A Twin-Screw Extruder for Oil Extraction: I. Direct Expression of Oleic Sunflower Seeds[1]

Corinne Dufaure, Juliette Leyris, Luc Rigal, and Zéphirin Mouloungui*

Laboratoire de Chimie Agro-Industrielle, U.A. INRA 31A1010, École Nationale Supérieure de Chimie de Toulouse, 31077 Toulouse Cedex 04, France

ABSTRACT: Lipids are traditionally removed from seeds by mechanical crushing and solvent extraction. During the mechanical crushing process the oilseed is cleaned, cracked, flaked, and cooked before entering a mechanical screw press. Seventy-five percent of the oil of sunflower seeds can be extracted by crushing, and the fatty cake then contains about 15% of oil. The oil levels remaining in the cake can be reduced to less than 2% by solvent extraction. However, the crude oil has to be refined as it contains many impurities and approximately 600 ppm phosphorus. A new process, in which sunflower seeds are pressed in a twin-screw extruder, is examined here. The screw profile was first optimized. Oleic sunflower seeds were crushed and 80% of the oil was removed. The resultant oil was of good quality, with acid numbers below 2 mg KOH/g of oil and total phosphorus contents of about 100 ppm. The influence of pressing temperature and of fresh seed moisture content was determined. High pressing temperature and low moisture content improved oil extraction. The quality of the meal was examined through the solubilization of its proteins in alkaline water at 50°C. The fatty meal proteins remained quite soluble, and therefore one can assume that they were still relatively close to their native conformation. The pressing of oleaginous material in a twin-screw extruder provides a new option to traditional processes.

Paper no. J9100 in *JAOCS 76,* 1073–1079 (September 1999).

KEY WORDS: Meal quality, oil expression, oil quality, oleaginous seeds, oleic sunflower oil, protein quality, twin-screw extruder.

This study is a follow-up to our work on unconventional uses of whole oleic sunflower seeds. We have already shown that whole oleic sunflower seed provides a suitable substrate for the *in situ* transformation of the lipid fraction into fatty alkyl esters used as lubricants in a thermo-mechanico-chemical twin-screw system (1,2). In this article, we report the use of the twin-screw system in a new oil extraction process. Current procedures for the industrial extraction of these oil seeds involve a two-step process of mechanical pressing followed by extraction with hexane. The mechanical pressing of oleaginous seeds is mainly carried out in a continuous single-screw press. This type of machine consists of a barrel with a single endless screw and a filtration element. The pressure on the contents of the barrel can be regulated by means of a constriction at the machine outlet. This category of machine includes single-screw presses, used to produce first-pressing oil, and expanders, which are simplified single-screw extruders fitted for direct steam injection. The latter are used to pretreat certain oleaginous seeds (notably soybean and cotton) prior to solvent extraction (3,4). The matter is extruded at the system outlet as steam-expanded pellets. These have a much higher permeability than the more typical flaked seeds whence the improved efficacy of solvent diffusion and the higher extraction yields (5). No direct oil extraction actually occurs within the expander, however, as there is no filtration system along the length of the barrel. This precludes the treatment of oil-rich matter such as rape and sunflower seeds as oil accumulates in the barrel and expansion of the matter at the machine outlet becomes impossible.

Manufacturers (Anderson International Corp., Cleveland, OH) have proposed expanders fitted with a deoiling cage, but the results are not convincing and the trials have apparently not been continued (6). Certain modern single-screw presses have two different stages (6). The claimed yields are high with only 3 to 5 % of the oil remaining in the cake at the end of pressing, but the type of seed is not indicated. In these industrial processes, several costly pretreatments of the raw material preceded single-screw pressing. In the case of sunflower seed these consist of drying to reduce the seed moisture content to 5-6%, possible dehulling, flaking on cylinders, and cooking. This final step allows adjustment of the moisture content of the resultant flakes to 3–5%, increases oil fluidity and thereby facilitates outflow from the cake, continues rupture of the seed cells and coagulates the proteins, thus reducing the number of particles carried over with the oil during filtration.

The oil collected during pressing contains solid particles in suspension that have passed through the filter and are known as the "foot." This is eliminated during clarification (by decantation, filtration, or centrifugation) to give an oil impurity content of about 0.1%. The oil can then be dried. A virgin-type oleic sunflower oil typically has an acid value (AV) below 2 mg KOH/g oil, an iodine value (IV) of 80 to 90 g of iodine per 100 g of oil, and a phosphorus content of approxi-

[1]Presented as an oral communication at the 2nd American Oil Chemists' Society Europe Symposium, October 1–4, 1998, at Cagliari, Italy.

*To whom correspondence should be addressed at Laboratoire de Chimie Agro-Industrielle, U.A. INRA 31A1010, École Nationale Supérieure de Chimie de Toulouse, 118 route de Narbonne, 31077 Toulouse Cedex 04, France. E-mail: lcacatar@cict.fr

mately 200 ppm (2). If the oil obtained by mechanical pressing is of good quality, the extraction yields are limited to approximately 70 to 80%. These yields can be increased to 98% by a supplementary extraction with hexane, but the resulting crude oil contains many impurities (AV of 6 to 8 mg KOH/g of oil and phosphorus content of about 600 ppm) and requires thorough refining before further use (7).

Twin-screw machines provide an alternative to traditional procedures but have until now received little study. The principle of twin-screw technology is to subject the plant matter to intense mechanical action provided by two conveying screws or counter-screws turning within a fixed barrel that is locally heated or cooled. The processing of the matter is thus extremely thorogh. Twin-screw presses exist but are little used. Isobe *et al.* (8) recently extracted oil from dehulled and whole sunflower seeds in a contra-rotating twin-screw press. The expression yield from whole seeds was low (75%) but could be increased to 93.6% if the raw material was dehulled. These authors recommended using this technique to press dehulled oleaginous seeds because the absence of hull fibers would not allow the use of a traditional one-screw press. In this case, the matter is very oily and a twin-counter screw machine must be used for conveying inside the barrel.

In 1994, Guyomard (9) tested oil extraction from dehulled rapeseeds in a twin-screw extruder. The machine was fitted with filter elements and a frontal plate with insert, as in the conventional single-screw press system. The resulting residual oil contents in the cake exceeded 25% on average. When a module containing two co-rotating but separate screws in a filtration barrel was added (so that they functioned as two single screws in a conventional press), extraction yields of 75 to 80% were obtained with rapeseed. However, no details were given about the quality of the extracted oil, which would be essential to subsequent application and to determine the degree of refining required. Most available information in fact relates to dehulled rapeseed, the rheological behavior of which is very different from whole sunflower seed owing to the different oil, protein, and fiber contents.

Because of the recent interest in oleic sunflower seeds as a crop, we investigated the possibility of pressing such seeds. Our aim, in the case of whole oleic sunflower seed, was to show that the twin-screw extruder could be used as a twin-screw press and oil separator with greater efficacy than the single-screw press. Our investigations were focused on an examination of the principal factors influencing oil expression yield and oil quality, i.e., barrel configuration and screw profile, seed input flow rate, screw rotation speed, composition of the raw material, and pressing temperature. We also examined the fatty meal quality. Oil expression using a twin-screw machine seems to provide a promising alternative to the oil expression processes currently in use.

EXPERIMENTAL PROCEDURES

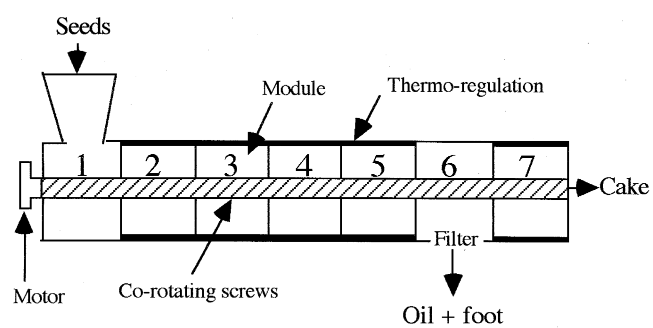
Twin-screw extruder design. Oil expression was carried out using two twin-screw extruders BC21 and BC45 made by Clextral (Firminy, France), involving co-rotation screws. A BC21-type extruder was used to determine the optimal profile for the operation (Scheme 1). The machine was operated by the Terminal Operator Intouch version 1.00 software (Clextral) to determine the parameters controlling the twin-screw extruder function (input flow rate, torque) and the temperatures of the different barrel modules. The barrel consisted of 7 modules 10 cm in length, and the input of whole seeds into the first module was controlled by a scale of LWF-D55 type (Ktron, Niederlenz, Switzerland). A filtration module was inserted at position 6, the filters consisting of four hemispherical dishes with perforations 1 mm in diameter, with an open outlet on the seventh barrel module (no frontal plate). Modules 2, 3, 4, 5, and 7 were heat-regulated with heating and cooling systems.

The profiles of the screws used in the BC21 twin-screw extruder consisted of trapezoidal double-thread screws to ensure efficient transport; monolobed paddle screws (designated DM), which exerted considerable pressure on the matter on the sides of the barrel; and bilobed paddle screws (BB), which had a pronounced shearing effect (Scheme 2). The presence of a reverse screw, or screw elements carrying the contents in the opposite direction, immediately beneath the filtration module guaranteed the formation of a so-called "dynamic" plug. The different screw profiles tested are presented in Figure 1. The nature of the screw is indicated together with the pitch and the mounting angle of the BB and the DM.

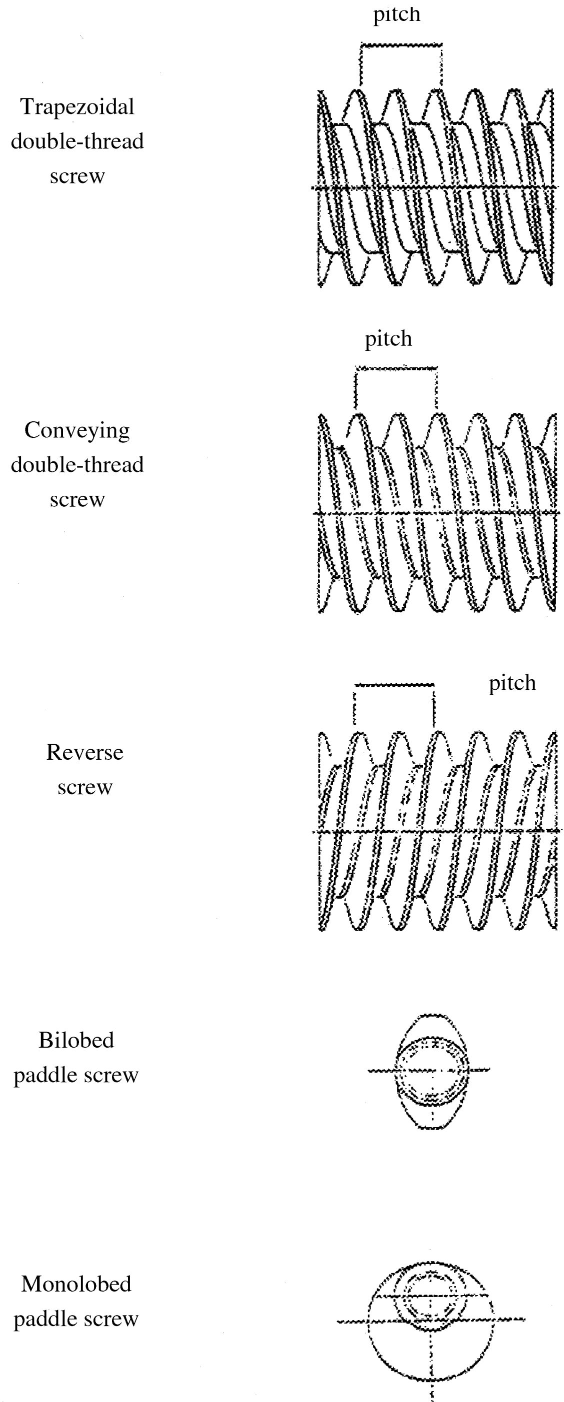
The configuration of the BC45 extruder was the same as for the previously described BC21 machine. It was fed with a type 40 volumic screw feeder (Clextral). It consisted of seven modules, each 20 cm in length. The perforations in the filter element at position 6 were 3 mm in diameter. The plant was piloted from a control cabinet which also recorded the operating parameters for the twin-screw (amperage) and the temperatures of the different barrel modules. The screw profile used is described in Figure 2.

Raw materials and chemicals. The sunflower seeds had a high oleic acid content (variety Olbaril, harvested 1995 and 1996, La Toulousaine de Céréales, St Orens, France), ranging from 88 to 90% [NFT 60-23 and NFT 60-223 (10)], and a protein content of approximately 18% [NF V18-100 (10)].

The oil content of the seeds used in experiment 1, expressed in relation to the dry matter content of uncleaned seed



SCHEME 1



SCHEME 2

(NF V03-905) was 52%. The dry matter content at storage was 93.7% (NF V03-903).

The oil content of the oleic sunflower seeds used in the second set of tests was 50.9% in relation to the dry matter. The seed moisture content was modified by oven-drying at 105°C, the duration of drying depending on the required dry matter content (93.8 to 97.6% dry matter).

The seeds were not flaked or rolled prior to crushing. All the solvents and other chemicals were of analytical grade.

Operating conditions. A number of seed input flow rates and screw rotation speeds were tested to determine the optimal screw profile (experiment 1). The temperature along the barrel was fixed at 80°C. Samples were taken when the machine had been operating for 5–10 min.

In experiment 2 the seed input flow rate was fixed at 22 kg/h with a screw rotation speed of 150 rpm. A preliminary trial had shown that it was impossible to express oil from the seed below 60°C, so the selected test temperatures ranged from 80 to 120°C. Apart from the choice of temperature exceeding 100°C so as to dry the oil at the machine outlet, these temperature limits were based on information reported in the literature and related to oleaginous seed processing prior to oil extraction in a single-screw press and solvent extraction (5,6). The choice of the lower limit was based on the fact that a temperature of 60–70°C is generally required to coagulate the membrane proteins of oleaginous seeds. This coagulation rigidifies the flexible cell walls that contain the lipid droplets and facilitates their rupture on crushing. Samples of the filtrate (oil containing the foot) and the remaining fatty cake were taken when the machine had been operating for approximately 20–25 min to ensure a stable flow rate and temperature.

Sample analysis. The filtrate was centrifuged to separate the foot from the liquid oil, which consisted of at least 99% triglycerides. The response investigated in both types of tests was the oil extraction yield obtained after centrifugation to remove the solid particles carried over. This was calculated from the relationship:

$$R\ (\%\ \text{mass}) = 100\left(Q_F \cdot T_F\right)/\left[\left(Q_F + Q_S\right) \cdot T_G\right] \qquad [1]$$

where Q_F is the outlet flow rate of the filtrate (oil + foot) in kg/h, T_F is the oil content of the filtrate as a percentage (determined by removing the foot by centrifugation), Q_S is the outlet flow rate of the solid (cake) in kg/h, and T_G is the oil content of the seed as a percentage. The dry matter and residual oil content of the cake were measured according to standards NF V03-921 and NF V03-924.

The quality parameters of a crude oil were determined in the second set of experiments (7) and included (i) the AV, expressed in mg of KOH/g of oil (standard NFT 60-204) which indicates the free fatty acid content of the oil. This measurement was obtained after extraction (AV1) then after 11 mon of storage in a cold room (4°C) away from air and light to evaluate oil stability (AV2). (ii) The IV, expressed in g of iodine fixed by 100 g of lipid (standard NF T 60-203), which permits quantification of the unsaturated oil components; the higher the IV the greater the unsaturation of the oil. (iii) The phosphorus content (P) expressed in mg of phosphorus per kg of oil [standard NF T60-227 (10): ashing of the sample followed by the spectrophotometric measurement of phosphorus as a yellow phosphovanadomolybdic complex] which represents mainly the carryover of phospholipids during the seed-pressing process.

The quality of the meal has been examined through the solubilization of its proteins in basic water at 50°C. The pH was adjusted to 12 with 1 N sodium hydroxide before introducing

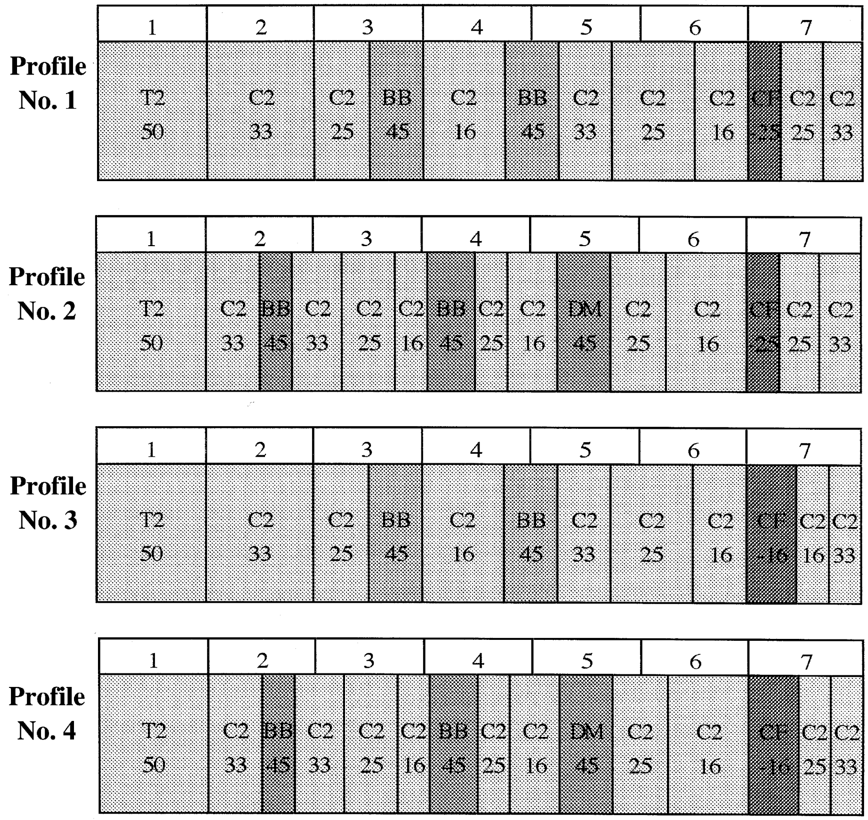


FIG. 1. Screw profiles tested for the expression of whole sunflower seeds in a BC21 twin-screw extruder. T2, trapezoidal double-thread screw; C2, conveying screw; BB, bilobed paddle screw; DM, monolobed paddle screw; CF, reverse screw. The numbers written under the nature of the screw indicate the pitch for T2, C2, and CF screws and the mounting angle of the BB and DM screws.

the cake in the batch reactor. The mass ratio solid/liquid was 1:20, and the rotation speed of the mechanical stirrer was 500 rpm. After 15 min of stirring, the liquid phase and the solid phase were separated by centrifugation (5 min, 1000 rpm). These operating conditions have been optimized for the extraction of industrial hexane-extracted meal so that we could compare the solubilization of the proteins of the fatty meal and of an industrial hexane-extracted meal (11). The total nitrogen content of each phase was measured according to the standard NF V18-100 (ashing of the sample in the presence of sulfuric acid and a metallic catalyst and titration of the liberated ammonia). The total nitrogen content is multiplied by 6.25 to evaluate the protein content of the samples.

RESULTS AND DISCUSSION

Stabilization of the machine. Only cake is extruded during the initial phase of operation of the twin-screw machine, and there is no filtration. This is followed by evacuation through the filter of a heavily loaded oil. A steady regime is attained after 5–10 min in the case of the BC21 extruder and after 20–25 min with the BC45 extruder. This is characterized by the evacuation from the filtration module of an oil with a very low content of solid particles, and a fatty cake.

The residence time of the matter in the twin-screw machine (time between input and output) was approximately 50 to 60 s in all the tests, as determined by introducing a small

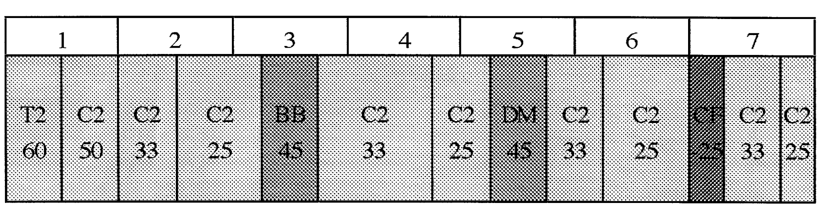


FIG. 2. Screw profile used for optimization of oil expression from sunflower seeds in a BC45 twin-screw extruder. For abbreviations and explanations of numbers written under the nature of the screw see Figure 1.

TABLE 1
Yields Obtained for Different Screw Profiles Used in a Twin-Screw BC21 Extruder for the Expression of Whole Sunflower Seeds

	Operating conditions[a]				
Profile[b]	Q_l = 14 kg/h Ss = 200 rpm T = 80°C	Q_l = 14 kg/h Ss = 150 rpm T = 80°C	Q_l = 14 kg/h Ss = 125 rpm T = 80°C	Q_l = 10 kg/h Ss = 125 rpm T = 80°C	Q_l = 16 kg/h Ss = 125 rpm T = 80°C
No. 1	17.3%	46.0%	68.6%	32.6%	69.2%
No. 2	32.6%	54.1%	63.1%	43.4%	/[c]
No. 3	55.1%	59.8%	75.4%	66.9%	Rem.
No. 4	62.6%	70.3%	81.6%	71.4%	63.9%

[a]Ss, screw rotation speed; Q_l, seed input flow rate; Rem., upward movement of the seeds through the feeder module.
[b]Profiles are given in Figure 1.
[c]Trial not done.

amount of seed stained with erythrosine and monitoring the resulting color of the filtrate and cake.

Experiment 1: Determination of an optimal screw profile. Oil yields exceeding 80% were attained (Table 1) with a high seed input flow rate (14 kg/h), in view of the size of the BC21 machine (21 mm between the screw axes). Minimal and maximal thresholds beyond which seed pressing was ineffective were apparent for each screw profile (Figure 3).

When the seed input flow rate was too slow the machine did not fill up, the "dynamic" plug was unable to form around the reverse screw and the pressure exerted on the triturated matter was therefore insufficient to express the oil from the fibrous matrix and evacuate it through the filter. All the crushed seeds were instead evacuated at the open end of the barrel. When the seed input flow rate was too high, the machine clogged and the contents were forced back through the feeder on the first module. An increase in the maximal threshold of seed input flow rate could be obtained by increasing the screw rotation speed but this also increased the minimal threshold as the contents were more rapidly evacuated from the machine.

Within the zone of seed pressing efficacy, the fourth profile produced the best yields (Table 1). The bilobed paddle screws (BB) mounted at an angle of 45° helped to cut up the matter and rupture the cells by shearing. The addition of monolobed paddle screws (DM) in module 5 just above the filtration module completed the effect of the bilobed paddle screws and facilitated diffusion of the lipid droplets released through the fibrous matrix toward the surface of the matter by radial compression on the barrel. Given identical lengths and pitch of the reverse screw, this produced an increase in the separated oil yield (profile 1 vs. profile 2, profile 3 vs. profile 4). Increasing the length of the reverse screw and reducing its pitch helped to compact the "dynamic" plug and increase the effect of counterpressure below the filtration module. This also resulted in higher oil extraction yields (profile 1 vs. profile 3, profile 2 vs. profile 4). This zone of compression was associated with an increase in temperature (5–10°C) due to friction.

When the seed input flow rate was kept constant, a reduction in the screw rotation speed increased the duration of passage of the matter through the twin-screw machine and clearly increased the oil expression yield, whatever the profile under consideration (Fig. 4). Note that there was a limit to using the twin-screw extruder as a press at this screw rotation speed (125 rpm) and temperature (80°C), as an increase of 2 kg/h in the seed input flow rate (i.e., an input of 18 kg/h) clogged the machine.

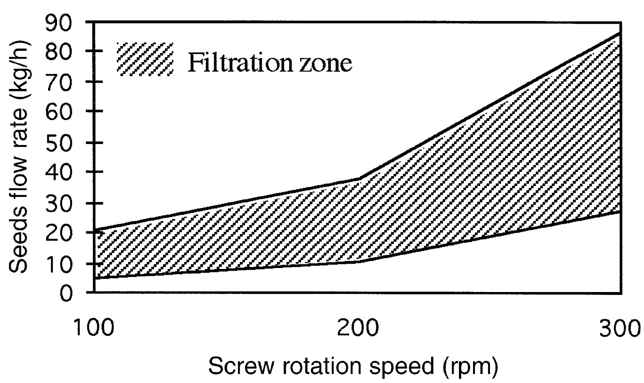


FIG. 3. Influence of screw rotation speed and seed input flow rate on the expression of sunflower seeds in a twin-screw extruder. BC21 twin-screw extruder, screw profile no. 1, temperature 80°C.

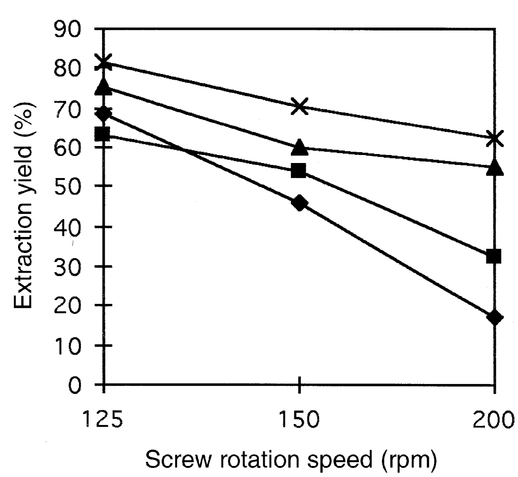


FIG. 4. Influence of screw rotation speed on the expression of sunflower oil in a twin-screw extruder. BC21 twin-screw extruder, temperature 80°C. ◆ Profile no. 1; ■ profile no. 2; ▲ profile no. 3; × profile no. 4.

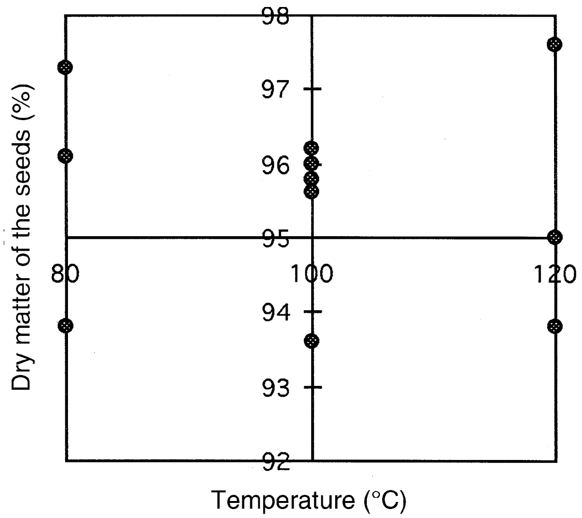


FIG. 5. Distribution of experimental points for the optimization of oil expression from whole sunflower seeds in a BC45 twin-screw extruder (points permitting or not permitting filtration).

Experiment 2: Influence of pressing conditions. The influence of the moisture content of the raw material and pressing temperature on the expression yield of oleic sunflower seed and on the quality of the resulting oil was investigated. The experimental points are presented in Figure 5. Some points did not permit separation of the oil at the filtration module, and this precluded any statistical treatment as isoresponses curves of the results obtained (extraction yield, foot content of the filtrate, oil content of the cake, AV, IV, and P content of the oil filtered) (Table 2) because these points induced discontinuity zones.

The expression yields of whole sunflower seeds attained 80%. They were highest with a high pressing temperature and natural moisture content of the seed. Increasing the temperature had a favorable effect on oil fluidity. The reduced effort required by the motor to ensure pressing was apparent from the lower energy consumed, which dropped from 30 A at 80°C to 26 A at 120°C.

Drying the seeds was found to increase friability to the detriment of elasticity. The resulting texture of the mixture obtained from the paddle screw zone of trituration made formation of the "dynamic plug" more difficult and reduced pressing efficacy. This was also apparent in the formation of a greater proportion of fine particles. These were carried toward the filter and would explain the increased amount of foot in the oil which, in the worst case (100°C and 96% dry matter), was increased from 10 to 25%. The very high proportion of solid matter in the oil was also apparent as a greater loss of oil on centrifugation of the filtrate, which would explain why the oil yields remained limited to 70% in this latter case, even though the percentage of oil in the cake was very low (15 to 16%).

A temperature of 120°C associated with partial drying of the seed (95% in relation to dry matter) seems to provide a good compromise for high oil yield (79%), a filtrate foot content limited to 14%, and a oil residue content of the fatty cake of less than 15%.

Oil quality was apparently satisfactory as the P content was limited to 100 mg/kg and the AV remained stable at less than 2 mg KOH/g oil. The observed variations in AV and IV were not very significant. The variations in P content were apparently more significant (Fig. 6). Thus, pressing of undried seed (dry matter contents of 93.6 to 93.8%) was always associated with low P contents in the expressed oil and a very low percentage of foot in the filtrate (10.4%) whatever the pressing temperature. At 120°C the P content of the oil increased with the dry matter of the seed (Fig. 6), along with the foot. High P values were associated with a high foot content of the filtrate. Drying the seeds therefore resulted in more effective crushing of the seed due to its loss of elasticity. This intense crushing pulverizes the cell walls and leads to more efficient coextraction of the membrane phospholipids.

TABLE 2
Effect of Temperature and Moisture on Oil Yield and Quality During the Expression of Oleic Sunflower Seeds in a BC45 Twin-Screw Extruder[a]

Temperature (°C)	Dry matter of seeds (%)	Extraction yield (%)	Foot content of the filtrate (%)	Oil content of the cake (%)	Acid value AV1 (mg KOH/g)	Acid value AV2 (mg KOH/g)	Iodine value (mg iodine for 100 g of oil)	Phosphorus content (mg/kg)	Intensity (A)
80	93.8	62	10.4	28.8	1.65	1.83	90.3	65	30
80	96.1	n.f.	n.f.	53.2	n.f.	n.f.	n.f.	n.f.	10
80	97.3	n.f.	n.f.	47.7	n.f.	n.f.	n.f.	n.f.	11
100	93.6	66	10.4	25.9	1.56	1.74	89.5	48	30
100	95.6	71	27	15.0	1.88	2.00	82.8	146	29
100	95.8	67	26	16.8	1.81	1.90	83.0	181	27
100	96.0	69	24	15.6	1.81	1.85	81.9	101	27
100	96.2	n.f.	n.f.	51.8	n.f.	n.f.	n.f.	n.f.	11
120	93.8	70.3	10.4	21.1	1.72	1.90	88.0	85	26
120	95.0	79	14	14.4	1.82	1.96	84.3	106	26
120	97.6	78	18.5	14.0	1.74	1.95	88.6	123	26

[a]n.f., no filtration permitted; AV1, acid value obtained after extraction; AV2, acid value obtained after 11 mon storage in a cold room (4°C). Intensity = electric current (amperes) required by the motor to ensure turning of the screws and matter conveying.

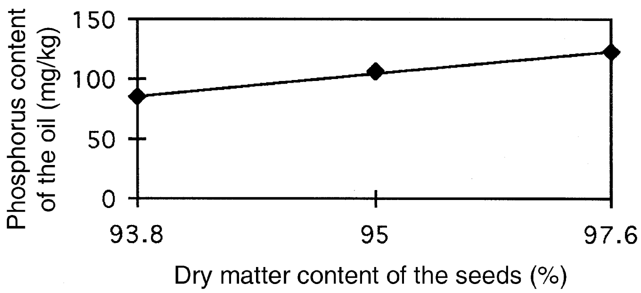


FIG. 6. Influence of the dry matter content of the seeds on the phosphorus content of the filtered oil.

The protein extraction yield for industrial hexane-extracted meal was 66%, whereas for twin-screw extruder pressed fatty meal it was 75–80%, under the following conditions: temperature, 50°C; initial pH, 12; mass ratio solid/liquid, 1:20; stirring rate, 500 rpm; time, 15 min. The solubility is a property often used to evaluate proteins denaturation. A decrease in solubility induced by a thermic treatment is commonly attributed to protein denaturation (12). We see that under the experimental conditions in this work (80, 100 and 120°C; up to 60 s residence time), the fatty meal proteins remain quite soluble: 75 to 80% of the total nitrogen can be extracted within 15 min at 50°C and with an initial pH of 12, whereas a value of only 66% is attained for the industrial meal proteins extracted under the same conditions. This yield is especially high, considering that the alkalinity of the extracting medium is actually reduced by the saponification of the fatty meal residual oil, when a high pH is know to increase the protein extraction yield (11). It therefore can be assumed that the proteins of the fatty meal are still relatively close to their native conformation.

Our results show that a twin-screw extruder can be configured to produce a good quality oil, with expression yields of 80%. These yields are comparable to those obtained by expression with a mono-screw press. The use of an experimental design technique enabled us to precisely quantify the effect of pressing temperature and moisture content of the raw material on the oil yield obtained by the expression of whole oleic sunflower seeds. The efficacy of expression in a twin-screw machine was associated with an optimal configuration of the system (screw profiles), control of parameters intrinsic to the operation (seed input flow rate, screw rotation speed, expression temperature), and control of external parameters (seed moisture content). The advantages of this one-step procedure were further enhanced by eliminating the flaking and cooking steps required in the classical mono-screw press. The injection of a solvent into the twin-screw machine provides an alternative to be developed to further increase these yields up to 90% (13). Moreover, the twin-screw process leads to the production of good-quality proteins that are little denaturated.

Twin-screw extruder pressing constitutes one possible advance in the oil production process. Other seeds such as rapeseed and crambe are under test.

ACKNOWLEDGMENTS

We would like to thank the Toulousaine de Céréales, the A.N.R.T. (convention C.I.F.R.E. n° 054/95), the A.N.V.A.R., the Chambre d'Agriculture de la Haut-Garonne and the Midi-Pyrénées Region for financial support.

REFERENCES

1. Lacaze-Dufaure, C., Z. Mouloungui, and J. Leyris, Method and Apparatus for Making Fatty Acid Esters from Oil-Containing Seeds, PCT WO 97/38069 (FR 96/4502) (1997).
2. Lacaze-Dufaure, C., Fractionation of High Oleic Sunflower Seeds: Pressing and Chemical Transformations of the Triglycerides into Esters Used as Lubricants and Additives, in a Batch Reactor and a Twin Screw Reactor, Ph.D. Thesis, Institut National Polytechnique from Toulouse, France, no. 1438 (1998).
3. Lusas, E.W., and L.R. Watkins, Oilseeds: Extrusion for Solvent Extraction, *J. Am. Oil Chem. Soc. 65*:1109–1114 (1988).
4. Melcion, J.P., Oleo-Proteagineous and Cooking-Extrusion, Cuisson-Extrusion, *Les Colloques de l'INRA, Nantes, 41*:235–247 (1987).
5. Lusas, E.W., L.R. Watkins, and K.C. Rhee, Separation of Fats and Oils by Solvent Extraction: Non-traditional Methods, *World Conference Proceedings, Edible Fats and Oils Processing: Basic Principles and Modern Practices,* edited by D.R. Erickson, American Oil Chemists' Society, Champaign, pp. 56–78 (1989).
6. Williams, M.A., Extraction of Lipids from Natural Sources, in *Lipid Technologies and Applications,* chapter 5, edited by F.D. Gunstone and F.B. Padley, Marcel Dekker Inc., New York, 1997, pp. 113–135.
7. Denise, J., Le Raffinage des Corps Gras, Les Éditions des Beffrois, Westhoek Editions, Paris, 1984, pp. 45–46.
8. Isobe, S., F. Zuber, K. Uemura, and A. Noguchi, A New Twin-Screw Press Design for Oil Extraction of Dehulled Sunflower Seeds, *J. Am. Oil Chem. Soc. 69*:884–889 (1992).
9. Guyomard, P., Study of the Use of a Twin-Screw Extruder in Pressing-Extrusion of Oleagineous Seeds, Ph.D. Thesis, Université Technologique de Compiègne, France (1994).
10. *Guide to the French Standard Methods of Analysis of Oleaginous Seeds,* edited by D. Ribaillier, AFNOR and CETIOM, Paris, 1987.
11. Leyris, J., Sunflower Meal Valorization: Characterization, Process and Model of Proteins Extraction—Utilization of the Protein Functional Properties for the Fabrication of New Natural Products, Ph.D. Thesis, Institut National Polytechnique from Toulouse, France, no. 1464 (1998).
12. Venktech, A., and V. Prakash, Sunflower Seed Total Proteins: Effect of Dry or Wet Heating, *J. Agric. Food Chem. 41*:15577–15582 (1993).
13. Rigal, L., Z. Mouloungui, C. Lacaze-Dufaure, and A. Gaset, Process and Apparatus for Oil Extraction of Oleagineous Seeds, French Patent FR 98/08063 (1998).

[Received December 16, 1998; accepted April 14, 1999]