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Aqueous extraction of residual oil from sunflower press cake using a twin-screw extruder: Feasibility study

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ABSTRACT

The objective of this study was to evaluate the feasibility of an aqueous process to extract the residual oil from sunflower press cakes using a co-rotating twin-screw extruder. Two different configurations were tested: the expression from whole seeds followed by the aqueous extraction, in two successive apparatus or in the same one. For the aqueous extraction stage, the oil yield depended on the operating conditions including screw rotation speed, screw profile, and inlet flow rates of press cakes and water. Liquid/solid separation required the addition of a lignocellulosic residue (wheat straw), upstream from the filtration zone. However, even with maximum fiber inlet flow (around 20% of the inlet flow rate of the solid matters for the highest amount of wheat straw), drying of the cake meal did not improve. The lixiviation of the material was also incomplete. Oil yield was better when the expression and the aqueous extraction were conducted in the same extruder. For all the trials carried out using such a configuration, the corresponding cake meal contained less than 10% residual oil, and the total oil yield was 78% in the best operating conditions. Nevertheless, the contribution of the aqueous extraction stage was extremely limited, less than 5% in the best trial, partly due to a ratio of the water to the press cake too low. For the aqueous extraction stage, the oil was extracted in the form of an oil-in-water emulsion whose stability was minimized because of its low proteins content due to their thermo-mechanical denaturation during the expression stage.

1. Introduction

Sunflower (*Helianthus annuus* L.) is cultivated for its seeds' high oil content. Oil represents up to 80% of its economic value. The industrial processes for oil production consist of four successive stages: trituration, pressing, extraction of the residual oil using hexane and refining (Isobe et al., 1992; Rosenthal et al., 1996). The extraction yields are close to 100% with very good oil quality. However, the use of hexane to remove oil from the press cake is an increasingly controversial issue and could be prohibited due to its carcinogenicity (Galvin, 1997). Consequently numerous solvents have been considered.

Water is an interesting alternative medium (Hagenmaier, 1974; Southwell and Harris, 1992). In the aqueous extraction process, the oil, being immiscible with water, separates readily from the extract. The fine crushing of the seeds is the first stage in cell disruption. It facilitates the diffusion of the soluble compounds and the release of the oil. Liquid/solid separation by centrifugation produces three fractions: the hydrophobic phase (oil-in-water emulsion), the hydrophilic phase and the

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insoluble phase (Rosenthal et al., 1996; Mechling, 2002; Evon et al., 2007). The oil is then recovered after demulsification by alcohol extraction. Besides, aqueous extraction of oil can be regarded as a process primarily aimed at solubilizing proteins which results in the release of the oil (Rosenthal et al., 1996).

The oil yield and the protein yield are 86% and 85%, respectively, when the aqueous extraction is carried out in the next operating conditions: dispersion of the ground seeds in water inside a batch reactor, extraction by simple stirring at room temperature, 45 min for the extraction duration, 1:10 for the ratio of the seeds to the water, 10 for pH (Hagenmaier, 1974).

Because all the cotyledon cells are not efficiently ruptured during the extraction process, a fraction of the oil remains trapped in the cellular matrix and oil yield is so limited. Extraction efficiency can be improved by mechanical lysis of the cells in a twin-screw extruder (Isobe et al., 1992; Bouvier and Guyomard, 1997; Lacaze-Dufaure et al., 1999a,b; Amalia Kartika et al., 2005a,b), including when the twin-screw extrusion technology is used to conduct the direct extraction of oil from whole sunflower seeds according to an aqueous extraction process (Evon et al., 2007).

Co-penetrating and co-rotating twin-screw extruders are most common (Dziezak, 1989). A very wide choice of screw elements is available. The screw elements affect different functions such as conveying, heating, cooling, shearing, crushing, mixing, chemical reaction, liquid/solid extraction, liquid/solid separation, and drying (Rigal, 1996).

The screw profile (or screw configuration) is defined by the arrangement of different characteristics of screw elements (pitch, stagger angle, and length) in different positions and spacings. It is the main factor influencing performance (product transformation, residence time distribution, and mechanical energy input) during extrusion processing (Gogoi et al., 1996; Choudhury et al., 1998; Gautam and Choudhury, 1999a,b).

The forward pitch screws mainly ensure conveying action. The monolobe paddles exert a radial compression and shearing action on the matter but have limited mixing ability. In combination with forward pitch screws, the bilobe paddles exert significant mixing, shearing, conveying, and axial compression actions on the matter. The bilobe paddles are favourable to intimate mixing required in the liquid/solid extraction of soluble constituents in the cell structure. Finally, the reversed pitch screws carry out intensive shearing and considerable mixing on the matter, and exert a strong axial compression in combination with forward pitch screws (Rigal, 1996). The reversed pitch screws are frequently used to place pressure on the matter, which is essential for the separation of liquid and solid phases by filtration.

When oleic sunflower oil is expressed, a longer reversed pitch screw improves oil yield, which can rise 80% in the optimized operating conditions (Lacaze-Dufaure et al., 1999a). When two reversed pitch screws are used, the oil yield increases with the distance between them and with the decrease of the screw pitch (Amalia Kartika et al., 2005a). The best oil yield obtained with such screw configuration is close to 70% but the introduction of a second filtration zone improves it to 85% (Amalia Kartika et al., 2005a). The corresponding press cake contains less than 13% residual oil and the quality of the oil produced is similar to that of classic extraction.

Twin-screw extruders can also be used as liquid/solid extractors. The injection of a solvent promotes oil extraction by solubilizing the triglycerides. An oil yield of 80% was obtained with 2-ethylhexanol (Lacaze-Dufaure et al., 1999b) and 85% with a mixture of sunflower methyl esters (Amalia Kartika, 2005).

However, pressing efficiency can be altered by the consistency of the mixture. Liquid/solid separation is more difficult than when the seeds are expressed without injection of a solvent. Thus, the best oil yield is only 55% when the injected solvent is water (Evon et al., 2007). The oil is then extracted in the form of an oil-in-water emulsion. This hydrophobic phase is stabilized by phospholipids and proteins at the interface, which are natural surface-active agents co-extracted during the process. The aqueous extraction also produces a hydrophilic phase. This largest fraction constitutes an aqueous extract of the soluble constituents from the seeds. Its dry matter contains a high proportion of water-soluble proteins, usable for their tensioactive properties after concentration.

Besides the direct aqueous extraction of the oil from sunflower seeds (Evon et al., 2007), this study aimed to show that a co-rotating twin-screw extruder could also be used in an aqueous extraction of the residual oil from press cakes produced after pressing of the whole seeds to improve the total oil yield. Consequently, water would replace hexane used traditionally in the second step of the conventional oil extraction processes.

2. Materials and methods

2.1. Materials

All trials were carried out using a single batch of sunflower seeds (La Toulousaine de Céréales, France). The moisture content of the seeds was $7.49 \pm 0.03\%$ and the lipid content was 50% (Table 1). The lipid extract contained 1.2 g of phospholipids per 100 g of oil. The cellulose, hemicelluloses, and lignins were mainly located in the hulls. The lignocellulosic residue used was wheat straw. Its moisture content was $6.20 \pm 0.02\%$. The wheat straw was cut into small pieces using a hammer mill (Electra VS 1, France) fitted with a 6 mm screen. All solvents and chemicals were analytical grade and were obtained from Sigma–Aldrich, Fluka, Prolabo and ICS (France).

2.2. Twin-screw extruder

Experiments were conducted with a Clextral BC 45 (France) co-penetrating and co-rotating twin-screw extruder. The extruder had seven modular barrels, each 200 mm in length, and different twin-screws which had segmental screw elements each 50 and 100 mm in length. Material (whole seeds or press cakes) was fed into the extruder inlet port by a volumetric screw feeder (Clextral 40, France). Some of the modules were heated by thermal induction and cooled by water circulation. Their number varied from two to four according to adopted shape. One or two filter sections consisting of six hemispherical dishes with perforations 1 mm in diameter were outfitted along the barrel to enable the filtrates containing the extracted oil to be collected. Screw rotation speed (S_S), the seed feed rate (Q_S) or the press cake feed rate (Q'_{C1}), and

Table 1 – Chemical composition of the sunflower seeds and the wheat straw used in this study, press cake A, and press cake B (% of dry matter).

	Seed	Wheat straw	Press cake A	Press cake B
Minerals	3.11 ± 0.01	7.69 ± 0.17	4.64 ± 0.02	4.73 ± 0.02
Lipids	49.70 ± 0.18	1.44 ± 0.09	17.19 ± 0.04	14.32 ± 0.10
Phospholipids	0.59 ± 0.02	–	–	–
Proteins	15.70 ± 0.09	2.44 ± 0.02	23.57 ± 0.02	24.54 ± 0.27
Cellulose	12.49 ± 0.20	39.72 ± 0.47	19.79 ± 0.27	20.34 ± 0.11
Hemicelluloses	6.88 ± 0.15	31.48 ± 0.31	14.08 ± 0.33	22.08 ± 0.03
Lignins	4.66 ± 0.10	11.51 ± 0.13	11.64 ± 0.49	13.61 ± 0.16

the barrel temperature (θ_c) were monitored from a control panel.

2.3. *Experimental*

2.3.1. *Expression and aqueous extraction in two successive twin-screw extruders*

2.3.1.1. *Expression stage*. A first extruder was used for the expression stage (Fig. 1). Whole sunflower seeds were fed into the extruder inlet port. Three operations then succeeded one another: conveying (forward pitch screws), trituration (succession of 10 monolobes paddles and two series of 5 bilobe paddles) and pressing (reversed pitch screws). The filtrate 1 (oil containing the foot) and the press cake were collected separately. The two screw profiles (profiles 1 and 2) tested in this study (Fig. 2) were inspired of those used by Amalia Kartika et al. (2005a,b). They were chosen to allow the production of two press cakes (press cake A and press cake B) with different residual oil contents.

The extruder was left to function for 20–25 min before any sampling to ensure the stabilization of the operating conditions. Such conditions include feed flow of seeds, temperature and current feeding the motor. Upon achieving steady operation, the filtrate 1 and the press cake were immediately collected over a period of 20 min to avoid any variability of the outlet flow rates. Sample collection time was determined

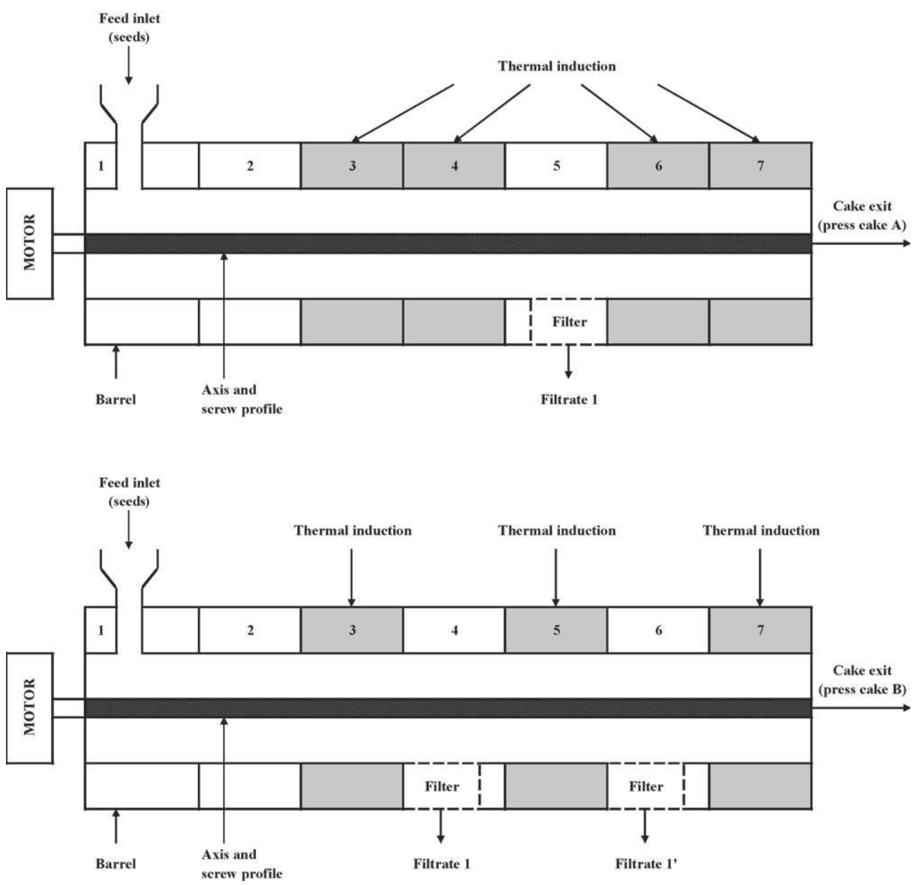


Fig. 1 – Schematic modular barrel of the Clextral BC 45 twin-screw extruders used for production of press cake A (S_S = 60 rpm, Q_S = 23.3 kg/h, θ_c = 80 °C), and press cake B (S_S = 105 rpm, Q_S = 23.9 kg/h, θ_c = 100 °C).

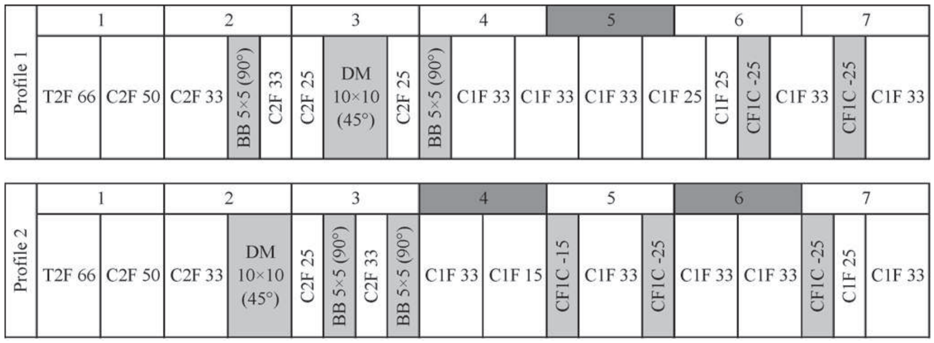


Fig. 2 – Screw configuration for production of press cake A (profile 1), and press cake B (profile 2). T2F, trapezoidal double-thread screw; C2F, conveying double-thread screw; C1F, conveying simple screw; DM, monolobe paddle-screw; BB, bilobe paddle-screw; CF1C, reversed screw. The numbers following the type of the screw indicate the pitch of T2F, C2F, C1F and CF1C screws and the length of the DM and BB screws.

with a stopwatch. For each test, sample collection was carried out once. The filtrate 1 and the press cake were weighed. The filtrate 1 was then centrifuged (8000 × g, 30 min, 20 °C) to eliminate the foot from the expressed oil.

The oil yields for the expression stage were calculated according to the following formulas:

$$R_{L1} = \frac{Q_{F1} \times T_{L1}}{Q_S \times L_S} \times 100,$$

where R_{L1} is the oil yield in proportion to the oil that the seed contains (%), Q_S the inlet flow rate of the whole sunflower seeds (kg/h), Q_{F1} the flow rate of the filtrate 1 (kg/h), T_{L1} the mass content of the expressed oil in the filtrate 1 (%), and L_S the oil content in the whole sunflower seeds (%).

$$R_{C1} = \frac{(Q_S \times L_S) - (Q_{C1} \times L_{C1})}{Q_S \times L_S} \times 100,$$

where R_{C1} is the oil yield based on the residual oil content of the press cake (%), Q_{C1} the flow rate of the press cake (kg/h), and L_{C1} the oil content in the press cake (%).

Although R_{L1} and R_{C1} are expressed in terms of the oil in the seed, R_{C1} is always higher than R_{L1} because it includes all the oil in the filtrate 1 (expressed oil and foot).

The energy consumed by the motor for the expression stage was determined according to the following formulas:

$$P_1 = U \times I_1 \times \cos\varphi \times \frac{S_S}{S_{max}},$$

where P_1 is the electric power supplied by the motor (W), U the motor's operating voltage (U = 460 V), I_1 the current feeding the motor (A), $\cos\varphi$ the theoretical yield of the extruder motor ($\cos\varphi$ = 0.95), and S_S and S_{max} the test speed and maximum speed (600 rpm) of the rotating screws (rpm), respectively.

$$SME_1 = \frac{P_1}{Q_S},$$

where SME_1 is the specific mechanical energy consumed by the motor per unit weight of sunflower seeds (W h/kg).

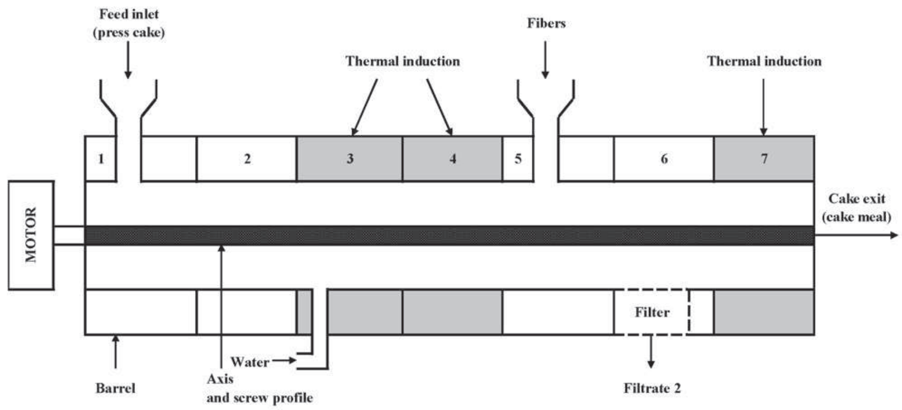
2.3.1.2. Aqueous extraction stage. The aqueous extraction of the residual oil from press cakes A and B was then carried out using a second stage in another extruder. Three modules (modules 3, 4 and 7) were heated to 80 °C (Fig. 3). Deionised water was injected using a piston pump (Clextral DKM K20-2-P32, France) at the start of module 3. The filter section was outfitted on module 6. Two screw profiles (profiles 3 and 4) were tested in this study (Fig. 3). For the two profiles, the press cake trituration zone is located in module 2. It consists of a succession of 10 monolobe paddles. The extraction zone was situated in modules 3, 4 and 5 and was composed of two series of 5 bilobe paddles, 20 cm apart. For each, the angle between bilobe paddles was 90° to allow the intimate mixing of liquid and solid. The reversed pitch screws were positioned in module 7, immediately downstream from the filtration module, to press the liquid/solid mixture. The two screw profiles tested differed only by the type of the reversed pitch screws in the pressing zone (Fig. 3).

To facilitate liquid/solid separation, wheat straw was introduced before the pressing zone, at the start of module 5, using a volumetric screw feeder (K-Tron Soder KCL-KT20, Switzerland).

The extruder was left to function for 20–25 min before any sampling to ensure the stabilization of the operating conditions. Such conditions include feed flows of press cake, water and wheat straw, temperature and current feeding the motor. Upon achieving steady operation, the filtrate 2 (mixture of the hydrophobic phase, the hydrophilic phase and the foot) and the cake meal (insoluble phase) were immediately collected over a period of 20 min to avoid any variability of the outlet flow rates. Sample collection time was determined with a stopwatch. For each test, sample collection was carried out once. The filtrate 2 and the cake meal were weighed. The filtrate 2 was centrifuged (2000 × g, 10 min, 20 °C) to remove the foot. The supernatant was then treated by high-pressure homogenisation (300 bar, two cycles) (APV 1000, Denmark) to obtain smaller and more regular oil droplets in the oil-in-water emulsion (Mechling, 2002). The homogenate was centrifuged again (2000 × g, 10 min, 20 °C), to separate the hydrophobic and hydrophilic phases (Mechling, 2002).

The oil yield for the aqueous extraction stage was calculated according to the following formula:

$$R_{L2} = \frac{Q_{F2} \times T_{L2} \times L_{L2}}{Q'_{C1} \times L_{C1}} \times 100,$$

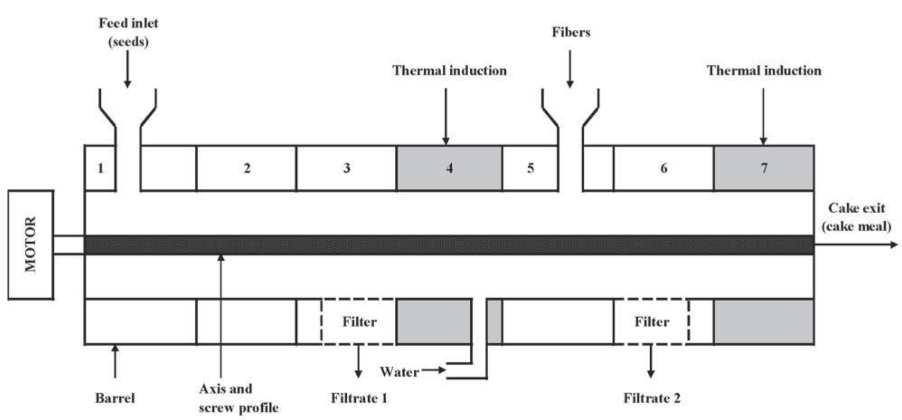


Barrel module	1	2	3	4	5	6	7

Profile 3																	
	T2F 66	C2F 50	C2F 33	DM 10×10 (45°)	C2F 33	C2F 25	BB 5×5 (90°)	C1F 33	C1F 25	BB 5×5 (90°)	C1F 33	C1F 33	C1F 33	C1F 25	CF1C -25	C1F 25	CF1C -25	C1F 25

Profile 4																	
	T2F 66	C2F 50	C2F 33	DM 10×10 (45°)	C2F 33	C2F 25	BB 5×5 (90°)	C1F 33	C1F 25	BB 5×5 (90°)	C1F 33	C1F 33	C1F 33	C1F 25	C1F 15	CF1C -15	C1F 25

Fig. 3 – Schematic modular barrel and screw configurations of the Clextral BC 45 twin-screw extruder used for aqueous extraction of residual oil from press cake A (profiles 3 and 4), and press cake B (profile 4) ($S_S = 48$ rpm, $\theta_c = 80\,°C$).



Barrel module	1	2	3	4	5	6	7

Profile 5																		
	T2F 66	C2F 50	C2F 33	DM 10×10 (45°)	C2F 25	BB 5×5 (90°)	C2F 33	C2F 25	C1F 15	CF1C -15	C1F 33	BB 5×5 (90°)	C1F 33	C1F 33	C1F 25	CF1C -25	C1F 25	CF1C -25	C1F 25

Fig. 4 – Schematic modular barrel and screw configuration of the Clextral BC 45 twin-screw extruder used for expression and aqueous extraction of oil from whole sunflower seeds in the same twin-screw extruder ($S_S = 70$ rpm, $\theta_c = 80\,°C$).

where R_{L2} is the oil yield in proportion to the oil that the press cake contains (%), Q'_{C1} the inlet flow rate of the press cake (kg/h), Q_{F2} the flow rate of the filtrate 2 (kg/h), T_{L2} the mass content of the hydrophobic phase (light phase) in the filtrate 2 (%), and L_{L2} the oil content in the hydrophobic phase of the filtrate 2 (%).

The energy consumed by the motor for the aqueous extraction stage was determined according to the following formula:

$$\mathrm{SME}_2 = \frac{P_2}{Q'_{C1} + Q_{LCR}},$$

where SME_2 is the specific mechanical energy consumed by the motor per unit weight of solid matters (press cake and fibers) (W h/kg), P_2 the electric power supplied by the motor (W), and Q_{LCR} the inlet flow rate of the lignocellulosic residue (kg/h).

2.3.2. Expression and aqueous extraction in the same twin-screw extruder

For this configuration, the expression stage and the aqueous extraction stage were realized in the same twin-screw extruder. Only two modules (modules 4 and 7) were heated to 80 °C and only one screw profile (profile 5) was tested in this study (Fig. 4). It was composed of two filtration zones (modules 3 and 6), both combined with a pressing zone (modules 4 and 7).

The expression stage was located in modules 2 and 3. It contained the seed trituration zone consisting of a succession of 10 monolobe paddles and 5 bilobe paddles, 5 cm apart. The angle between bilobe paddles was 90°. The filtrate 1 was collected in the first filtration zone (module 3).

The aqueous extraction stage was located at the end of module 4 and in module 5. Deionised water was injected at the end of module 4. Situated in module 5, a second series of 5 bilobe paddles allowed the intimate mixing of liquid and solid. To facilitate liquid/solid separation, wheat straw was introduced before the second pressing zone, at the end of module 5. The filtrate 2 was collected in the second filtration zone (module 6).

The extruder was left to function for 20–25 min before any sampling to ensure the stabilization of the operating conditions. Such conditions include feed flows of seeds, water and wheat straw, temperature and current feeding the motor. Upon achieving steady operation, the two filtrates (filtrate 1 and filtrate 2) and the cake meal were immediately collected over a period of 20 min to avoid any variability of the outlet flow rates. Sample collection time was determined with a stopwatch. For each test, sample collection was carried out once. The two filtrates and the cake meal were weighed. The filtrate 1 and the filtrate 2 were then treated as described earlier.

2.4. Analytical methods

The moisture contents were determined according to the French standard NF V 03-903. The oil content of the solids was determined according to the French standard NF V 03-908. The oil content of the hydrophobic phases was calculated after demulsification of the oil-in-water emulsions using a mix of ethanol and diethyl ether (3/1) (Mechling, 2002). The phospholipids content of the lipid extract from sunflower seeds was calculated according to the French standard NF T 60-227. The protein contents were determined according to the French standard NF V 18-100. An estimation of the three parietal constituents (cellulose, hemicelluloses, and lignins) contained in the solids was made by the ADF-NDF method of Van Soest and Wine (1967, 1968). All determinations were carried out in duplicate.

2.5. DSC analysis of sunflower press cakes

Differential Scanning Calorimetry (DSC) analysis was used to evaluate the denaturation level of proteins in sunflower press cakes (Rouilly et al., 2003). The study was performed on a PerkinElmer (USA) Pyris 1 power compensation calorimeter fitted with an intracooler cooling system. The purge gas used was nitrogen of analytical quality at a flow rate of 20 mL/min. Temperature and energy calibration was carried out with indium (T_f = 156.6 °C) and distilled water (T_f = 0 °C) before the beginning of the tests.

All analyses were performed with hermetic 60 μL stainless steel capsules fitted out with O-rings resistant to an internal pressure of 40 bar (PerkinElmer, USA). Reference cell was empty. They were carried out at a heating speed of 20 °C/min from 25 °C and stopped at 200 °C. Before analysis, sunflower seeds and press cakes were equilibrated in climatic chamber (60% RH, 25 °C) during three weeks. The sample mass was around 10 mg and all measurements were done in triplicate. Peak integration was realized with a sigmoid base line.

3. Results and discussion

3.1. Expression and aqueous extraction in two successive twin-screw extruders

3.1.1. Expression stage

Profile 2 required a higher screw rotation speed and twice more energy than profile 1 (Table 2) to produce the same flow rate of press cake. A second pressing zone was introduced, positioned in module 7, in order to increase the compression in the first reversed screws (Fig. 2). Pressing was therefore much more efficient even if no filtrate (filtrate 1′) was collected in module 6. The moisture content of press cakes A and B were 8% and 1% (Table 2), respectively. It confirmed the better pressing efficiency of profile 2, even if part of the moisture of press cake B was also lost via evaporation due to the higher barrel temperature (80 °C for press cake A, and 100 °C for press cake B). The oil content therefore decreased from 17% to 14% of the dry matter (Tables 1 and 2). The mass content of the foot in the filtrate 1 was also very high with profile 1 (around 30%), what is not acceptable for oil extraction. The expression stage had only a mechanical action, and then only the oil was released from the seeds. The other seed components (minerals, proteins, cellulose, hemicelluloses, and lignins) were not extracted, and they were concentrated in the press cakes (Table 1).

DSC measurements indicated that part of the proteins were denatured during the expression stage because their denaturation peak was partially eliminated in press cake A: 7.5 J/g of dry protein for the corresponding average enthalpy in press

Table 2 – Results of the expression experiments conducted with the Clextral BC 45 twin-screw extruder and using whole sunflower seeds for production of press cakes A and B.

	Press cake A	Press cake B
Operating conditions		
Screw profile	1	2
S_S (rpm)	60	105
Q_S (kg/h)	23.3	23.9
θ_c (°C)	80	100
Filtrate from expression stage (filtrate 1)		
Q_{F1} (kg/h)	9.3	8.6
T_{L1} (%)	70.4	93.2
T_{F1} (%)	29.6	6.8
Moisture (%)		
H_{F1}	5.72 ± 0.02	4.73 ± 0.56
Lipids (% of dry matter)		
L_{F1}	49.25 ± 0.51	55.33 ± 3.01
Press cake		
Q_{C1} (kg/h)	13.5	13.7
H_{C1} (%)	8.17 ± 0.05	1.22 ± 0.23
L_{C1} (% of dry matter)	17.19 ± 0.04	14.32 ± 0.10
Oil yields (%)		
R_{L1}	61.1	72.9
R_{C1}	80.1	82.4
Energy consumed		
I_1 (A)	55.0	71.0
P_1 (W)	2421.7	5437.4
SME_1 (W h/kg)	104.1	227.8

T_{F1} is the mass content of the foot in the filtrate 1 (%). H_{F1} is the moisture content in the foot of the filtrate 1 (%). L_{F1} is the oil content in the foot of the filtrate 1 (%). H_{C1} is the moisture content in the press cake (%).

cake A instead of 10.9 J/g of dry protein in the sunflower seeds (Table 3). In the case of profile 2 (press cake B), no denaturation peak was detected, which indicated that the mechanical action and the barrel temperature were strong enough to cause the complete denaturation of sunflower proteins.

3.1.2. *Aqueous extraction stage*

Oil extraction with water required the addition of fibers in the extruder to perform an efficient liquid/solid separation. Wheat straw was the lignocellulosic residue chosen due to its high cellulose and lignins contents and its low lipids and proteins contents (Table 1).

The press cakes had larger cellulose content than the seeds (Table 1), and then a higher hydrophilic character. Under the tested operating conditions, the inlet flow rate required for the water was approximately 30 kg/h for press cake A and 25 kg/h for press cake B to collect a filtrate. Below these limit values, no filtrate was recovered. At the same time, the inlet flow rate of the wheat straw had to remain below a limit value to avoid the clogging of the twin-screw extruder.

Trials presented were conducted at the allowed maximum value for the inlet flow rate of the wheat straw (Table 4). However, even if the addition of wheat straw improved the compression of the mixture of water and press cake in the reversed screws, it remained too low to achieve an efficient liquid/solid separation. Thus, the liquid/solid separation was not optimal, as revealed by the moisture contents of these press cakes, higher than when the aqueous extraction of the oil was conducted directly on the seeds (Evon et al., 2007). The best oil yield (R_{L2}) obtained with the twin-screw extrusion technology was particularly low, below 6% of the remaining oil in the press cake. The filtrate 2 contained 29–49% foots. These high values were linked to the small size of the solid particles in the press cakes, 384 μm for press cake A and 346 μm for press cake B, compared to the perforations of dishes in the filter section (1 mm in diameter).

Thus, the extraction stage allowed the extraction of only 1% of the oil contained initially in the seeds, in the best situation (Table 5; trial 6). This result was due to the difficulty to obtain a good compression of the mixture in the reversed screws, even for the higher inlet flow rate of the wheat straw, but also because part of the extract remained trapped in the porous structure of the cake meal.

The hydrophobic phases were richer in lipids (Table 4) than those produced by direct aqueous extraction of the oil from the seeds (Evon et al., 2007). As the proteins content was lower, the stability of these oil-in-water emulsions was reduced, especially when denaturation of proteins was complete during the expression stage (press cake B).

On the contrary, free oil from the expression stage was preferred over stable emulsions, and it was easier to separate free oil from the filtrate 1 than to break the emulsion to free the oil extracted with water at the level of the extraction stage.

3.2. *Expression and aqueous extraction in the same twin-screw extruder*

Water and wheat straw could be introduced in the twin-screw extruder only under certain limit values. When the inlet flow rate of the water was above 45 kg/h, water went back up to

Table 3 – Average temperature, average enthalpy reported to the sample mass, to the mass of dry matter and to the mass of dry protein (Table 1) of the denaturation peak observed on DSC scans of sunflower seeds and press cakes A and B, in pressure resistant pans.

	Sunflower seeds	Press cake A	Press cake B
Moisture content (%)	6.34 ± 0.03	7.61 ± 0.01	6.33 ± 0.16
Peak temperature (°C)	149.9 ± 1.0	152.2 ± 0.6	No peak
Enthalpy (J/g of sample)	1.60 ± 0.02	1.63 ± 0.02	0.00
Enthalpy (J/g of dry matter)	1.71 ± 0.02	1.76 ± 0.03	0.00
Enthalpy (J/g of dry protein)	10.88 ± 0.15	7.46 ± 0.11	0.00

Table 4 – Results of the aqueous extraction experiments conducted with the Clextral BC 45 twin-screw extruder and using press cakes A and B (S_S = 48 rpm, θ_c = 80 °C).

	Trial					
	1	2	3	4	5	6
Operating conditions						
Screw profile	3	3	4	4	4	4
Press cake	A	A	A	B	B	B
Q'_{C1} (kg/h)	16.6	16.5	16.8	15.4	14.8	14.2
Q_W (kg/h)	35.2	35.2	35.2	36.1	24.6	35.5
Q_{LCR} (kg/h)	3.0	3.6	2.5	3.0	3.4	3.7
C_F (kg/h rpm)	0.41	0.42	0.40	0.38	0.38	0.37
Filtrate from aqueous extraction stage (filtrate 2)						
Q_{F2} (kg/h)	7.4	8.3	4.7	7.8	1.7	10.5
T_{L2} (%)	1.6	1.3	1.4	1.8	2.3	1.9
T_{H2} (%)	65.0	64.8	62.6	69.6	48.4	68.9
T_{F2} (%)	33.4	33.9	36.0	28.6	49.3	29.2
Moisture (%)						
H_{L2}	37.60 ± 1.03	38.37 ± 0.32	32.25 ± 0.29	40.89 ± 0.87	36.05 ± 0.99	40.13 ± 0.45
H_{H2}	95.35 ± 0.03	95.22 ± 0.01	95.30 ± 0.04	95.64 ± 0.05	93.45 ± 0.02	95.38 ± 0.01
H_{F2}	71.28 ± 0.16	71.73 ± 0.03	72.00 ± 0.20	73.61 ± 0.31	68.12 ± 0.16	74.19 ± 0.39
Lipids (% of dry matter)						
L_{L2}	95.73 ± 1.27	95.15 ± 1.84	93.86 ± 0.06	98.12 ± 0.25	96.29 ± 0.57	98.58 ± 0.32
L_{F2}	14.91 ± 0.13	15.68 ± 0.09	14.43 ± 0.12	13.16 ± 0.06	18.09 ± 0.14	18.50 ± 0.10
Cake meal (insoluble phase)						
Q_{C2} (kg/h)	47.3	47.0	49.9	46.7	41.1	42.9
H_{C2} (%)	64.07 ± 0.02	62.98 ± 0.12	65.59 ± 0.20	63.22 ± 0.44	57.49 ± 0.42	62.11 ± 0.33
L_{C2} (% of dry matter)	16.18 ± 0.10	14.83 ± 0.07	15.98 ± 0.59	13.18 ± 0.21	11.50 ± 0.43	9.47 ± 0.10
Oil yield (%)						
R_{L2}	2.7	2.3	1.5	3.7	1.1	5.9
Energy consumed						
I_2 (A)	8.0	9.0	6.0	7.0	9.0	10.0
P_2 (W)	291.3	308.8	218.5	244.7	326.3	337.9
SME_2 (W h/kg)	14.9	15.4	11.3	13.2	17.9	18.9

Q_W is the inlet flow rate of the water (kg/h). C_F is the device's filling coefficient (kg/h rpm); it is defined as the ratio of the inlet flow rate of the solid matters ($Q'_{C1} + Q_{LCR}$) to the screw rotation speed (S_S). T_{H2} and T_{F2} are the mass contents of the hydrophilic phase (heavy phase) and the foot in the filtrate 2 (%), respectively. H_{L2}, H_{H2} and H_{F2} are the moisture contents in the hydrophobic phase, the hydrophilic phase and the foot of the filtrate 2 (%), respectively. L_{F2} is the oil content in the foot of the filtrate 2 (%). Q_{C2} is the flow rate of the cake meal (kg/h). H_{C2} is the moisture content in the cake meal (%). L_{C2} is the oil content in the cake meal (%). I_2 is the current feeding the motor (A).

Table 5 – Overall results of the expression and aqueous extraction experiments conducted with two successive Clextral BC 45 twin-screw extruders and using whole sunflower seeds.

Press cake	Trial					
	1 A	2 A	3 A	4 B	5 B	6 B
Oil yields in proportion to the oil that the seed contains (%)						
R_{L1}	61.1	61.1	61.1	72.9	72.9	72.9
R_{L2c}	0.5	0.5	0.3	0.7	0.2	1.0
R_{TL}	61.6	61.6	61.4	73.6	73.1	74.0
Total oil yield based on the residual oil content of the cake meal (%)						
R_{TC}	79.1	80.2	79.4	81.7	83.1	86.5
SME (W h/kg)	116.6	117.2	113.4	239.7	244.5	246.1

R_{L2c} is the oil yield for the aqueous extraction stage in proportion to the oil that the seed contains (%). R_{TL} is the total oil yield in proportion to the oil that the seed contains (%). R_{TC} is the total oil yield based on the residual oil content of the cake meal (%). SME is the total specific mechanical energy consumed by the motors of the two successive twin-screw extruders per unit weight of sunflower seeds (W h/kg).

the fibers feeder. At the same time, when the inlet flow rate of the wheat straw was above 3 kg/h, it caused the clogging of the twin-screw extruder. The three trials performed were situated within the working limits of the extruder. The oil yield for the expression stage (R_{L1}) ranged from 65% to 76% (Table 6). Aqueous extraction was therefore realized on a material partially deoiled.

The oil yield (R_{L1}) increased with the inlet flow rate of the water (Table 6; trials 7 and 8). The water, introduced immediately downstream from the first reversed screws, would act as an additional barrier to the material transport, increasing the efficiency of its compression in the first reversed screws.

Nevertheless, the action of the water was not only mechanical, at the expression stage. Water was also considered as a solvent in the aqueous extraction stage. The corresponding oil yield (R_{L2}) was higher (Table 6) when the aqueous extraction was conducted in the same extruder, and could reach 4% of the oil contained initially in the seeds (14% of the remaining oil in the solid after the expression stage) in the case of trial 9. For this trial, the inlet flow rate of the whole sunflower seeds was lower, decreasing the device's filling coefficient. Thus, not only the compression of the material in the first reversed screws was less efficient, but also the oil yield for the expression stage (R_{L1}). But, as the inlet flow rate of the seeds was lower, the ratio of the water to the oil was higher in the aqueous extraction stage.

This result showed that a main limitation of the aqueous extraction came from the low value of the ratio of the water to the residual oil (20:1 in the case of trial 9). Two phenomena limited this ratio: the inlet flow rate of the water in one side, and the filling of the extruder by the seeds in the other side. This was confirmed by the aqueous extraction of the

Table 6 – Results of the expression and aqueous extraction experiments conducted with the same Clextral BC 45 twin-screw extruder and using whole sunflower seeds (profile 5, S_S = 70 rpm, θ_C = 80 °C).

	Trial		
	7	8	9
Operating conditions			
Q_S (kg/h)	14.0	13.7	8.9
Q_W (kg/h)	20.8	26.6	25.4
Q_{LCR} (kg/h)	1.8	1.8	1.9
C_F (kg/h rpm)	0.23	0.22	0.15
Filtrate from expression stage (filtrate 1)			
Q_{F1} (kg/h)	5.0	5.3	2.9
T_{L1} (%)	90.4	92.0	89.4
T_{F1} (%)	9.6	8.0	10.6
H_{F1} (%)	5.58 ± 0.21	11.96 ± 0.35	5.58 ± 0.19
L_{F1} (% of dry matter)	52.68 ± 0.67	30.17 ± 0.41	65.51 ± 0.59
Filtrate from aqueous extraction stage (filtrate 2)			
Q_{F2} (kg/h)	4.7	11.2	20.4
T_{L2} (%)	2.9	1.2	1.3
T_{H2} (%)	73.2	78.9	80.9
T_{F2} (%)	23.9	19.9	17.8
Moisture (%)			
H_{L2}	26.13 ± 0.19	30.17 ± 0.23	31.09 ± 0.27
H_{H2}	95.33 ± 0.02	96.32 ± 0.02	97.01 ± 0.02
H_{F2}	72.43 ± 0.11	75.94 ± 0.14	75.67 ± 0.12
Lipids (% of dry matter)			
L_{L2}	98.14 ± 0.30	97.20 ± 0.82	97.20 ± 0.57
L_{F2}	10.03 ± 0.08	10.61 ± 0.07	6.05 ± 0.10
Cake meal (insoluble phase)			
Q_C (kg/h)	27.0	25.6	12.9
H_C (%)	66.30 ± 0.06	68.04 ± 0.66	57.03 ± 0.11
L_C (% of dry matter)	9.10 ± 0.16	10.05 ± 0.28	5.67 ± 0.08
Oil yields in proportion to the oil that the seed contains (%)			
R_{L1}	70.6	76.5	64.5
R_{L2}	1.5	1.5	4.4
R_{TL}	72.1	78.0	68.9
Total oil yield based on the residual oil content of the cake meal (%)			
R_{TC}	87.2	87.0	92.3
Energy consumed			
I (A)	47.0	45.0	52.0
P (W)	2396.2	2294.3	2651.1
SME (W h/kg)	151.3	148.2	245.5

residual oil from press cakes A and B in batch reactor using a 1-L blender (Waring, USA). The two press cakes (75 g) and 425 g of deionised water were blended for 5 min. The slurry was centrifuged (2000 × g, 10 min, 20 °C) to remove the insoluble phase. The corresponding oil yields were 19% and 41%, respectively, with higher ratios of the water to the residual oil (36:1 in the case of press cake A, 40:1 in the case of press cake B).

When the inlet flow rate of the whole sunflower seeds was too low (Table 6; trial 9), even if the aqueous extraction stage was more efficient, the total oil yield remained low (69% for R_{TL}) with a high energy consumption. The high mechanical action led to the partial physical destruction (lysis) of the cells, and the solid particles were partially recovered as foots in the two filtrates (filtrate 1 and filtrate 2). After removing 70% of the oil and losing around 30% of the solids as foots, the residual oil content of the cake meal was low, about 6% of its dry matter. This residual oil had been significantly diluted in the cake meal by the wheat straw. Taking into account for this dilution, the real residual oil content of the cake meal (without wheat straw) would be significantly higher (close to 9% of its dry matter).

The configuration combining the expression and the aqueous extraction in the same twin-screw extruder was therefore beneficial for the overall effectiveness of the process. The total oil yield (R_{TL}) was as high as 80% of the oil contained initially in the seeds, and the residual oil contents of the corresponding final cakes (Table 6) were the lowest obtained in this study. The oil yield was better than when the aqueous extraction of the oil was conducted directly on the seeds (Evon et al., 2007) or even when the expression stage and the aqueous extraction stage were conducted in two successive extruders (Table 5).

4. Conclusion

The aqueous extraction of the residual oil from sunflower press cakes was carried out using the twin-screw extrusion technology. However, the introduction of wheat straw used as a lignocellulosic residue upstream from the filtration module was essential to enable the liquid/solid separation.

Higher oil yield was obtained when the expression and the aqueous extraction were conducted in the same twin-screw extruder, and the residual oil content of the cake meal was only 6% of dry weight in the best run. Nevertheless, in the two tested configurations, the contribution of the aqueous extraction stage for the total oil yield was limited (less than 5% of the lipids contained initially in the seeds). During the aqueous extraction process, the oil was extracted in the form of oil-in-water emulsions. The presence at their interface of natural surface-active agents co-extracted during the process, the phospholipids and the proteins, was observed. Nevertheless, these hydrophobic phases had a low stability over time due to their proteins contents particularly low.

These modest results were due to the insufficient particle size reduction (persistence of the structure of cotyledon cell walls within the seeds) and to the low ratio of the water to the press cake, but also to the thermo-mechanical denaturation of the proteins caused by the expression stage. Another factor in the incomplete aqueous extraction of the residual oil from the press cakes was the technological limits of the twin-screw extruder that did not enable a full separation of liquid and solid phases, even with the maximum inlet flow rate of the wheat straw.

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