling, water holding capacity and emulsifying activity [16, 17]. Besides, this approach can enhance their palatability and preserves functional compounds naturally found associated to fiber fractions [15, 18–20]. Therefore, the objective of this work was to investigate and add value to the by-products generated during the isolation of bean proteins, focusing on the hulls and the solid residue obtained after protein solubilization at alkaline pH. The drying kinetics of the fiber-rich fraction were evaluated due to its high moisture content, which makes drying a necessary step for food applications. Additionally, the composition of both fractions was determined to support the identification of potential uses. To broaden their applicability, ball milling was employed to produce high-viscosity suspensions, enhancing their potential use in food products as thickening agents, while also improving palatability and stability through particle size reduction.

## Materials and Methods

### Materials

Carioca beans obtained from local market (Campinas, Brazil) were used to obtain the fiber-rich fraction resulting from protein extraction. A 1:10 m/v suspension of the broken bean flour with distilled water was prepared. The suspension had its pH adjusted to 10 with the addition of 40% NaOH and was stirred for 60 min on a magnetic stirrer (IKA C-MAG HS 7). The suspension was centrifuged at 5000 rpm for 15 min in order to separate the solid residue. After centrifugation, the supernatant was used for protein concentration and the bottom body was collected and filtered through an 80-mesh sieve (ABNT 80, 180 µm opening). Therefore, filtration allowed the separation of the starch-rich fraction and the fiber-rich fraction retained on the sieve. The fiber-rich residue was dried in an oven for 5 h at 60 °C, crushed and sieved through a 20-mesh sieve. The hulls were obtained by an industrial process.

### Drying of the Fiber-Rich Fraction

The fiber-rich fraction was dried in a forced-air oven with air circulation and renewal at a speed of 1.7 m/s for 5 h at 60 °C (Marconi, Piracicaba–Brazil). The fiber-rich fraction was placed in a container so as to present a thinner film of approximately 3 mm. Around 130 g of sample were inserted in each drying container. The mass of the material during drying was recorded at different times to construct the drying curve. The dimensionless moisture content (MR) and the drying rate (R) were calculated using the Eqs. 1 and 2, respectively [21].

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (1)$$

$$R = \frac{M_{t_1} - M_{t_2}}{t_1 - t_2} \quad (1)$$

where,  $M_t$  is the moisture content at time  $t$  during the drying process,  $M_0$  is the initial and  $M_e$  is the equilibrium moisture content,  $t_1$  and  $t_2$  are the drying times in minutes,  $M_{t_1}$  and  $M_{t_2}$  correspond to the moisture contents at time  $t_1$  and  $t_2$ .

The models simple exponential, Page, Henderson and Pabis, logarithm, two-term exponential and Wang and Singh were adjusted to the curve as described by Menezes et al. [22]. To obtain the drying kinetic parameters, the Levenberg–Marquardt algorithm was used by the Statistica software version 14.0 (Tibco, Palo Alto, USA) with a confidence level of 95%.

## Composition Determination

The moisture analysis was performed based on the methodology provided by AACC International Method 44-15.02. 2 to 5 g of sample was weighed in previously tared crucibles. The samples were dried in an oven (Marconi, Piracicaba–Brazil) for 5 h at 105 °C. For total fiber, the enzymatic-gravimetric method of the 19th edition of the AOAC (985.29) was used, with some adaptations. The method consists of gelatinization and hydrolysis of starch with alpha-amylase, followed by hydrolysis of proteins using protease and hydrolysis of residual starch with amyloglucosidase. Total dietary fiber was precipitated with 95% ethanol and the residue was filtered, washed with solvents, dried and weighed. Finally, a correction of the total dietary fiber value was made by subtracting the blank and the protein and ash contents of the residues. For the quantification of insoluble and soluble fibers, AOAC method 991.43 was used, performed in a similar manner to that described previously. The fat by acid hydrolysis and ash contents were determined according to the methodologies described by IAL [23] and Latimer Jr [24], respectively. Extractions of phenolic compounds were performed using 70% acetone 1:15 in relation to the sample mass according to literature data [25]. The quantification of the total phenolic compound content was performed according to a methodology described by Singleton et al. [26] using a gallic acid standard curve and the Folin-Ciocalteu reagent.

## Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR)

The samples were analyzed using scanning electron microscopy (SEM) in a Tescan Vega LMU system (Tescan, Brno, Czech Republic). Micrographs were captured using back-scattered electron (BSE) detectors under a working distance of 15 mm and an accelerating voltage of 20 kV. The specimens were mounted on aluminum stubs with carbon adhesive and coated with a gold layer using a Balzers SCD 050 sputter coater (Balzers, Liechtenstein) at 40 mA for 40 s. FTIR analysis was performed using a Perkin Elmer 100 spectrophotometer (PerkinElmer, Waltham, USA) equipped with an attenuated total reflectance (ATR) accessory to obtain spectra in the wavelength range of 4000 to 650  $\text{cm}^{-1}$ . For each sample, 32 spectra were collected per replicate, with analyses performed in triplicate.

## Ball Milling Treatment

A stirred ball mill (PE 5, Netzsch, Pomerode, Brazil) with yttrium-stabilized zirconium oxide spheres (ZetaBeads® 3.0, Netzsch) was used. After pre-tests, a quantity of 7.680 kg of

spheres, 150 g of FBWF or 75 g of HDF and 1.850 L of water were defined for 6 h at a constant rotation of 400 rpm. The temperature was controlled by a thermostat bath with water circulation (ECO Gold Lauda DR.R. Wobser GMBH and Co. KG, Lauda-Königshofen, Germany) at 25 °C. Aliquots were removed every hour for apparent viscosity analysis in a Brookfield rotational viscometer model DV-III (AMETEK, Middleboro, USA) coupled to a circulating water bath (TC 500, AMETEK Brookfield, Middleboro, USA). Spindle 15 was used and the measurements were done with the shear rate varying from 4.8  $\text{s}^{-1}$  to 48  $\text{s}^{-1}$  at 25 °C. The resulting data were adjusted to the Ostwald-de-Waele model [7] using Statistica 14.0 (Tibco, Palo Alto, USA). The particle size of samples was analysed in by laser diffraction (L950, Horiba Instruments, Inc., Kyoto, Japan). The effect of the temperature on the viscosity of fiber co-products threatened by ball milling was performed using a RVA (Rapid Visco Analyser RVA 4500, Perten Instruments, Hägersten, Sweden). It was used the “Standard 2” program and samples solid content was 7.5% (w/w) for both fractions.

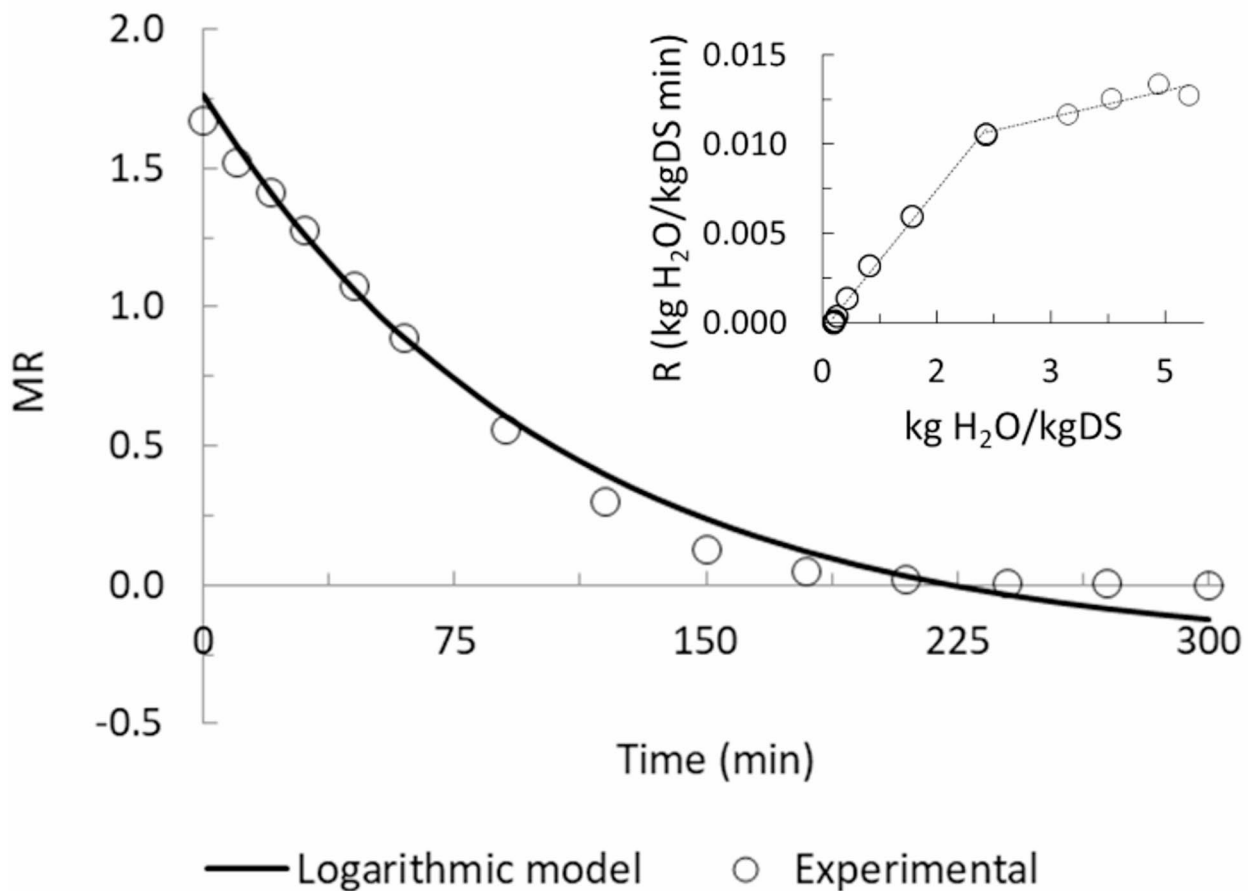
## Statistical Analysis

The measurement results are expressed as the mean  $\pm$  standard deviation. All results were tested for normality using the Shapiro-Wilk test. Homogeneity of variance for groups with more than two samples was evaluated using Levene's test. A t-test or Mann-Whitney test was applied to assess significant differences between the samples at a 95% confidence level, depending on whether the distribution was normal or non-normal, respectively. To evaluate differences in particle size parameters, the Kruskal-Wallis test was applied, followed by the Dunn post-hoc test ( $\alpha=0.05$ ), due to the heterogeneous variance of the results. All statistical analyses were conducted using Microsoft Excel 2016 (Microsoft Corporation, Redmond, USA) and Real Statistics Resource Pack [27].

## Results and Discussion

### Drying Kinetics of Fiber-Rich Residue

To use the fiber-rich residue as an ingredient, it must be dried to reduce moisture and prevent microbial growth. This process extends shelf life while enhancing stability, handling, and suitability for various food formulations [21, 28, 29]. Figure 1 shows the drying curve of the fibrous biomass obtained after wet fractionation (FBWF) of carioca bean protein. The initial moisture content of the wet FBWF was  $84.8 \pm 0.4\%$  and the final moisture was  $11.8 \pm 0.1\%$ . Through the drying curve, it can be determined a 4 h to reach drying



**Fig. 1** Dimensionless drying curve of fibrous biomass obtained after wet fractionation of carioca bean protein and adjustment of the logarithmic model. Where MR is the dimensionless moisture content, R is the drying rate and DS is dry solid

equilibrium at 60 °C, characterized as rapid drying, typical of materials with abundant free water. The yield obtained for the dried FBWF was 17 g for every 100 g of flour.

The drying curve obtained is characteristic of the thin-layer drying process, which includes a constant rate period, where surface water evaporates steadily, and a falling rate period [28]. In this work, the first stage had a duration of around 90 min. This behavior, also reported for almond bagasse, involves an initial stage driven by product heating, followed by a second stage dominated by internal water diffusion, influenced by structural characteristics such as porosity, particle size, and compartmentalization [30].

The data obtained through dimensionless moisture were adjusted to different mathematical models to describe the drying kinetics. Predicting and optimizing thin-layer drying models, where an  $R^2$  closer to 1 indicates a more appropriate model for explaining the data, is key to scaling experimental results to industrial operations [21]. Table 1 presents a comparison of different models and their parameters. The Logarithmic showed the best fit ( $R^2=0.9953$ ), indicating it

best accurately describes the drying behavior. The Henderson and Pabis and Two-term exponential models, both with an  $R^2$  of 0.9894, also adjusted well to the experimental data. The Simple Exponential and the Wang and Singh model showed lower accuracy ( $R^2<0.85$ ). The logarithm model is a modification of the Henderson and Pabis model, have been used to predicts drying kinetics for various fruits and vegetables, including apple and pumpkin [29, 31].

### Fibrous Biomass and Hulls Composition

Table 2 presents the results of the moisture and fiber content of the samples obtained from the carioca bean protein extraction, i.e. fibrous biomass from wet fractionation (FBWF) and hulls from dry fractionation (HDF). Both fractions are mainly composed of carbohydrates (>69%), primarily in the form of dietary fiber. The remaining carbohydrate content originates from the bean cotyledon, such as starch. In the case of FBWF, the starch content is likely higher, as this material is insoluble in water, and larger starch granules may

**Table 1** Comparison of models and parameters obtained by adjusting mathematical models for the drying kinetics of the fiber-rich residue obtained after wet extraction

Model*	Equation	$R^2$	Constants*
Simple exponential	$MR = e^{-kt}$	0.8118	$k=0.007$
Page	$MR = e^{-kt^v}$	0.8848	$k=0$ ; $v=3.804$
Henderson and Pabis	$MR = A e^{-kt}$	0.9894	$A=1.816$ ; $k=0.013$
Logarithm	$MR = A e^{-kt} + c$	0.9953	$B=2.000$ ; $k=0.010$ ; $c=-0.235$
Two-term Exponential	$MR = C e^{-k_0 t} + D e^{-k_1 t}$	0.9894	$C=0.880$ $k_0=0.013$ ; $D=0.937$ ; $k_1=0.013$
Wang and Singh	$MR = 1 + E_t + F t^2$	0.8328	$E=-0.004$ ; $F=-3 \times 10^{-6}$

\*Models described by Menezes et al. (2013).  $k$ ,  $v$ ,  $A$ ,  $B$ ,  $C$ ,  $D$ ,  $E$ ,  $F$ ,  $k_0$  and  $k_1$  are model constants;  $t$  is the drying time (min);  $MR$  is the dimensionless moisture content

**Table 2** Moisture, protein, fiber and total phenolic compounds (TPC) of co-products from wet and dry fractionation

Parameter	Fibrous biomass– wet fractionation (FBWF)	Hulls– dry fractionation (HDF)
Moisture (%)	$11.85 \pm 0.12^a$	$10.10 \pm 0.71^a$
Protein (%)	$10.66 \pm 0.28^b$	$14.52 \pm 0.14^a$
Fat (%)	$1.39 \pm 0.00^b$	$1.88 \pm 0.03^a$
Ash (%)	$2.92 \pm 0.05^b$	$4.09 \pm 0.11^a$
Total carbohydrates (%)	$73.18 \pm 0.31$	$69.41 \pm 0.73$
Total dietary fibers (%)	$33.88 \pm 0.0^a$	$49.77 \pm 0.0^a$
Insoluble fibers (%)	$32.71 \pm 0.0^a$	$48.2 \pm 0.0^a$
Soluble fibers (%)	$1.17 \pm 0.4^b$	$1.60 \pm 0.0^a$
Total phenolic compounds (mg/g)	$0.34 \pm 0.01^b$	$42.46 \pm 2.09^a$

Note: samples with the same letter in a row do not differ significantly ( $p > 0.05$ ) according to the t-test (normal distribution according to Shapiro-Wilk test: moisture, protein, soluble fibers, fat, ash and total phenolic compounds) or Mann-Whitney (not normal distribution according to Shapiro-Wilk test: total dietary fibers and insoluble fibers). The carbohydrate content of the sample was calculated by difference

be retained on the sieve during its collection. Total dietary fiber (TDF) content of protein extraction residue is 33.88% what is smaller than result found for common black beans protein extraction residue (42%) due to different grain composition and extraction procedure [4]. The TDF of carioca beans whole flour vary from 18.89 to 23.72% [32, 33] and of hulls is around 71% [1]. These results show that during the protein extraction process, fibers are concentrated in both FBWF and HDF fractions. The majority of fibers were insoluble (~97%), that is in accordance with hulls composition. Carioca beans consist of approximately 89% cotyledons, 1.5% epicotyls, and 10% hulls [1]. As a result of the industrial processing method, which involves abrasion peeling, the hulls obtained contained remnants of cotyledons.

Legume hulls are predominantly composed by cellulose and hemicelluloses and cotyledons by pectic substances and soluble fibers [34]. Besides, the ash content is higher in the hulls [1]. Therefore, whole beans show a percentage of soluble fiber higher than hulls, i.e. 15% and 6%, respectively [1, 33]. In the fibrous residue, occurs an insoluble fibers

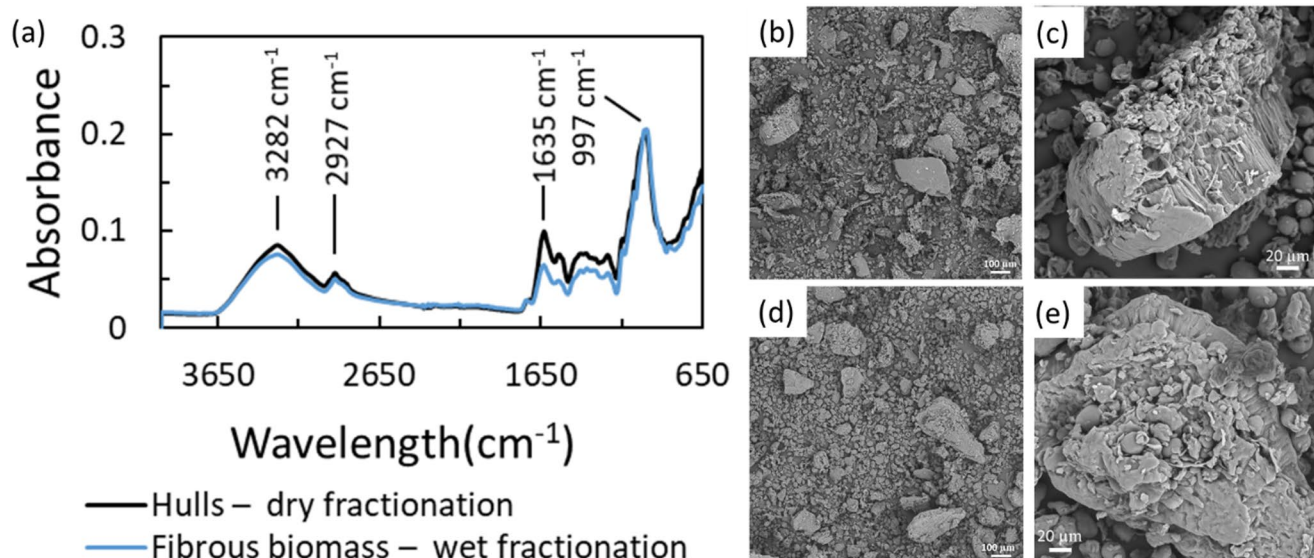
concentration due to the solubilization of soluble fibers together with the protein extract. The distribution of fibers in the residue is similar to those presented by Sandoval-Peraza et al. [35] for black bean fibrous residue obtained by pH 11 extraction.

The TPC of HDF ( $42.46 \pm 2.09$ ) were higher than those observed for FBWF  $0.34 \pm 0.01$ . Whole carioca beans present 2.82 mg/g of TPC for the same extraction method employed in this work [25]. However, most of the phenolic compounds present in beans are found in the seed coat [36]. Therefore, smaller value found for fibrous residue is due to the co-extraction of phenolic compounds during protein concentration and the presence of other compounds in this bean fraction. Whole carioca beans are composed mainly by conjugated phenolic compounds (64%) followed by bound (26%) and free phenolic compounds (10%) [37]. Conjugated and free phenolics are extracted alongside proteins due to their solubility; some free phenolics can dissolve in water, while conjugated phenolics can be released during alkaline treatment [38]. Additionally, phenolics have an affinity for proteins and may bind to them, further facilitating their co-extraction [39].

Fig. 2 shows the FTIR spectra and the microscopic images obtained by MEV for FBWE and HDF. Both FBWF and HDF presented similar spectra with peaks at 3282, 2927, 1635, and 997  $\text{cm}^{-1}$ . These peaks are attributed to the following: the stretching vibrations of O–H and N–H groups; the asymmetric stretching vibrations of C–H in  $\text{CH}_2$  groups; the amide I band; and carbohydrates C–OH stretching vibrations, respectively [40]. The plant cell wall consists of complex polysaccharides, phenolic compounds, and proteins, held together by ionic and covalent bonds [1]. Therefore, the both peaks observed at 3282, 2927 and 997  $\text{cm}^{-1}$  are associated to carbohydrates while the peak at 1635  $\text{cm}^{-1}$  is related to proteins.

The SEM images reveal the characteristic structure of the bean coat, consisting of a layer of macrosclereid cells, osteosclereid cells, and parenchyma tissue [41]. Additionally, remnants of internal grain components, such as starch and proteins, are also visible. This can be attributed to the





**Fig. 2** FTIR (a) and SEM of hulls (b, c) and fibrous biomass (d, e) with the magnification of 200x (b, d) and 1000x (c, e)

hulling process to obtain HDF, which involved abrasion, removing not only the hull but also portions of the grain's interior. For FBWE, the presence of insoluble components from the entire grain is evident, as the protein extraction process utilized whole flour.

### Ball Milling

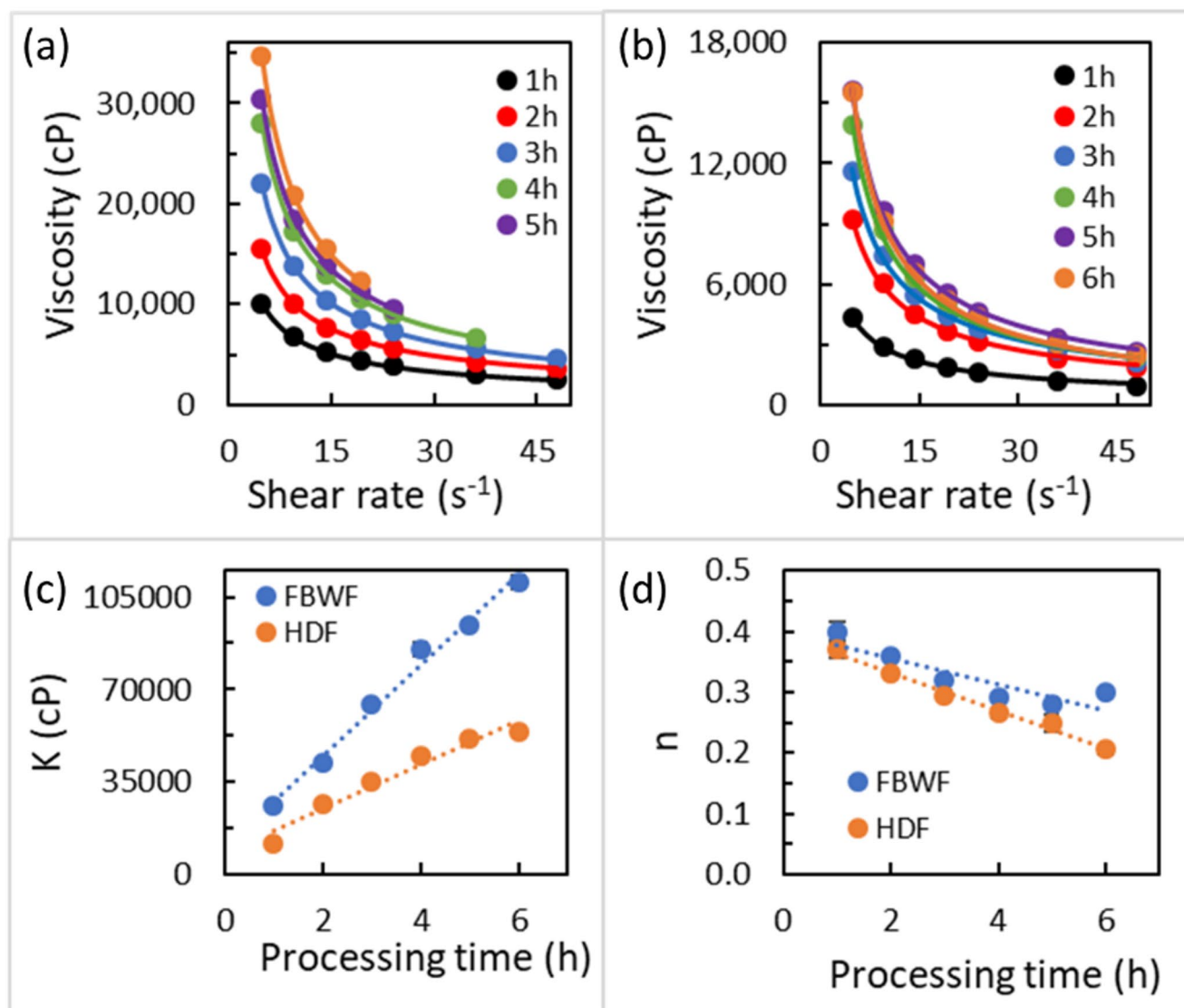
The fiber-rich co-products were processed for 6 h in a ball mill, where the process was monitored by means of samples of the material taken every 1 h. Figure 3 shows the results of the effect of ball milling on suspension apparent viscosity in function of shear rate and processing time. Results showed a decrease in the viscosity with the increase in shear, demonstrating the shear-thinning behavior of the dietary fibers in the aqueous solution as was also observed for other fiber rich suspensions [7]. As expected, with the increase in the milling time, the suspension viscosity rise. Apparent viscosity in function of shear rate curves were adjusted to Ostwald-de-Waele model where  $K$  is the consistency index (cP) and  $n$  is the flow behavior index [7] presented in Fig. 3c and d. The  $R^2$  obtained varied from 0.999999 to 0.998351 showing a good adjustment to the data.

The  $K$  constant obtained increased linearly with the processing time as presented in Table 3, showing that suspension became more consistent with the ball milling (Fig. 3c). This behavior is related to the decrease in particle size and defibrillation of the lignocellulosic complex due to the shear forces applied during the treatment in a ball mill [42]. As a result, hydroxyl groups of the cellulose molecules are exposed, which tend to come closer together, forming a network with a greater capacity to stabilize water molecules producing a gel-like behavior [43]. This can be an advantage

to the incorporation of these materials as thickening agent in foods. Besides, viscous fibers, a property commonly associated to soluble fibers, are linked to changes in blood glucose and cholesterol levels, delayed gastric emptying, and a slower transit time through the small intestine [44].

The particle size distribution of FBWF and HDF are presented in Table 3; Fig. 4. Both co-products exhibited multiple peaks, reflecting the presence of diverse compounds, as discussed in Sect. 3.2. Following ball milling, a significant reduction in particle size was observed, with median sizes ( $D_{50}$ ) decreasing from 375.2 and 1,012.1  $\mu\text{m}$  to 70 and 26  $\mu\text{m}$  for FBWF and HDF, respectively. This reduction is attributed to the friction, collision, and shear forces generated by the interaction between the milling balls and the samples [42]. The particle size distribution curves reveal a bimodal pattern for FBWF and a unimodal pattern for HDF treated by ball mill for 6 h showing a uniform particle distribution for processed hulls. For FBWF, it was feasible to process a suspension containing 15% solids without significant material adhesion to the mill walls, maintaining grinding efficiency. In contrast, when processing HDF at the same solid concentration, the material adhered to the walls, impairing the grinding. Consequently, the HDF suspension was processed with a reduced solid concentration of 7.5% to ensure efficient grinding. Notably, despite the smaller particle size of HDF, its final viscosity was lower compared to FBWF. This is likely due to the greater presence of starch and other compounds in FBWF, which contribute to its viscosity and enable the processing using higher solid content Fig. 5.

Figure 5 illustrates the impact of heating and cooling on the viscosity of samples treated in a ball mill for 6 h, with a solid concentration of 7.5% for both fractions. The viscosity

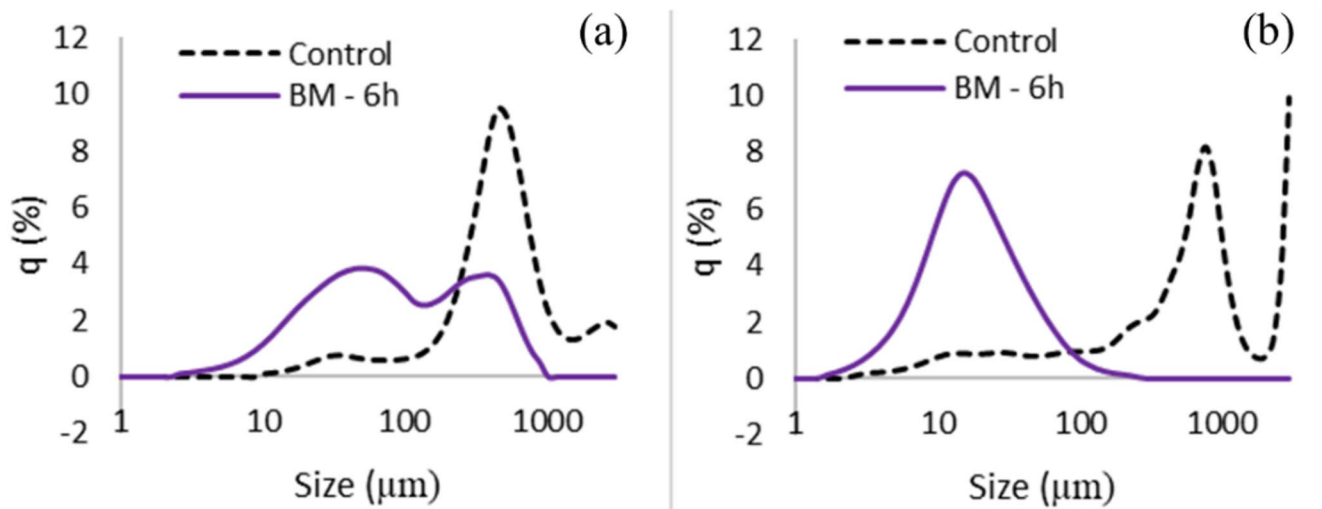


**Fig. 3** Viscosity fibrous biomass FBWF (a) and hulls HDF (b) and power law constants K (c) and n (d) in function of ball milling processing time

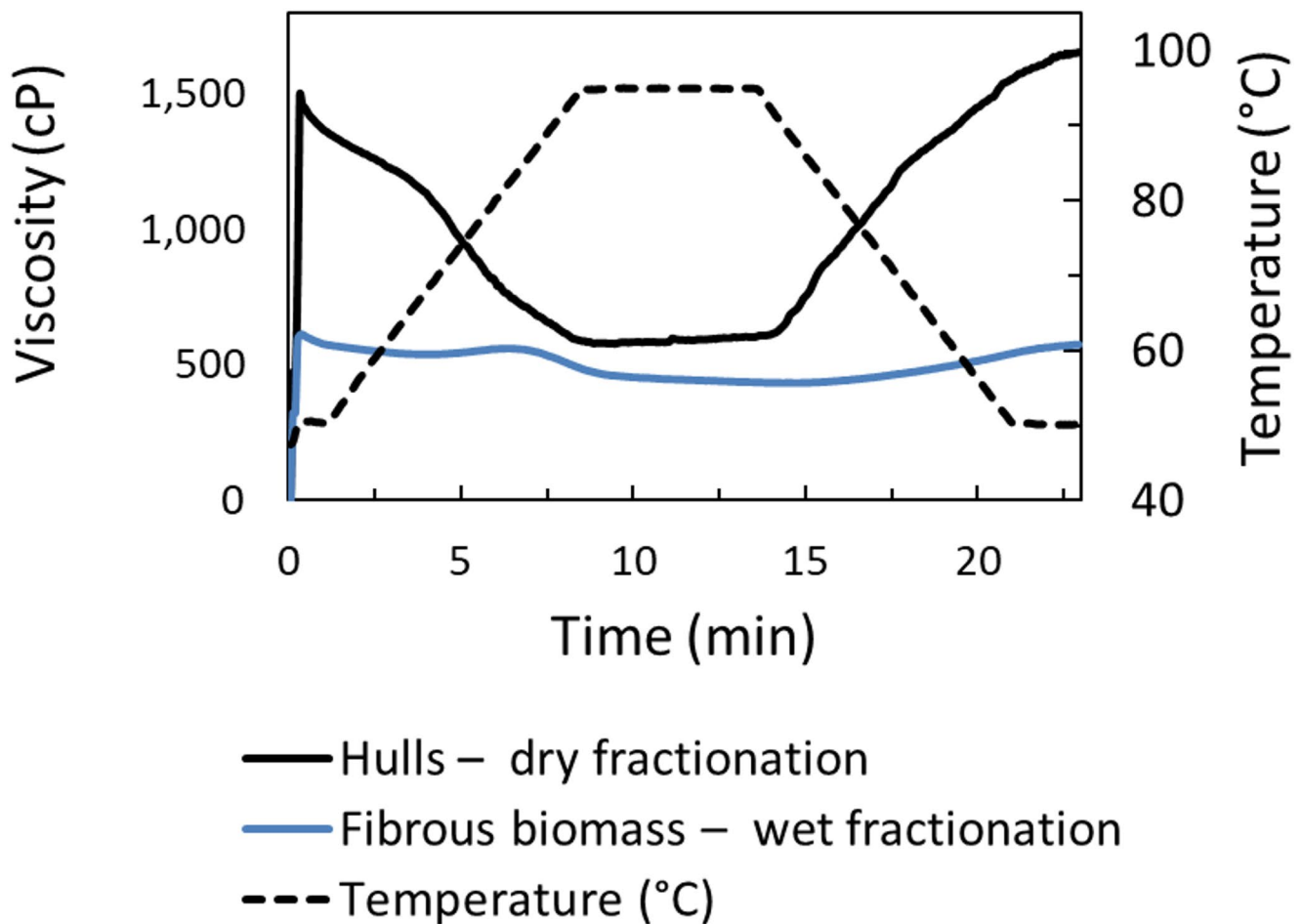
**Table 3** – Ostwald-de-Waele constant K in function of milling time (t) and particle size distribution parameters of fibrous biomass and hulls processed by ball mill

Parameter	Fibrous biomass– wet fractionation (FBWF)		Hulls– dry fractionation (HDF)	
<i>K in function of processing time</i>				
Equation	$K = 17,144t + 10,381$		$K = 8,403t + 7,916$	
R <sup>2</sup>	0.98788		0.95409	
<i>Particle size</i>				
	Control	BM – 6 h	Control	BM – 6 h
D10	80.0±9.9 <sup>a</sup>	14.5±0.2 <sup>ab</sup>	501.1±71.2 <sup>a</sup>	10.5±0.1 <sup>b</sup>
D50	375.2±15.3 <sup>a</sup>	70.6±2.9 <sup>ab</sup>	1,012.1±86.4 <sup>ab</sup>	26.3±0.5 <sup>ab</sup>
D90	696.4±33.1 <sup>a</sup>	379.3±24.6 <sup>ab</sup>	1,744.6±203.8 <sup>ab</sup>	75.1±4.2 <sup>ab</sup>
Span	1.6±0.0 <sup>b</sup>	5.2±0.3 <sup>a</sup>	1.2±0.1 <sup>b</sup>	2.5±0.1 <sup>ab</sup>

Samples presented heterogeneous variance according to Kruskal-Wallis test, therefore Kruskal-Wallis was applied. The same letter in a line do not differ significantly according to Dunn post-hoc test ( $p > 0.05$ )



**Fig. 4** – Fibrous biomass (a) and hulls (b) particle size before and after 6 h of ball mill treatment



**Fig. 5** – Rapid visco analysis (RVA) curves of fibrous co-products treated in ball mill for 6 h

of the FBWF suspension remained nearly stable throughout the analysis, indicating minimal temperature dependence. In typical starch viscosity profile, heating increases viscosity due to gelatinization, which enhances structure and

mouthfeel, while the rise in viscosity during cooling reflects starch retrogradation as it regains its original structure [45]. Milling disrupts starch granules through shear and compressive forces, altering their morphology and functional



properties [46]. The minimal temperature effect on the FBWF suspension is likely due to the irreversible swelling of starch and fiber during milling, as the granules' interior had already been exposed to water and was unaffected by further heating.

In general, a reduction in pasting properties (peak and final viscosity) occurs in multicomponent systems, as in this study (fiber-starch-protein mixture) compared to starch-water dispersions [47]. As for the influence of the grinding process, such as abrasive and ball grinding, these modify the crystallinity and, consequently, alter the water absorption, thermal parameters and rheological behavior of the fraction enriched with starch [48, 49], as well as the other components.

The HDF resulted in a suspension with temperature-dependent viscosity, with higher viscosities at smaller temperatures. This can be attributed to the different composition of both fractions, since the wet extraction residue contains starch and the dry extraction residue contains mainly fibers. This behavior may be interesting for fat substitutes since it is characteristic of a melting behavior of fat crystals with temperature. In both fractions, the treatment probably promotes changes in their interactions with water, due to damaged starch and a decrease in fiber size. The differences in RVA curve for both samples are due to distinct composition since hulls are composed mainly by fibers and residue of wet extraction by other compounds such as starch.

## Conclusion

This study explored the valorization of fibrous co-products generated during the protein concentration process of carioca beans, aiming to enhance their potential applications and contribute to sustainable food production. The fibrous biomass from wet fractionation (FBWF) drying process followed typical thin-layer kinetics, and the logarithmic model provided the best fit for describing the behavior. The fibrous co-products, FBWF and HDF, exhibited distinct compositions, with HDF containing higher levels of protein (14.52%) and fiber (49.77%) compared to FBWF, which had 10.66% protein and 33.88% fiber. Additionally, the HDF exhibited higher total phenolic content, attributed to the natural abundance of phenolics in the hulls and their co-extraction during protein concentration, which reduced their presence in the FBWF. Ball mill processing effectively reduced particle size and enhanced viscosity of both fiber rich co-products. Differently from FBWF, the HDF showed a decrease in viscosity during heating showing a melting like behavior. These findings suggest that co-products from bean processing, when adequately treated, can be repurposed as fiber-rich additives and thickening agents.

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**Author Contributions** Elaine Kaspchak: Conceptualization; Data curation; Formal analysis, original draft writing; Supervision. Leonardo Petkevicius Augusto: Formal Analysis, Investigation; Visualization. Ana Maria Barbosa dos Santosb: Formal Analysis, Investigation; Visualization. Clara Takayama Arbach: Formal Analysis, Investigation. Paula Fernanda Janetti Bócoli: Formal Analysis, Investigation. Elizabeth Harumi Nabeshima: Investigation; Methodology; Visualization; Writing - review & editing. Maria Teresa Bertoldo Pacheco: Funding acquisition; Resources. Mitie Sonia Sadahira: Project administration; Resources; Supervision, Writing - review & editing.

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**Data Availability** No datasets were generated or analysed during the current study.

## Declarations

**Competing Interests** The authors declare no competing interests.

## References

1. T.M. Shiga, B.R. Cordenunsi, F.M. Lajolo, *Carbohydr. Polym.* **83**, 362 (2011)
  2. A.C. Fernandes, B.E. Dutra, R.P. Proença, *Nutr. Em Pauta.* **85**, 68 (2007)
  3. L. de Paiva, R. Gouvêa, T. Caldeira, de M.C. Lima Azevedo, I. Galdeano, J.R. Felberg, Lima, and C. Grassi Mellinger, *Food Hydrocoll.* **137**, 108351 (2023)
  4. J.C. Ruiz-Ruiz, G. Dávila-Ortiz, L.A. Chel-Guerrero, and D. A. Betancur-Ancona, *Food Bioprocess Technol.* **5**, 1531 (2012)
  5. P.J.M. Pelgrom, R.M. Boom, M.A.I. Schutyser, *Food Bioprocess. Technol.* **8**, 1495 (2015)
  6. S. Fernando, *LWT.* **141**, 110961 (2021)
- Annotation ID="4" Type="" Text="The following mandatory elements (Volume number) of the reference are missing in the Manuscript. Please check and verify." Category="Completeness" 7. G. Fayaz, M. Mhamadi, D. Rodrigue, S.L. Turgeon, S. Khalloufi, *Food Bioprocess. Technol.* (2023)
8. F.G.B. Los, A.A.F. Zielinski, J.P. Wojeicchowski, A. Nogueira, I.M. Demiate, *Curr. Opin. Food Sci.* **19**, 63 (2018)
  9. D. Truong, G. Kumar, G. Williamson, L. Devkota, S. Dhital, *Food Hydrocoll.* **156**, 110350 (2024)
  10. A. Kilua, H. Chihiro, K. Han, K. Homma, N. Fukuma, *Bioact Carbohydrates Diet. Fibre.* **24**, 100232 (2020)
  11. V. De Freitas, *Foods* **12**, 1052 (2023)
  12. B.D. Oomah, S. Ward, P. Balasubramanian, *Food Res. Int.* **43**, 1410 (2010)
  13. J. Zhang, M. Li, C. Li, Y. Liu, *Grain Oil Sci. Technol.* **2**, 97 (2019)
  14. A. Miś, A. Nawrocka, D. Dziki, *Food Bioprocess. Technol.* **10**, 1031 (2017)
  15. F.C. Leal, K.M. Ueda, M.S.T. Arantes, T.A. de Lima, F.A. Hansel, W.L.E. Magalhães, C.V. Helm, R.A. de Freitas, F.O. Farias, M.R. Mafra, and L. Igarashi-Mafra, *Food Chem.* **440**, 138195 (2024)

16. Y. Lu, T. Kokje, M.A.I. Schutyser, L. Zhang, LWT. **169**, 114013 (2022)
17. Y. Lu, L. Zhang, M.A.I. Schutyser, LWT. **186**, 115206 (2023)
18. T. Gonçalves, T. Gabriel, G. De Lima, M. Matos, W. Luiz, E. Magalhães, L. Benathar, B. Tavares, C.V. Helm, J. Food Process. Preserv. **44**, e14464 (2020)
19. T.A.D.M. De Lima, G.G. De Lima, B.S. Chee, J.G. Henn, Y.J. Cortese, M. Matos, C.V. Helm, W.L.E. Magalh, and M. J. D. Nugent, Polymers (Basel). **14**, 2776 (2022)
20. G.G. De Lima, N.B. De Miranda, T.G. Timm, M. Matos, T. Angelina, M. De Lima, W. Luiz, E. Magalhães, L. Benathar, B. Tavares, F.A. Hansel, C.V. Helm, Food Funct. **11**, 9820 (2020)
21. J. Ao, J. Wang, H. Shen, Y. Xie, Y. Cai, M. Xi, Y. Hou, M. Li, A. Luo, Food Bioprocess. Technol. **17**, 4679 (2024)
22. M.L. Menezes, C.C. Kunz, P. Perine, N.C. Pereira, de Barros, Acta Sci. - Technol. **35**, 291 (2013)
23. IAL, *Métodos Físico-Químicos Para Análise De Alimentos* (IV) (IAL, São Paulo, 2008)
24. G.W. Jr Latimer, in 19th ed. (Gaithersburg, 2012), p. 2
25. F.G.B. Los, A.A.F. Zielinski, J.P. Wojeicichowski, A. Nogueira, I.M. Demiate, Food Anal. Methods. **12**, 148 (2019)
26. V.L. Singleton, J.A. Jr. Rossi, J.A. Jr. Rossi, Am. J. Enol. Vitic. **16**, 144 (1965)
27. C. Zaiontz, (2025)
28. U.E. Inyang, I.O. Oboh, B.R. Etuk, Adv. Chem. Eng. Sci. **8**, 27 (2018)
29. C. Ertekin, M.Z. Firat, Crit. Rev. Food Sci. Nutr. **57**, 701 (2017)
30. S. Duarte, E. Betoret, C. Barrera, Sustainability. **15**, 29794 (2023)
31. D.I. Onwude, N. Hashim, R.B. Janius, N.M. Nawi, K. Abdan, Compr. Rev. Food Sci. Food Saf. **15**, 559 (2016)
32. J.A.C. Bento, D.K. Morais, R.S. de Berse, P.Z. Bassinello, M. Caliar, M.S. Soares, Júnior, Appl. Food Res. **2**, 100027 (2022)
33. D.M. Dias, N. Kolba, D. Binyamin, O. Ziv, M.R. Nutti, H.S.D. Martino, R.P. Glahn, O. Koren, E. Tako, Nutrients. **10**, 1 (2018)
34. S.O. Keskin, T.M. Ali, J. Ahmed, M. Shaikh, M. Siddiq, M.A. Uebersax, Legum Sci. **4**, e117 (2022)
35. M. Sandoval-Peraza, D. Betancur-Ancona, L. Chel-Guerrero, J. Food Nutr. Res. **53**, 127 (2014)
36. L. Mojica, A. Meyer, M.A. Berhow, E.G. de Mejía, Food Res. Int. **69**, 38 (2015)
37. A.C. Telles, L. Kupski, E.B. Furlong, Food Chem. **214**, 293 (2017)
38. Y. Wang, X. Zhang, G. Chen, J. Yu, L. Yang, J. Funct. Foods. **24**, 359 (2016)
39. M.M. Contreras, G. Irene, I. Romero, E. Castro, Biol. Life Sci. Forum. **6**, 60 (2021)
40. B. Jeganathan, J. Gao, T. Vasanthan, F. Temelli, J. Food Compos. Anal. **112**, 104695 (2022)
41. A.C. Miano, E. Saldaña, L.H. Campestrini, A.F. Chiorato, P.E.D. Augusto, Food Res. Int. **107**, 182 (2018)
42. F.T. Seta, X. An, L. Liu, H. Zhang, J. Yang, W. Zhang, S. Nie, S. Yao, H. Cao, Q. Xu, Y. Bu, H. Liu, Carbohydr. Polym. **234**, 115942 (2020)
43. A.G. Souza, D.F. Santos, R.R. Ferreira, V.Z. Pinto, D.S. Rosa, Int. J. Biol. Macromol. **165**, 1803 (2020)
44. C.L. Dikeman, G.C. Fahey, Crit. Rev. Food Sci. Nutr. **46**, 649 (2006)
45. A. Nartea, A. Kuhalskaya, B. Fanesi, O.L. Orhotohwo, K. Susek, L. Rocchetti, V. Di Vittori, E. Bitocchi, D. Pacetti, R. Papa, Compr. Rev. Food Sci. Food Saf. **22**, 1953 (2023)
46. S.P. Bangar, A. Singh, A.O. Ashogbon, H. Bobade, Int. J. Biol. Macromol. **237**, 124069 (2023)
47. L.J. Symons, C.S. Brennan, J. Food Sci. **69**, 257 (2004)
48. L.C. González, M.A. Loubes, M.P. Tolaba, Food Hydrocoll. **82**, 155 (2018)
49. P.M. Palavecino, M.C. Penci, P.D. Ribotta, J. Food Eng. **262**, 22 (2019)

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