

# METHYL ESTER EXTRACTION OF SUNFLOWER OIL IN A TWIN SCREW EXTRUDER

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## ABSTRACT

Sunflower oil was extracted from whole sunflower seeds using methyl ester as the solvent. Experiments were conducted in a co-rotating in-screw extruder. The oil extraction yield was measured as function of screw configuration and solvent-to-solid (S/S) ratio. The position of the reverse screw elements affected oil extraction yield. Higher oil recovery was produced as the S/S ratio was increased up to 90% and the oil removed from seeds under S/S ratio of 0.65. The methyl ester is thus a promising alternative solvent for extraction of sunflower oil.

**Keywords :** twin-screw extruder, sunflower oil, methyl ester and extraction

## INTRODUCTION

In the food industry, twin-screw extruder is principally used in the production of various products such as snacks, cereals and pet food. Today, the application of twin-screw extruder has been successfully carried out to extract oil from oleaginous seeds (Isobe et al., 1992; Guyonard, 1993; Bouvier and Guyonard, 1997; Lacaze-Dufaure et al., 1999; Amalia Kartika et al., 2005, 2006).

Conventional industrial oil extraction from oilseeds is usually realized through mechanical pressing with a hydraulic or single expeller press, followed by solvent extraction. The combination of these operations produced oil extraction yield up to 98% with residual oil content in cake meal of 0.5 - 1.5% (Campbell, 1983). The solvent extraction most commonly used today is percolation extraction with a counter-current flow using hexane as a solvent (Johnson and Lusas, 1983; Wan et al., 1995; Conkerton et al., 1995; Proctor and Bowen, 1996; Hu et al., 1996). However, toxicological risks, flammability, health and environmental concerns have motivated interest to replace hexane. Several alternative solvents have been reported by a few researchers (Hron et al., 1982; Hron and Kolton, 1984; Johnson et al., 1986; Lusas et al., 1990; Hron et al., 1992; Abraham et al., 1993; Hron et al., 1994; Devitori et al., 2000; Hammounjai, 2000; Hojilla-Evangelista and Johnson, 2002; Kwiatkowski and Cheryan, 2002; Kiriamiti, 2002; Gomez and Martinez de la Ossa, 2002). The application of solvent derived from vegetable oils, such as fatty acid methyl esters, has attracted attention recently from a few researchers due to its environmental

benefits, and because vegetable oils are non-toxic, renewable and biodegradable (Gérin, 2002).

The great capability of the twin-screw extruder to conduct diverse functions and processes comes from its characteristics. According to Dziezak (1989), those advantages include (i) ability to provide better process control and versatility, especially in pumping efficiency, controlling residence time distribution and uniformity of processing, (ii) ability to process specialty formulation (iii) flexibility in design of the machine, which permits self-cleaning mechanisms and rapid change over of screw configuration without disassembling the extruder.

A twin-screw extruder is based around elements, namely screws, including (i) forward pitch screw, which principally conducts a conveying action, (ii) monolobe paddle (DM), which primarily exerts a radial compression and shearing action, (iii) bilobe paddle (BB), which exerts a significant mixing and shearing action, conveying and axial compression actions in combination with forward pitch screw, and (iv) reversed pitch screw, which carries out intensive shearing and considerable mixing, and exerts a strong axial compression in combination with a forward pitch screw (Rigal, 1996). The arrangement of different characteristics of screw elements (pitch, stagger angle, length) in different positions determine screw profile/configuration that is the main factor influencing performance (product transformation, residence time distribution, mechanical energy input) during extrusion processing (Gogoi et al., 1996; Choudhury et al., 1998; Gautam and Choudhury, 1999a, 1999b; Amalia Kartika et al., 2005, 2006). Furthermore, by modularity of its configuration and screw profile, the twin-screw extruder enables a large number of basic operations, such as material transport, grinding/

crushing, mixing, chemical reaction, liquid-solid extraction, liquid-solid separation and drying, to be carried out in a single step (Rigal, 1996), not possible with conventional presses.

In the case of oil extraction of sunflower seeds using a co-rotating twin screw extruder, the screw configuration and the operating conditions had an important influence on the oil extraction yield, the energy input and the quality of oil extracted (Amalia Kartika et al., 2006). Higher oil extraction yield and specific mechanical energy were reached as the reversed screw elements were moved with increased spacing between elements and with smaller pitch elements. A systematic increase in oil extraction yield was observed as the barrel temperature, the screw rotation speed and the feed rate were decreased. Highest oil extraction yield of 85% with very good oil quality (acid value below 2 mg of KOH/g of oil and total phosphorus content below 100 mg/kg) was obtained under operating conditions of 120°C, 75 rpm and 19 kg/h. However, the quality of the cake meal was low because the residual oil content was high (> 12%) while the moisture content was low (< 1.5%). For further utilization of the cake meal, those qualities were very favorable, particularly for extraction of residual oil contained in the cake meal, as solvent extraction is an adaptable method to treat this type of cake meal. In addition, the characteristic particle size distribution of cake meal, which was dominated by particles less than 0.5 mm in diameter (> 80%) facilitates the application of this method.

This study set out to evaluate the application of twin-screw extruder to conduct linoleic methyl ester extraction of sunflower oils in a continuous mode. The characterization of extraction performance was observed by the determinations of oil extraction yield and mechanical energy input as function of screw configuration and solvent-to-solid ratio.

## MATERIALS AND METHODS

### Materials

All trials were carried out using whole and uncleaned sunflower seeds, which were supplied by La Toulousaine de Cereales. The sunflower seeds were of the classic and oleic types. The oil content of seeds used, expressed in relation to the dry matter content of uncleaned seed, was 48.5% (classic type) and 42.2% (oleic type). The seed moisture content at storage was 6.6% (classic type) and 6.2% (oleic type). The linoleic methyl esters were of the sunflower oils type, which was supplied by COGNIS. All solvents and chemicals were analytical grades that were obtained from Sigma-Aldrich, Fluka, Prolabo and ICS.

### Twin-Screw Extruder

Experiments were conducted with a CLEXTRAL BC 45 co-rotating twin-screw extruder. It was built with seven modular barrels, each 200 mm in length, and different twin-screws which had segmental screw elements each 50 and 100 mm in length. The modules were heated by thermal induction and cooled by water circulation. Seeds were fed into the extruder inlet port by a volumic screw feeder, and linoleic methyl esters were injected on module 3 by a piston pump. The filter section consisting of six hemispherical dishes with perforations 1 mm in diameter was outfitted on module 4 to separate extracted oil. Figure 1 shows three screw profiles tested.

### Experimental

For all experiments, the temperature along the barrel and the feed rate were fixed at 80°C and 25 kg/h, respectively. The screw rotation speed was fixed at 166 - 210 rpm, while the solvent-to-solid ratio was varied from 0 to 0.63. To ensure a stable flow rate and barrel temperature, the extruder was operated for 20 - 25 minutes before processing the actual samples. Upon achieving steady operation, filtrate (oil/linoleic methyl ester mixture containing the foot) and cake meal samples were immediately collected over a period of 20 minutes. The filtrate and cake meal were weighed. The filtrate was further centrifuged to separate the foot from the oil/linoleic methyl ester mixture. The moisture and residual oil contents of the cake meal were measured according to standards NF V03-903 and NF V03-908. The linoleic methyl ester content of filtrate and residual oil contained in cake meal was determined by gas chromatography using FAME method. Oil extraction yield was calculated from the following relationship :

$$R = 100 \times [(Q_{sd}T_{sd} - Q_{cd}T_{cd}) / (Q_{sd}T_{sd})]$$

where  $R$  is the oil extraction yield based on residual oil content of cake meal (% mass).  $Q_{sd}$  is the inlet flow rate of the dry seed (kg/h) and  $Q_{cd}$  is the outlet flow rate of the dry cake meal (kg/h).  $T_{sd}$  and  $T_{cd}$  are the oil contents of the seed (%) and the cake meal (%), respectively, in relation to the dry matter.

### Oil Quality Analysis

The quality parameters of a crude oil included (i) the acid value, expressed in mg of KOH/g of oil (NF T 60-204), which is an indication of the free fatty acid content of the oil, and (ii) the iodine value, expressed in terms of the number of centigrams of iodine absorbed per gram of oil (AOCS-Cd 1d-92), which is a measure of the unsaturation of oils.

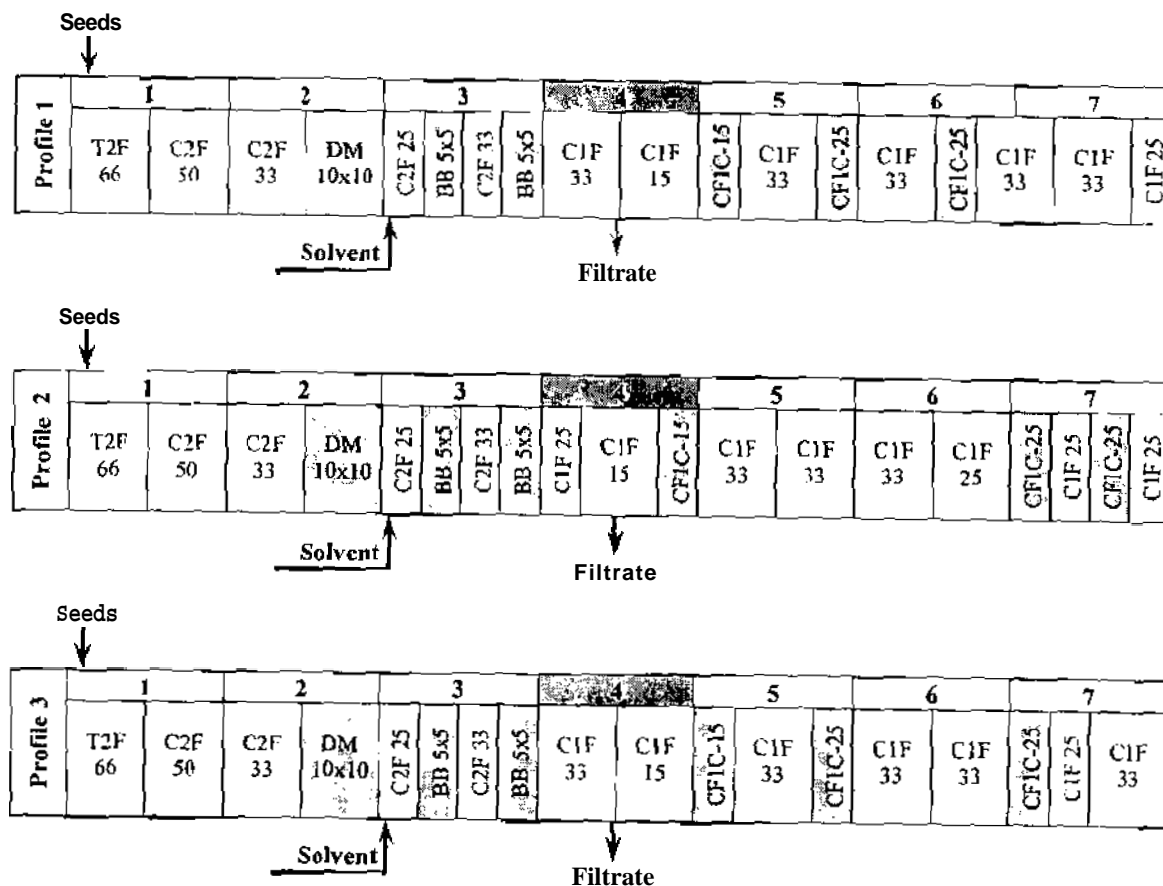


Figure 1. Screw profiles tested for oil extraction of sunflower seeds in a twin-screw extruder BC 45

### Specific mechanical energy

The specific mechanical energy (SME) was calculated from the following equation :

$$SME = [P/Q_S] \text{ and } P = [(460 I \times 0.95 S_s) / 600]$$

where  $I$  is the electric current (A),  $P$  is the motor power (W),  $Q_S$  is the inlet flow rate of the seed (kg/h) and  $S_s$  is the screw rotation speed (rpm). 460 (Volt) and 600 (rpm) are used to describe the efficiency of the extruder.

### Results and discussion

The injection of linoleic methyl ester on module 3 improved the oil extraction yield and decreased the residual oil content of cake meal (Table 1). The injection of linoleic methyl ester facilitated the extraction processing by solubilizing the oil contained in materials, the oil extraction yield was thus increased by approximately 10 - 20%. More oil extraction yield was observed when the solvent-to-solid (S/S) ratio was increased, and

the reversed screw elements were configured with increased spacing between elements.

In addition, the solvent-to-solid (S/S) ratio affected the specific mechanical energy. The augmentation of the specific mechanical energy with an increase of the solvent-to-solid (S/S) ratio increased the pressing efficacy of the reversed screw elements due to an increase in the residence time in the whole extruder, the oil extraction yield thus increased. The flow rate of filtrate increased when the solvent-to-solid (S/S) ratio increased, followed by a decrease in the flow rate of cake meal and the foot content of filtrate.

For all operating conditions tested, the quality of filtrate was good (Table 2). The acid value remained stable (< 2 mg of KOH/g of oil) and the iodine values were acceptable. The linoleic methyl ester content of filtrate remained at 50 - 66%. More linoleic methyl ester content was observed when the solvent-to-solid (S/S) ratio was increased. These qualities were advantageous for direct utilization of filtrate such as bio-fuels. Increasing the use of bio-fuels for energy purposes is of particular interest because they have considerable environmentally friendly potential, provide means of energy

renewable and may even offer new employment possibilities (Demirbas and Mustafa Balat, 2006). Actually, these bio-oils are being investigated as a substitute for fossil fuels to generate heat, power and/or chemicals.

The operating conditions generally improved the quality of cake meal, mainly the residual oil content. The values obtained were low (< 10%) for all operating conditions tested (Table 1). However, the residual linoleic methyl ester contained in cake meal was still high (> 8%). Although these qualities were disadvantageous for direct utilization of cake meal, they can be converted into usable energy by combustion, gasification and pyrolysis (Yorgun et al., 2001; Gerçel, 2002), or transformed into the

composite agro-materials, such thermoplastics materials (Leyris et al., 1998; Rouilly et al., 2004).

### CONCLUSION

This study shows that the application of twin-screw extruder to conduct solvent extraction of sunflower oils in a continuous mode have been successfully carried out, and was a promising alternative technology for oil processing of sunflower seeds. Highest oil extraction yield of 90.7% with good oil quality and moderate specific mechanical energy (282.1 W.h/kg) was obtained under S/S ratio of 0.65 and screw profile of 1).

Table 1. Influence of screw configuration and solvent-to-solid (S/S) ratio on methyl ester extraction of sunflower oils in a twin-screw extruder BC 45

Profile	Seed type	S <sub>s</sub> (rpm)	Q <sub>s</sub> (kg/h)	S/S ratio	FILTRATE		CAKE MEAL				R (%)	SME (W.h/kg)
					Flow rate (kg/h)	Foot content (%)	Flow rate (kg/h)	Moisture content (%)	Solvent content (%)	Oil content (%)		
1	Oleic	166	23.79	0	5.43	9	16.29	7.81	0	23.47	62.7	2490
	Oleic	166	23.07	0.54	17.97	3	14.33	5.87	9.96	10.28	84.9	374.7
	Oleic	166	26.36	0.63	23.04	4	12.90	3.27	10.21	7.76	90.7	282.1
2	Oleic	210	25.28	0.49	18.78	10	14.58	1.54	9.15	10.29	85.3	402.4
	Oleic	210	26.67	0.62	23.33	7	13.25	1.65	10.68	9.15	88.7	312.6
3	Oleic	175	27.30	0	6.74	13	14.52	2.99	0	16.91	77.9	289.5
	Oleic	210	22.30	0.56	19.94	10	14.20	6.29	9.88	7.93	88.0	377.2
	Classic	210	25.43	0	10.82	33	14.37	1.12	0	19.57	75.9	327.9
	Classic	210	21.84	0.57	21.32	14	11.46	1.08	7.68	8.89	89.8	360.7

Table 2. Influence of screw configuration and solvent-to-solid (S/S) ratio on oil quality

Profile	Seed type	S <sub>s</sub> (rpm)	Q <sub>s</sub> (kg/h)	S/S ratio	Acid value (mg KOH/g oil)	Iodine value (mg iodine/100 g oil)	Solvent content (%)
1	Oleic	166	23.79	0	3.15	85.00	0
	Oleic	166	23.07	0.54	1.28	112.02	55.11
	Oleic	166	26.36	0.63	2.27	114.90	65.73
2	Oleic	210	25.28	0.49	1.47	111.18	50.64
	Oleic	210	26.67	0.62	1.64	113.73	61.12
3	Oleic	175	27.30	0	2.41	85.16	0
	Oleic	210	22.30	0.56	1.30	114.80	54.87
	Classic	210	25.43	0	1.32	86.16	0
	Classic	210	21.84	0.57	0.88	104.22	51.60

## REFERENCES

- Abraham, G., Hron, R.J., Kuk, M.S., Wan, P.J., 1993. Water accumulation in the alcohol extraction of cottonseed. *J. Am. Oil Chem. Soc.* 70, 207-208.
- Amalia Kartika, I., Pontalier, P.Y., Rigal, L., 2005. Oil extraction of oleic sunflower seeds by twin screw extruder: influence of screw configuration and operating conditions. *Industrial Crops and Product* 22, 207-222.
- Amalia Kartika, I., Pontalier, P.Y., Rigal, L., 2006. Extraction of oleic sunflower oil by twin screw extruder: screw configuration and operating conditions effects. *Bioresource Technology* 97, 2302-2310.
- Bouvier, J.M., Guyomard, P., 1997. Method and installation for continuous extraction of a liquid contained in a raw material. PCT/FR97/00696.
- Campbell, E.J., 1983. Sunflower oil. *J. Am. Oil Chem. Soc.* 60, 387-392.
- Choudhury, G.S., Gogoi, B.K., Oswalt, A.J., 1998. Twin-screw extrusion of pink salmon muscle and rice flour blends: effects of kneading elements. *Journal of Aquatic Food Product Technology* 7, 69-91.
- Conkerton, E.J., Wan, P.J., Richard, O.A., 1995. Hexane and heptane as extraction solvents for cottonseed: a laboratory-scale study. *J. Am. Oil Chem. Soc.* 72, 963-965.
- Demirbas, M.F., Mustafa Balat, 2006. Recent advances on the production and utilization trends of bio-fuels: a global perspective. Article in Press. *Energy Conversion and Management*.
- Devittori, C., Gummy, D., Kusy, A., Colarow, L., Bertoli, C., Lambelet, P., 2000. Supercritical fluid extraction of oil from millet bran. *J. Am. Oil Chem. Soc.* 77, 573-579.
- Dziezak, J.D., 1989. Single- and twin-screw extruders in food processing. *Food Technology*, pp. 164-174.
- Gautam, A., Choudhury, G.S., 1999a. Screw configuration effect on starch breakdown during twin-screw extrusion of rice flour. *Journal of Food Processing Preservation* 23, 355-375.
- Gautam, A., Choudhury, G.S., 1999b. Screw configuration effect on residence time distribution and mixing in twin-screw extruder during extrusion of rice flour. *J. Food Process Engineering* 22, 263-285.
- Gérin, M., 2002. *Solvants industriels: santé, sécurité, substitution*. Masson, Paris.
- Gerçel, H.F., 2002. The production and evaluation of bio-oils from the pyrolysis of sunflower-oilcake. *Biomass and Bioenergy* 22, 307-314.
- Gogoi, B.K., Choudhury, G.S., Oswalt, A.J., 1996. Effects of location and spacing of reversed screw and kneading element combination during twin-screw extrusion of starchy and proteinaceous blends. *Food Research International* 29, 505-512.
- Gomez, A.M., Martínez de la Ossa, E., 2002. Quality of borage seed oil extracted by liquid and supercritical carbon dioxide. *Chemical Engineering Journal* 88, 103-109.
- Guyomard, P., 1994. Study of the use of a twin-screw extruder in pressing - extrusion of oleagineous seeds. PhD Thesis, Université Technologique de Compiègne, France.
- Hanmounjai, P., Pyle, L., Niranjana, K., 2000. Extraction of rice bran oil using aqueous media. *J. Chem. Technol. Biotechnol.* 75, 348-352.
- Hojilla-Evangelista, M.P., Johnson, L.A., 2002. Factors affecting oil extraction/water adsorption in sequential extraction processing of corn. *J. Am. Oil Chem. Soc.* 79, 815-823.
- Hron, R.J., Koltun, S.P., Graci, A.V., 1982. Bio-renewable solvents for vegetable oil extraction. *J. Am. Oil Chem. Soc.* 59, 674A.
- Hron, R.J., Koltun, S.P., 1984. An aqueous ethanol extraction process for cottonseed oil. *J. Am. Oil Chem. Soc.* 61, 1457-1460.
- Hron, R.J., Abraham, G., Kuk, M.S., Fisher, G.S., 1992. Acidic ethanol extraction of cottonseed. *J. Am. Oil Chem. Soc.* 69, 951-952.
- Hron, R.J., Kuk, M.S., Abraham, G., Wan, P.J., 1994. Ethanol extraction of oil, gossypol and aflatoxin from cottonseed. *J. Am. Oil Chem. Soc.* 71, 417-421.
- Hu, W., Wells, J.H., Tai-Shun Shin, Godber, J.S., 1996. Comparison of isopropanol and hexane for extraction of vitamin E and oryzanols from stabilized rice bran. *J. Am. Oil Chem. Soc.* 73, 1653-1656.
- Isobe, S., Zuber, F., Uemura, K., Noguchi, A., 1992. A new twin-screw press design for oil extraction of dehulled sunflower seeds. *J. Am. Oil Chem. Soc.* 69, 884-889.
- Lacaze-Dufaure, C., Mouloungui, Z., Leyris, Z., Rigal, L., Gaset, A., Silvestre, F., 1996. Procédé et dispositif pour la fabrication d'ester d'acides gras à partir de graines oleagineuses. FR2747128-A1.
- Lacaze-Dufaure, C., Leyris, J., Rigal, L., Mouloungui, Z., 1999. A twin-screw extruder for oil extraction: I. Direct expression of oleic sunflower seeds. *J. Am. Oil Chem. Soc.* 76, 1073-1079.
- Leyris, J., Silvestre, F., Rigal, L., Gaset, A., 1998. Procédé de fabrication d'objets à partir de matière première végétale par formage ou thermoformage. FR 2 784 047.

- Johnson, L.A., Lusas, E.W., 1983. Comparison of alternative solvents for oils extractions. *J. Am. Oil Chem. Soc.* 60, 229-242.
- Johnson, L.A., Farnsworth, J.T., Sadek, N.Z., Chamkasem, N., Lusas, E.W., Reid, B.L., 1986. Pilot plant studies en extracting cottonseed with methylene chloride. *J. Am. Oil Chem. Soc.* 63, 647.
- Kiriamiti, H.K., Rascol, E., Marty, A., Condor, J.S., 2002. Extraction rates of oil from high oleic sunflower seeds with supercritical carbon dioxide. *Chemical Engineering and Processing* 41, 711-718.
- Kwiatkowski, J.R., Cheryan, M., 2002. Extraction of oil from ground corn using ethanol. *J. Am. Oil Chem. Soc.* 79, 825-829.
- Lusas, E.W., Watkins, L.R., Rhee, K.C., 1990. Separation of fats and oils by solvent extraction: non-traditional methods. *Proceedings of the World Conference on Edible Fats and Oils Processing: Basic Principles and Modern Practises*. AOCS Press, Champaign, IL, pp. 56-78.
- Proctor, A., Bowen, D.J., 1996. Ambient-temperature extraction of rice bran oil with hexane and isopropanol. *J. Am. Oil Chem. Soc.* 73, 811-813.
- Rigal, L., 1996. Twin-screw extrusion technology and the fractionation of vegetable matter. *CLEXTRAL Conference, Firminy-France*.
- Rouilly, A., Peyrat, E., Orliac, J., Jorda, J., Sivestre, F., Rigal, L., 2004. New agro-materials from raw agricultural by-products. *Proceeding of 2<sup>nd</sup> World Conference on Biomass for Energy, Industry and Climate Protection, Rome, Italy*.
- Wan, P.J., Pakarinen, D.R., Hron, R.J., Richard, O.L., Conkerton, E.J., 1995. Alternative hydrocarbon solvents for cottonseed extraction. *J. Am. Oil Chem. Soc.* 72, 653-659.
- Yorgun, S., Şensöz, S., Koçkar, Ö.M., 2001. Flash pyrolysis of sunflower oil cake for production of liquid fuels. *Journal of Analytical and Applied Pyrolysis* 60, 1 - 12.