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Electrostatic separation for sustainable production of rapeseed oil cake protein concentrate: Effect of mechanical disruption on protein and lignocellulosic fiber separation

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ABSTRACT

Rapeseed oil cake (ROC) is a biomass of particular interest for biorefinery and food activities, due to its high protein, lignin, polyphenol and fiber contents. An environmental separation process combining ultrafine milling and electrostatic separation was recently developed for proteo lignocellulosic plant fractionation. Several of the parameters of the fractionation process (milling mode, electrode voltage, charge, particle size, etc.) can affect the efficiency of protein separation and extraction. We assessed the impact of three different mechanical modes of milling ball milling, impact grinding and jet milling on the electrostatic separation (ES) of lignin and protein from ROC biomass. The initial biomass and the ES fractions were characterized and correlations were identified between particle colors and sizes and their protein and fiber contents. Mechanical grinding had a significant impact on the surface properties of the particles, influencing their charge and the electrostatic separation of lignin and protein from biomass. The combination of impact grinding and electrostatic separation appear the most efficient and effective technologies for rapeseed oil cake protein concentrate.

1. Introduction

Rapeseed oil cake (ROC) is a by product of oil extraction. It is produced in large quantities, with worldwide production estimated at 35 million ton in 2012. ROC has potential for use as a raw material in the production of materials, antioxidants, aromatic compounds, feed/food and energy, due to it high protein, polyphenol and fiber contents. ROC biomass typically contains about 30 40% protein and 15 20% lignin [1,2]. It is also rich in carbohydrates, including pectins, hemicelluloses and cellulose, which together form a complex network within plant cell walls [2].

ROC is currently used in the production of animal feed, due to its high protein content. However, ROC biomass could also be used as an ingredient in the manufacture of human foods, due to its high antioxidant and fiber contents. This new valorization of ROC valorization will depend on the quality and quantity of the proteins extract, which will, in turn, depend on the extraction and purification methods used. Various studies have shown that rapeseed proteins have several very useful properties [3 6]. ROC protein extraction methods should conserve protein structure and function, so as to preserve the organoleptic quality of ROC proteins. Chemical methods are currently used to extract proteins from ROC [7]. These methods generate effluent and may modify the functional proprieties of these macromolecules, which may be highly problematic. Most of the fractionation and separation operations used are based on expensive chemical processes (pulping, hydrolysis, solvent extraction, steam and ammonia explosion, ionic liquid extraction etc.) that cannot isolate the main compounds (polyphenols proteins, carbohydrates etc.) without a loss of integrity. Indeed, plant materials have a robust supramolecular structure, the deconstruction of which often requires harsh conditions. In such conditions, the native functions of the compounds may not be fully preserved [8]. Furthermore, these processes require large amounts of energy, water and/or solvent. The downstream steps in purification and processing involve solvent removal, which also greatly increases the final cost of the extracted biomolecules and biopolymers. In this context, several processes have been proposed for the extraction and concentration of proteins and for the removal of lignin polymers by alternative technologies not requiring the use of chemical compounds and generating no effluent [9 12].

A process combining mechanical milling and electrostatic separation (ES) was recently proposed as a promising method for fractionating different plants materials to obtain protein rich fractions [13]. Electrostatic separation processes lead to enrichment in proteins, due to the removal

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of lignin from the protein fraction. However, several parameters can influence the fractionation of proteins, lignin and lignocellulosic materials from different plants materials [14 16]. Particle charge plays a major role in separation, as highlighted by Wang and coworkers in a theoretical study of the application of ES to a gluten starch mixture [17]. The conductivity of a particle depends on its chemical composition, surface properties, moisture content, size, shape and density. Some of these physicochemical proprieties also depend on the type of milling [18], the first step in the electrostatic fractionation process.

This study investigated the effects of three mechanical dissociation modes: ball milling (BM), impact grinding (IG) and jet milling (JM) on the electrostatic fractionation of proteins and lignocellulosic polymers from ROC biomass, to generate fractions highly enriched in protein or fiber (Fig. 1).

2. Materials and methods

2.1. Raw material

Rapeseed oilcake with moisture content of 8.1% was obtained from a French commercial retailer specializing in oil meal production after the cold pressing of partially dehulled rapeseed *(B. napus L.)*, and dried.

2.2. Mechanical dissociation modes

A knife milling system (Retsch SM 100, Germany) was initially used to reduce the particle size of ROC pellets, using a grid with a screen size of 2 mm. The knife milled ROC particles were then ground ultrafine by one of three different mechanical dissociation methods operating at pilot scale (1 10 kg/h). The modes of grinding tested were: impact grinding (100 UPZ, Hosokawa Alpine, Germany) with a 0.1 mm screen size grid operating at room temperature and 18,000 rpm; jet milling (Hosokawa Alpine, Germany) with a feed rate of 1.1 kg/h at 2000 rpm; and ball milling with a 2 L porcelain jar containing 70 balls (35 balls of 25 mm in diameter and 35 balls of 20 mm in diameter). Ball milling was then conducted in a planetary ball mill at 51 rpm for 1 to 20 h with a ball: powder: empty space ratio of 1:1:1.

2.3. Electrostatic separation (ES) technology

Fine milling was coupled with electrostatic separation technology, to separate proteins and lignin from ROC biomass. A pilot scale electrostatic separator (TEP System, Tribo Flow Separations, Lexington, USA) was used to fractionate ultrafine ROC biomass powder (F0) into two fractions of different compositions (F+ and F−) [1]. The feed system of the ES was operated at 150 rpm; ROC particles were conveyed by compressed air into a charging line, and charged by triboelectric effects, due to the impact of ROC particles on each other and with the charging line walls. The charged particles were then introduced into a separation chamber containing two high voltage electrodes (10,000 V), in which the positively charged particles (F+) were attracted to the negative electrode and the negatively charged particles (F−) to the positive electrode. A particle recovery system equipped with two cyclones was used

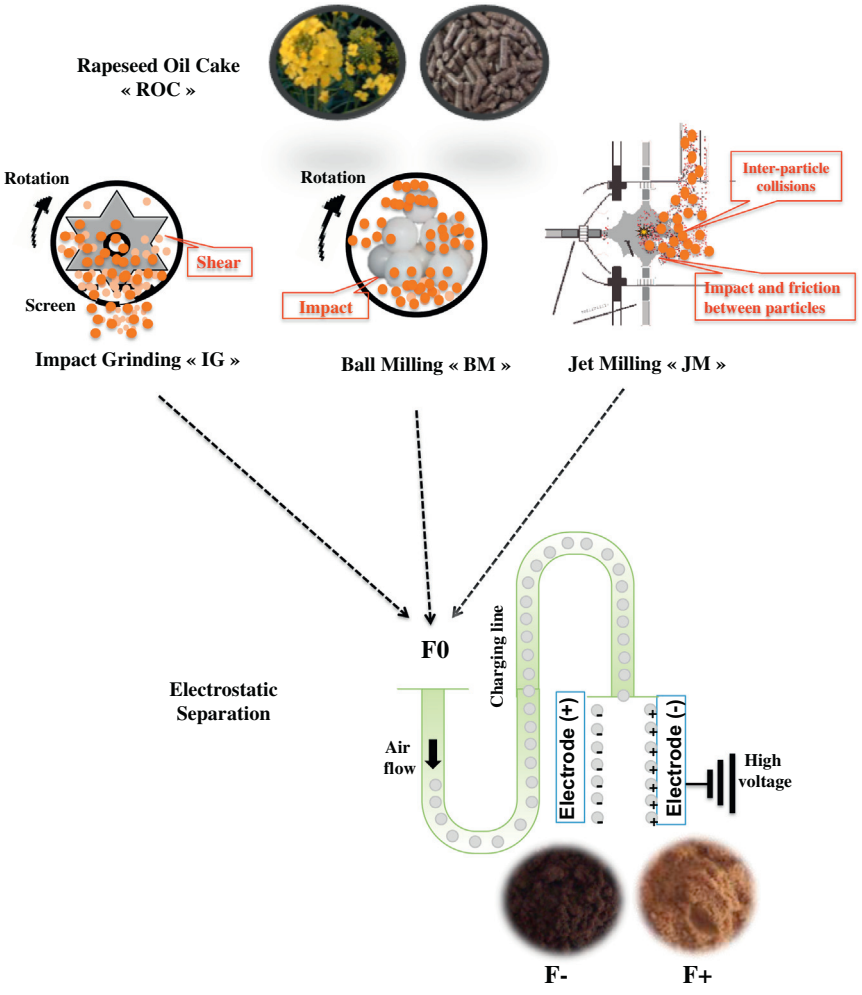


Fig. 1. Fractionation scheme for ROC biomass coupling electrostatic separation with various types of milling.

to recover two fractions: one consisting of the positively charged parti cles (F+) and the other of the negatively charged particles (F−).

2.4. Particle size and color

Following ROC biomass fractionation, the particle sizes of the F0, F+ and F− fractions were determined by laser diffraction (Mastersizer 2000, Malvern Instrument) at room temperature in ethanol. All frac tions were characterized in terms of median particle diameter (d50) and granulometric distribution (expressed as a percentage by volume, vol%). The color of each fraction was determined with a colorimeter (Minolta CR 410 Colorimeter, Nieuwegein, the Netherlands) and the CIELAB color space system: L* provides information about luminance, and a* and b* provide information about sample color (a*: from green to red, and b*: from dark blue to yellow). Measurements were made in duplicate, and the mean value was calculated for each of the three pa rameters (L*, a*, and b*).

2.5. Analysis of the composition of ROC fractions

The dry matter and ash contents of the various fractions were deter mined. Biomass was dried at 60 °C for 2 h and then incinerated at 900 °C for 1 h. Protein content was determined by the Kjeldahl method. Cellu lose and hemicellulose contents were determined by high performance anion exchange chromatography with pulse amperometric detection (HPAEC PAD) on an ICS 5000 ion chromatography system equipped with a CarboPac PA20 column (Dionex, Sunnyvale, CA), after two step hydrolysis with sulfuric acid [19]. Briefly, ROC biomass was hydrolyzed, in duplicate, with 70% H_2SO_4 for 1 h at 30 °C and then with 4% H_2SO_4 for 65 min at 120 °C. Klason lignin was determined as the insoluble residue remaining after the hydrolysis of cell wall polysaccharides with sulfuric acid [1]. For the extraction of water soluble carbohydrates, ROC frac tions were mixed with water (10%, w/v) and incubated for 1 h at 60 °C, in duplicate. Monosaccharides (glucose, fructose, galactose, arab inose, xylose, and mannose) and sucrose were identified directly, by high performance anion exchange chromatography with pulse amper ometric detection (HPAEC PAD) on an ICS 5000 ion chromatography system equipped with a CarboPac PA20 column (Dionex, Sunnyvale, CA).

2.6. Scanning electron microscopy

Scanning electron microscopy (SEM) was performed with a Phenom G2 Pure electron microscope (Phenom World BV, Eindhoven, the Netherlands), for observation of the ultrafine RSM and SFM fractions. The samples were placed on double sided adhesive tape and fixed to SEM stubs. Excess particles were removed with a dry airstream. No sam ple pretreatment was required.

2.7. Measurement of specific energy consumption

The total energy (E_{TER}) consumed during the fractionation process was defined as the sum of the energies required for milling (E_M) and electrostatic separation (E_{ES}). Total energy consumption (expressed in $Wh.kg^{-1}$ ROC) was determined as follows:

$$E_{TER} \quad E_M + E_{ES} \tag{1}$$

E_M and E_{ES} were measured with a wattmeter (Barakat et al. 2014). Energy consumption was calculated as follows:

$$E_M \quad \frac{\int_0^t (P_t - P_0)dt}{m} \quad \frac{\int_0^t \Delta P_t dt}{m} \tag{2}$$

Where *Pt* is the power (watts) consumed at time *t*, *P0* is the mean power consumption (watts) under idle conditions (without biomass), and m is the mass (kg) of biomass to be ground. Three power measurements (*P0* and *Pt*) were obtained for each sample.

2.8. Statistical analysis

The results presented are the means of duplicate analyses. All data were subjected to analysis of variance with XLstat® (Addinsoft, Brook lyn, USA). Significant differences ($P < 0.05$) between individual means were identified in Tukey's test.

3. Results and discussion

3.1. Ultrafine milling of ROC biomass

The ROC used in this study consisted of 35.8 g protein/100 g dry bio mass, 6.1 g ash/100 g dry biomass and 13.4 g lignin/100 g dry biomass. These findings are consistent with previous reports, indicating values of between 30 and 40 g protein/100 g dry biomass, between 10 and 24 g lignin/100 g dry biomass (Pustjens et al., 2013; Jeong et al., 2014) and between 5 and 7 g ash/100 g dry biomass [1,2,20,21]. Raw ROC (after knife milling with a 2 mm screen size grid) was subjected to three different types of milling, to dissociate the various constituents (protein, lignin, cellulose and ash) before electrostatic separation (Fig. 1).

The objective was to produce the particles of the same size (D_{50}) by the various milling modes and to assess the impact of the mode of dis sociation on the fractionation of proteins and fiber from biomass. The raw material was fragmented and deconstructed by the application of different mechanical stresses: impact, compression, friction, and shear ing (Fig. 1). For example, in jet milling (JM), the particles are projected against each other in an air stream, and the major mechanical stresses generated are impact and friction between particles (Fig. 1).

Knife milling (KM) involves the use of a rotor to drive different tools; rotor speed is generally adjustable. A sieve or a screen is used to control particle size in the final product. Such mills mostly generate impact and shear stresses (Fig. 1). During ball milling, the biomass is exposed to im pact and compression stresses induced by collisions between the balls, particles and container walls [22]. The particle size distributions of sam ples prepared by different milling modes are presented in Fig. 2. Median particle diameters of 42, 43 and 42 μm were obtained by impact grind ing, ball milling and jet milling, respectively. Mechanical dissociation mode had a significant effect on particle size distribution (Fig. 1), even though the three milling modes produced particles with almost identi cal d_{50} values (i.e. 42 μm). Particle size distribution was bimodal for BM but not for the other milling modes. Samples generated by BM may,

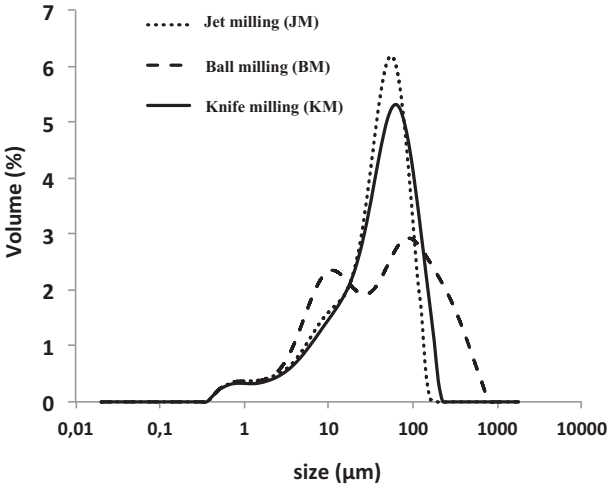


Fig. 2. Impact of milling mode (ground by jet, ball and impact) on particle size distribution of ROC samples

therefore, contain heterogeneous particles with different compositions. This effect can be explained by the greater damage to ROC particles caused by the stresses generated during BM (impact and compression). Silva and coworkers investigated the effect of milling mode on wheat straw structure [23,24]. They showed that BM was the only milling method capable of generating significant changes in macromolecular structure. SEM of the separated fractions (Fig. 3) revealed clear differ ences between the negatively (F−) and positively (F+) charged frac tions and the raw ROC biomass (F0). SEM of the ROC raw material (F0) showed it to consist of a mixture of granular globular bodies and cellular matrices of fiber rich constituents. Following ultrafine milling and electrostatic separation, the globular protein bodies were concen trated in the positive fraction, whereas the cellular and fibrous matrices were recovered in the negatively charged fraction (F−). These findings clearly confirm the impact of milling mode on the fragmentation and deconstruction of the supramolecular structure of ROC, which can influ ence directly the physicochemical proprieties of particles and, thus, the separation of protein and fiber from ROC biomass.

3.2. Electrostatic fractionation of proteins and lignocellulose from ROC biomass

Electrostatic separation (ES) has been used in several studies for the efficient fractionation of different biomasses, such as wheat bran and sunflower oil cakes, to separate cellulose, proteins, lignin and ash with out water or effluent [1,25,26]. Electrostatic separation was performed on each of the milled ROC sample prepared with the different dissocia tion modes, with F0 denoting the initial material (Fig. 1). Samples were separated into two fractions: positively charged particles (F+) and neg atively charged particles (F−). The particle size distribution, color and biochemical composition of the three samples corresponding to the three different milling modes were then determined.

Table 1
Biochemical composition of the ROC raw materials (F0) and electrostatically separated fractions (F and F+). Values in the same row followed by the same letter are not significantly different ($P > 0.05$).

	Ball milling (BM)		Jet milling (JM)		Knife milling (KM)	
(% DM)	**F**	**F +**	**F**	**F +**	**F**	**F +**
Yield	**40.2**	**59.8**	**45.9**	**45.8**	**34.8**	**58.0**
Ash	5.6	6.0	5.3	6.0	5.4	6.3
Lignin	**21.5**	**10.0**	**28.0**	**6.7**	**26.5**	**4.3**
Protein	**25.7**	**40.0**	**18.0**	**43.0**	**19.6**	**43.8**
Water-Soluble Carbohydrates	**10.2**	**12.6**	**8.4**	**9.6**	**7.1**	**10.1**
Sucrose	3.5	6.8	4.0	7.0	3.8	7.5
Glucose	1.8	0.3	1.1	0.5	1.0	0.3
Fructose	2.3	3.3	1.7	0.2	0.6	0.1
Oligosaccharides	2.7	2.2	1.6	1.9	1.7	2.3
Structural Carbohydrates	**22.8**	**20.9**	**27.8**	**21.1**	**26.7**	**22.0**
Arabinose	6.9	6.1	7.6	6.4	7.3	6.9
Galactose	2.7	2.7	3.3	2.7	3.3	2.9
Glucose	10.3	9.3	14.2	9.1	13.1	8.9
Xylose	2.7	2.8	2.5	2.8	2.8	3.2
Mannose	0.1	0.1	0.2	0.1	0.2	0.1
Color						
a*	1.6	2.0	2.6	3.5	2.7	4.2
b*	14.7	21.2	12.6	26.9	13.7	27.0
L*	64.3	72.3	51.2	70.1	51.4	67.7

The chemical composition and structural features of these fractions are presented in Table 1. The ROC, (F+) and (F−) fractions differed in terms of their appearance; "L*", "a*" and "b*" values were higher for positively charged fractions. F+ fractions were more "yellowish" than

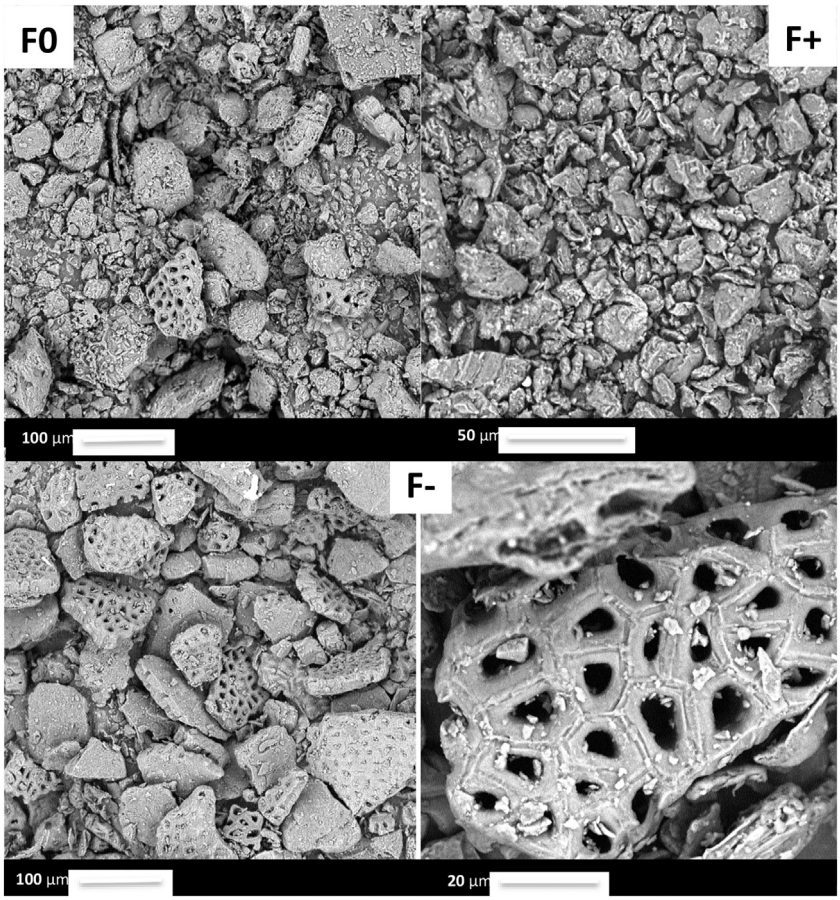


Fig. 3. Scanning electron micrographs of the negatively (F) and positively (F+) charged fractions and starting materials

F− fractions, which were almost brown. F+ and F− yields were similar for JM. By contrast, F+ yields were higher than F− yields for the impact and ball milling techniques (about 60% F+ and 40% F−). As there was no significant difference in particle sizes between F0 samples, these dif ferences in yield can be assumed to reflect physicochemical differences between particles due to the mode of dissociation. Protein content was higher in the positively charged fractions, whereas lignin content was higher in the negatively charged fractions, confirming published results [1,25]. These results confirm the SEM observations, which clearly indi cated a more lignocellulosic structure for F− than for F+.

The highest protein concentration (44.8%) and the lowest lignin con tent (4.3%) were obtained with KM technology (Table 1). By contrast, the highest lignin concentration (28%) was obtained with JM. This con tent is twice that of the raw ROC material (13.4% for F0). The lignin con tents of the negatively charged fractions (F−) obtained by KM and JM were not significantly different.

The triboelectric charging of particles is, therefore, controlled exclu sively by the properties of the surface layer. The positive or negative sur face charge density acquired during triboelectric charging is thus determined by the ability of the surface of the material to donate or accept electrons when it comes into contact with another material [27].

3.3. Influence of the fractionation process on physicochemical properties

Principal Component Analysis (PCA) and Hierarchical Archimedean Couplae (HAC) analysis were performed to assess the correlations be tween the physical and chemical properties of fractions obtained by electrostatic separation (Fig. 4). These analyses were performed on data for all fractions, focusing on six variables: three biochemical attri butes (protein, ash and lignin contents) and L*, a*, b* values. These anal yses classified samples into groups with significantly different biochemical compositions.

The HAC performed on the PCA data identified three different groups of samples (Shown by ellipses in Fig. 4B): the negatively charged sam ples, the positively charged samples and starting materials. The nega tively charged samples had high lignin content. The group corresponding to the starting material was located in the middle of the map as these samples had intermediate contents. Finally, the posi tively charged samples had high protein concentrations, and high values for L*, a* and b*. Protein content was positively correlated with L* and b* values. Thus, b* and L* values can be used as indicators of the efficiency of ROC biomass fractionation by ES.

3.4. Comparison of milling modes in terms of energy requirement and efficiency

The total energy requirement (E_{TER}) of the different milling modes was calculated according to Eqs. (1) and (2) [28 30]. The total energy requirement (E_{TER}) of milling processes depends on the physicochemi cal proprieties of the biomass used and the distributions of particle size before and after milling, which are also highly dependent on the equipment or machine used. Generally, at the laboratory scale, E_{TER} or specific energy can be measured only with size reduction machines equipped with a size screen, which is not possible for BM and JM. Thus, total energy (E_{TER}) alone, rather than specific energy, was used to compare the various milling modes (Fig. 5).

The specific energy requirements for the reduction of particle size in ROC biomass were estimated at 1.73, 3.54 and 4.72 kWh.kg^{-1} ROC for KM, BM and JM, respectively (Fig. 5). The energy consumed for electro static separation was estimated at 1.64 kWh.kg^{-1} ROC. Energy con sumption was clearly lower for KM than for the other two milling modes. BM and JM required more energy than IG to achieve a similar final particle size of 42 μm. We conclude from these data that E_{TER} is highest for JM, followed by BM and, finally, KM, which has the lowest E_{TER}.

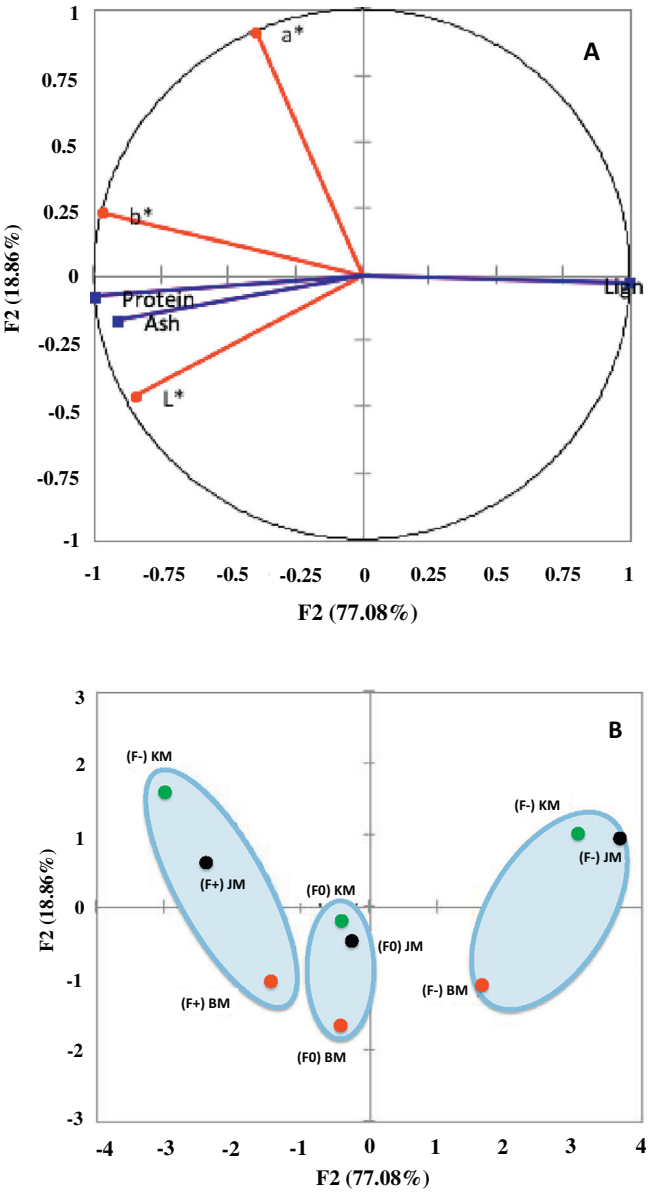


Fig. 4. PCA and HAC analyses of the biochemical composition of the fractions. A: correlation circle with the active variables (color) and supplementary variables (biochemical composition) and B: similarity map of factors 1 and 2, and clustering of the samples into three classes.

Fig. 5 also show the efficiencies of lignin and protein extraction and separation from lignocellulose biomass as a function of grinding mode in the absence of added water and chemical catalyst. Protein extraction yield was estimated at 55, 66 and 74% for JM, BM and KM, respectively. Lignin extraction yields of 96, 65 and 69% were obtained for JM, BM and KM, respectively. Thus, the extraction yields for lignin and protein clearly depend on the mode of milling. JM technology extracted lignin more efficiently than the other techniques, with yields reaching 96% without the addition of water, chemicals or solvents. By contrast, KM was the most efficient technique for protein extraction, with a yield of 74%.

$$\text{Dry.}EE\text{biomass} \quad \frac{\textit{Extracted lignin or protein yield } (kg/kg\ biomass)}{\textit{Total energy } (kWh/kg)} \qquad (3)$$

E_{TER} and energy efficiency (η) are generally used to compare the per formance and efficiency of different pretreatment processes [31,32]. However, few data have been published for comparisons of energy

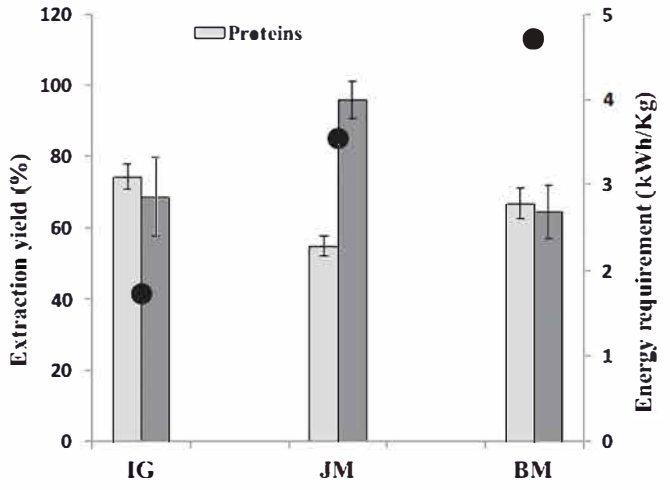


Fig. 5. Energy requirement (kWhkg ^{1}) and extraction yields for lignin and protein of the different milling modes coupled to ES.

consumption and energy efficiency between chemical, physicochemical and mechanical treatments of lignocellulosic biomass. We calculated dry fractionation energy efficiency (Dry.$EE_{biomass}$) coefficient (Eq. (3)), to compare the efficiencies of different milling modes coupled to ES for the extraction and separation of proteins and lignocellulosic fibers.

The most effective dry fractionation process has the highest Dry.$EE_{biomass}$ values. The highest Dry.$EE_{biomass}$ obtained here was 0.429 kg protein kWh^{-1} and 0.398 kg lignin kWh^{-1}, for KM, and the lowest Dry.$EE_{biomass}$ was 0.142 kg protein kWh^{-1} and 0.137 kg lignin kWh^{-1}, for BM (Table 2). The high energy consumption of BM, which gave the lowest Dry.$EE_{biomass}$ value, can be attributed largely to the much longer milling time than for KM.

These results indicate that the energy efficiency dry fractionation (Dry.$EE_{biomass}$) can be used to compare various fractionation methods. However, it is not straightforward to compare the energy requirements of different milling modes at laboratory scale, as these requirements depend on the type, power, and size of the milling apparatus, and the experimental conditions. In our study comparing different milling modes at laboratory scale, knife milling (KM) coupled with ES seemed to be the most efficient of the mechanical pretreatments tested, in terms of energy and the fractionation and separation of protein and lignocellulosic fibers.

4. Conclusion

We used electrostatic separation to assess the effect of the mode of mechanical dissociation on rapeseed oil cake fractionation and proteins concentrates. Three different modes of dissociation were used to grind the raw material: ball, impact and jet milling technologies. Milling mode affected the protein contents of the fractions obtained by electrostatic separation (ES). The biochemical composition and physical properties of ROC biomass fractions (protein, lignin, polysaccharides, color, particle size) and dry fractionation energy efficiency indicated that knife milling (KM) coupled with ES was the most effective technology for the fractionation and separation of proteins and lignocellulosic fibers.

Table 2
Extraction yields for lignin and protein from biomass, energy requirement and dry energy efficiency for the different modes of milling.

Milling mode	Protein yield (%)	Lignin yield (%)	E_{TER} (kWh/kg)	Dry.$EE_{biomass}$ (kg/kWh)	
				Protein	Lignin
KM	74.20	68.82	1.73	0.429	0.398
JM	55.01	95.91	3.54	0.155	0.271
BM	66.82	64.50	4.72	0.142	0.137

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