# TWIN-SCREW EXTRUSION: A SINGLE STEP FOR THREE TREATMENTS OF SUNFLOWER SEEDS

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**Abstract**

A new application of a twin-screw extruder as a reactor to conduct a thermomechanical pressing and a solvent extraction of sunflower oils in a single step and in a continuous mode was studied. Experiments were carried out using a corotating twin screw extruder and whole sunflower seeds with methyl ester as the solvent. The oil extraction yield and the specific mechanical energy were measured as a function of solvent-to-solid (S/S) ratio. Results showed that the oil yield increased when the S/S ratio increased. The best extraction gives 96% of oil yield and < 5% of residual oil content under an S/S ratio of 72%. Besides, this condition conducted lower energy consumed. Moreover, among other things the level of structural modification and the rheological behavior of Extruded Sunflower Cake Meal (ESFCM), the extraction by-product, can give interesting data in order to predict the possibility of forming this material with plastic technologies.

## Introduction

The most common industrial processes for the oil extraction from oleaginous seeds are hydraulic pressing, expeller pressing and solvent extraction. The hydraulic press is comparatively effective, but this process is discontinuous. The application of the continuous oil extraction process using single-screw presses has replaced the conventional method (Isobe et al., 1992; Wang and Johnson, 2001; Crowe et al., 2001; Singh et al., 2002). For materials having relatively high oil contents, such as sunflower, a two-step process is carried out which consists of a continuous prepress stage followed by solvent extraction. Hexane is an excellent solvent for such an extraction system. However, concerns about flammability and toxicity have motivated interest in replacing hexane. Several alternative solvents have been reported by Johnson and Lusas (1991).

The twin-screw extruder played an important role in the food industry, transforming the material physically and chemically in a single step. The main application of the twin-screw extruder is usually found in the production of various products such as snacks, cereals and pet food. Recently, several studies have expanded the utility of the twin-screw extruder as a reactor to conduct a thermo-mechano-chemical action plus a liquid/solid extraction, as in hemicellulose extraction (N'Diaye et al., 1996; N'Diaye and Rigal, 2000), in a continuous mode. The utilization of the twin-screw extruder has also been successfully carried out for the extraction of vegetable oil (Guyomard, 1994; Bouvier and Guyomard, 1997; Dufaure et al., 1999a, 1999b).

Based on those results, more systematic studies should thus be realized to optimize the utility of the twin-screw extruder for the oil extraction of oleaginous seeds. This study purposed to evaluate the new application of the twin-screw extruder as a reactor to conduct simultaneously thermomechanic pressing and solvent extraction of sunflower oils in a single step. The characterization of extraction performance was observed as function of solvent-to-solid ratio.

Moreover, sunflower cake like many other vegetable materials can be transformed by plastic technologies (Rouilly and Rigal, 2002a). The purpose of our study was to evaluate the potential of Extruded Sunflower Cake Meal (ESFCM) to form like a common thermoplastic. Visual and microscopic observation provides some indications about the level of structural modification due to extrusion.

## Materials and Methods

All trials were carried out using whole sunflower seeds rich in linoleic acid (average oil content of 45.31% and 6.58% of moisture content), which were supplied by La Toulousaine de Céréales (France). Methyl ester was supplied by Cognis. All solvents and chemicals were analytical grade obtained from Sigma-Aldrich, Fluka, Prolabo and ICS (France).

Experiments were conducted with a corotating twin-screw extruder (Model BC 45, Clextral, France). It was built with seven modular barrels, each 200 mm long and different twin-screws which had segmental screw elements each 50 and 100 mm in length. Three modules were heated by thermal induction and cooled by water circulation. Material was fed into the extruder inlet port by a volume screw feeder (type 40, Clextral, France). Two filter sections were outfitted on modules 4 and 6 to separate extracted oil. Figure 1 shows the schematic modular barrel of the twin-screw extruder and screw profile tested.

Barrel temperature, screw rotation speed and seed input flow rate were fixed at 80C, 210 rpm and 29 kg/h, respectively. Filtrate (oil/solvent mixture containing the foot) and cake meal samples were collected when extraction operations were under steady state conditions (after 20-25 minutes). The cake meal was further analyzed to determine the residual oil/solvent content (NF V03-908). The concentration of solvent (methyl ester) in filtrate and residual oil was determined using the FAME method.

The calculation of oil extraction yield was determined by the following relationship: R (% mass) = [100*(Qs*Ts – Qc*Tc)]/(Qs*Ts), where Qs is the inlet flow rate of seeds (kg/h), and Qc is the outlet flow rate of the cake meal (kg/h). Ts and Tc are the oil content of the seeds (%) and the cake meal (%), respectively.

Optical microscopy on a binocular microscope (Nikon SMZ 1500) equipped with a digital camera (Nikon DMX 1200) gives some information about the structural modification level of ESFC. Samples were analyzed at their equilibrium moisture content.

A capillary rheometer (Rheomex, Haake PolyLab System, Karlsruhe, Germany) was used to determine the apparent viscosity of material in low moisture conditions. It is a single screw extruder equipped with a capillary die (diameter 3 mm, length/diameter 10). The apparent viscosity was calculated from measurable data: mass flow converted into volume flow rate with known density, and difference between atmospheric pressure and pressure in the capillary die (Rouilly, 2002c). Analysis conditions tested are the ones commonly used to determine the apparent viscosity of sunflower cake produced from the ordinary industrial process of extraction: the water content of ESFCM was 25 % (mixing ESFCM with water by a Perrier 32.00 mixer [Montrouge, France]; the temperature of the capillary die was 120C, and the measurement was made in five steps of screw rotation from 30 to 150 rpm (Rouilly, 2002c).

Thermal analysis were performed on a Pyris 1 power modulated Differential Scanning Calorimeter (Perkin Elmer). The measurement cells were purged with dry nitrogen. Airtight steel capsules with an O-ring seal were used. The samples of ESFCM were equilibrated at 60 % of relative humidity (RH) before being tested. Ordinary sunflower cake (OSFC) was ground on a 1-mm diameter slotted plate of a Cyclotec cutting mill before being equilibrated as described before. Approximately 10 mg of each sample was used and the samples were tested in triplicate. The measurements were taken between 50C and 180C. The heating rate was 10C/min.

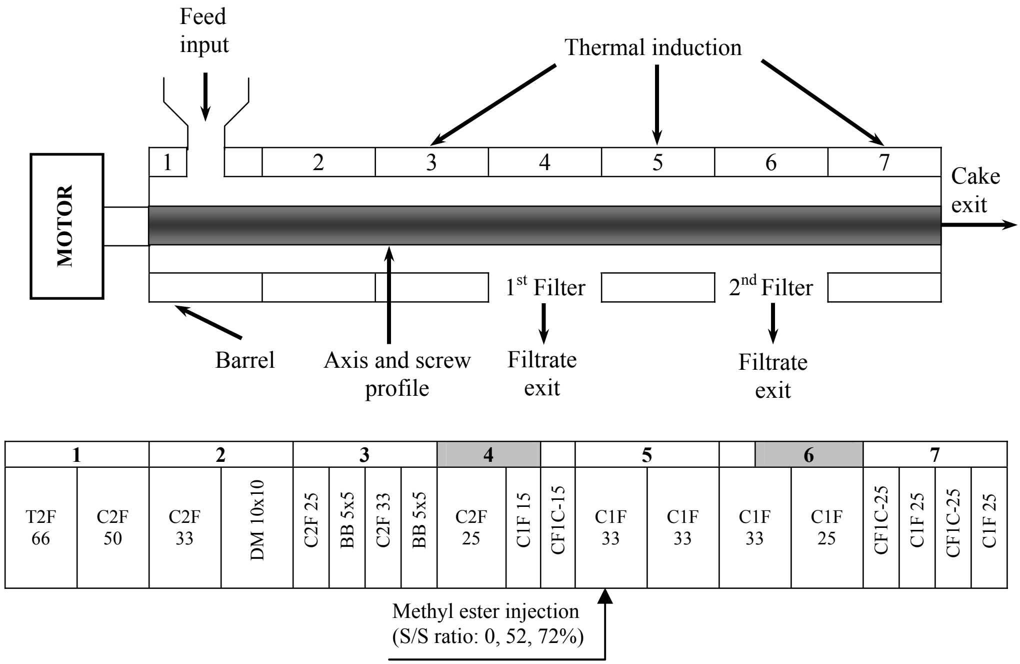


Figure 1. Schematic modular barrel and screw profile of twin-screw extruder.

## Results and Discussion

The simultaneous operation of mechanical pressing and solvent extraction in a single step on oil processing of sunflower seeds by a twin-screw extruder positively affected the oil extraction yield and the specific mechanical energy (SME). The injection of methyl ester in module 5 improved the oil recovery and decreased the SME (Figure 2). More oil yield was observed when the S/S ratio was increased. The highest oil yield of 96% was obtained with an S/S ratio of 72%. In addition, this condition consumed lower energy.

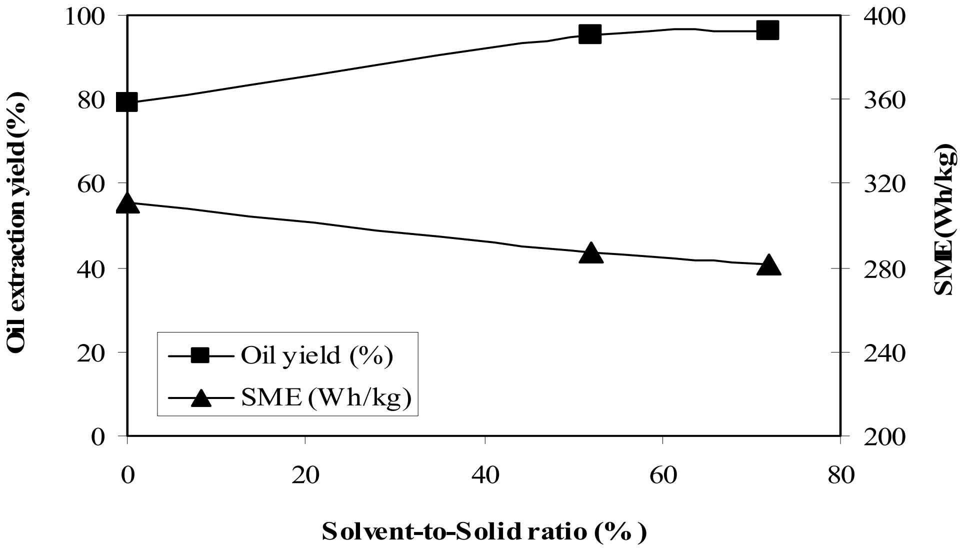


Figure 2. Variation oil extraction yield and specific mechanical energy as function of S/S ratio.

In connection with other extraction performance parameters (Table 1), the results showed that this system particularly affected the residual oil content of cake meal. This parameter decreased significantly when the operation was conducted with solvent injection. Through such screw configuration (Figure 1), the residual oil in the pre-pressed materials was removed further by the solvent and was separated by a second filter. Two filtrates were thus produced by this operation, in which the single expression/extraction can not handle it. The main advantage of this combined process is that it allows thermo-mechanical pressing to be applied to oilseeds and further solvent extraction for pressed materials, which would be quite difficult to process by the direct extraction method, in the same time and with the same machine.

The macroscopic appearance of those three ESFCM is different. ESFCM0, ESFCM52 and ESFCM72 were produced from the extrusion of sunflower seeds with a solvent-to-solid ratio, 0 %, 52 % and 72 %, respectively. First, ESFCMs don't look like common SFCs. Indeed there are many visible hull particles in SFCs, but there aren't in ESFCMs. Hulls of these cakes are totally defibrated. ESFCM0 is lighter brown in color than ESFCM52 and ESFCM72. Nevertheless charred particles were observed by optical microscopy that agrees with the smell. ESFCM52 and ESFCM72 also produce a burnt odour, but the optical microscopy does not disclose any distinct carbon. Their dark brown colour seems due to the residual solvent content. ESFCM52 and ESFCM72 are bright like an oil coated material. Moreover, the three ESFCMs are sticky. The residual oil (+ solvent) content explains this property (Table 1). This stickiness is a problem when measuring the melted phase rheology of ESFCM. It is impossible to obtain any processing in the usual measurement conditions of sunflower cake (Rouilly, 2002c). The feeding is irregular because the powder sticks on the screw, which no longer feeds the capillary die. The volumetric flow rate is not measurable anymore. Finally ESFCMs burn in the barrel and create a plug. Therefore the feeding of a plasticization screw of an injection press or one of an extruder is also impossible.

Table 1. Extraction performance as function of solvent-to-solid ratio.

| Parameters | Solvent-to-solid ratio (%) | | |
|---|---|---|---|
| | 0 | 52 | 72 |
| Flow rate of filtrate 1 (kg/h) | 11.03 | 11.43 | 11.27 |
| Foot content of filtrate 1 (%) | 26 | 31 | 30 |
| Solvent content of filtrate 1 (%) | 0 | 0 | 7.73 |
| Flow rate of filtrate 2 (kg/h) | 0 | 15.20 | 20.34 |
| Foot content of filtrate 2 (%) | 0 | 51 | 44 |
| Solvent content of filtrate 2 (%) | 0 | 95.48 | 95.94 |
| Flow rate of cake meal (kg/h) | 15.48 | 12.20 | 11.57 |
| Residual oil (+ solvent) content of cake meal (%) | 17.48 | 27.46 | 27.13 |
| Solvent content of cake meal (%) | 0 | 81.62 | 83.67 |
| Moisture (+ solvent) content of cake meal (%) | 0.99 | 2.40 | 2.72 |

Figure 3 shows that globulins of ESFCMs are denaturized. According to Rouilly et al. (2003), a peak corresponding to the temperature of denaturation of sunflower protein is observed around 152C for the common SFC, but it disappears for ESFCMs. Sunflower proteins of ESFCMs are thus completely denaturized by the extraction step during extrusion. Indeed in this process sunflower seeds underwent strong conditions during the extraction. The two reverse pitch screws create two strong pressure zones where the SME (Figure 2) and shear stress are high and consequently self-heating could occur. This could explain the burnt odour and charred particles. ESFCM proteins melted well and acquired their adhesive property.

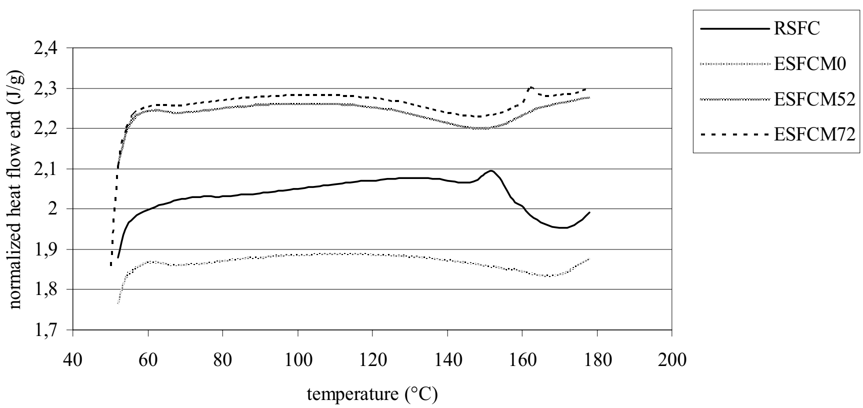


Figure 3. Thermograms in airtight pans of sunflower cake samples after conditioning at 60 % of relative humidity.

## Conclusions

Simultaneous operation of a twin-screw extruder to conduct mechanical pressing and solvent extraction in a single step was a promising alternative technology for oil processing of sunflower seeds, and methyl ester is an excellent alternative solvent to replace hexane.

In actuality, sunflower cake meal produced from this process doesn't allow formation by extrusion or injection-molding. Efficient oil extraction results in protein denaturation and fiber degradation.

## References

Bouvier, J.M. and Guyomard, P. 1997. Method and installation for continuous extraction of a liquid contained in a raw material. PCT/FR97/00696.

Crowe, T.W., Johnson, L.A., and Wang, T. 2001. Characterization of extruded-expelled soybean flours. J. Am. Oil Chem. Soc. 78:775-779.

Dufaure, C., Leyris, J., Rigal, L., and Mouloungui, Z. 1999a. A twin-screw extruder for oil extraction: I. Direct expression of oleic sunflower seeds. J. Am. Oil Chem. Soc. 76:1073-1079.

Dufaure, C., Mouloungui, Z., and Rigal, L. 1999b. A twin-screw extruder for oil extraction: II. Alcohol extraction of oleic sunflower seeds. J. Am. Oil Chem. Soc. 76:1073-1079.

Guyomard, P. 1994. Study of the use of a twin-screw extruder in pressing – extrusion of oleagineous seeds. Ph.D. Thesis, Université Technologique de Compiégne, France.

Isobe, S., Zuber, F., Uemura, K., and Noguchi, A. 1992. A new twin-screw press design for oil extraction of dehulled sunflower seeds. J. Am. Oil Chem. Soc. 69:884-889.

Johnson, L.A. and Lusas, E.W. 1983. Comparison of alternative solvents for oils extraction. J. Am. Oil Chem. Soc. 60:229-242.

N'Diaye, S. and Rigal, L. 2000. Factors influencing the alkaline extraction of poplar hemicelluloses in a twin-screw reactor: correlation with specific mechanical energy and residence time distribution of the liquid phase. Bioresource Technology. 75:13-18.

N'Diaye, S., Rigal, L., Larocque, P., and Vidal, P.F. 1996. Extraction of hemicelluloses from poplar, *Populus tremuloides*, using an extruder-type twin-screw reactor: a feasibility study. Bioresource Technology. 57:61-67.

Rouilly, A. and Rigal, L. 2002a. Agro-materials: a bibliographic review. Journal of Macromolecular Sciences. Part C Polymer Reviews. 42:441-479.

Rouilly, A. 2002b. Nouveaux agro-matériaux composites à matrice protéique ou polysaccharidique: étude du fractionnement, de la transformation et de la mise en forme par extrusion et par injection-moulage de la pulpe de betterave et du tourteau de tournesol. Ph.D. Thesis, Institut National Polytechnique de Toulouse, France.

Rouilly, A., Orliac O., Silvestre F., and Rigal L. 2002c. Injection-molding of sunflower oil cake. 12th European Conference and Technology Exhibition Biomass for Energy, Industry and Climate Protection. Amsterdam, Netherlands.

Rouilly, A., Orliac, O., Silvestre, F., and Rigal, L. 2003. Thermal denaturation of sunflower globulins in low moisture conditions. Thermochimica Acta. 398: 95-201.

Singh, K.K., Wiesenborn, D.P., Tostenson, K., and Kangas, N. 2002. Influence of moisture content and cooking on screw pressing of crambe seed. J. Am. Oil Chem. Soc. 79:165-170.

Wang, T., and Johnson, L.A. 2001. Refining normal and genetically enhanced soybean oils obtained by various extraction methods. J. Am. Oil Chem. Soc. 78:809-815.