# Lipid of Sunflower Seeds Produced in Japan

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## ABSTRACT

The fatty acid composition and tocopherol content of sunflower seeds from experimental plantings in Japan were determined. Lipid content of sunflower seed was almost the same irrespective of the variety and the average lipid content was 38.8%. The saturated fatty acids were low and the combined percentage of linoleic acid and oleic acid was ca. 90%. The ratio of oleic acid to linoleic acid largely varied with the planting location and date. There was a strong correlation between the percentage of linoleic acid or oleic acid and the temperature during maturation of seed. The sunflower seed contained predominantly  $\alpha$ -tocopherol and small amounts of  $\beta$ -,  $\gamma$ - and  $\delta$ -tocopherol. There was no correlation between  $\alpha$ -tocopherol content and the percentage of linoleic acid.

## INTRODUCTION

The production of sunflower oil in the world reached a level of 4.76 million tons in 1978 and at present, among edible vegetable oils, ranks second only to soybean oil. Most of the sunflower seeds are produced in the USSR, the USA, Argentina and Rumania. In Europe sunflower seed has been utilized as a source of edible oils for many years. The production of sunflower seed in the USA has rapidly increased; 0.27 million tons in 1974 and 3.49 million tons in 1979 (1). However, in Japan the production of sunflower seed is negligible. Sunflower oil is not popular and only a small amount of sunflower oil is imported.

In Japan the overproduction of rice plant is a serious problem and part of the paddy fields is being converted into fields, for other crops such as soybean, wheat, potato, etc. Sunflower was expected to be one of these crops, to become an indigenous oil source like rice bran. Experimental plantings were carried out in several experiment stations in cooperation with the National Federation of Agricultural Cooperative Association. This work was undertaken to determine fatty acid composition and tocopherol content of seeds from the experimental plantings in Japan.

## EXPERIMENTAL

## Extraction of Lipid

Sunflower seeds were obtained from experimental plantings carried out in 1981 in the agricultural research center located in Ibaraki Prefecture and the following stations: Fukushima Prefecture, Gifu Prefecture, Okayama Prefecture and Ehime Prefecture Agricultural Experiment Station.

Ten g of seed were homogenized in 100 mL of chloroform/ methanol (2:1, v/v) with an Ace Homogenizer at 15,000 rpm for 6 min. The homogenate was filtered and the extraction procedure from the residue was repeated twice. The combined filtrate was concentrated and dried, and then the extract was dissolved in 50 mL of chloroform. The insoluble materials were removed by filtration and the resultant clear filtrate was concentrated, dried in vacuo and then weighed as a total lipid.

## Gas Chromatography

Total lipid was saponified and the recovered fatty acids were converted to methyl esters by the addition of diazomethane (2). Gas chromatographic analysis was carried out with a Shimadzu GC-7AG instrument equipped with a flame ionization detector (Shimadzu Seisakusho Co. Ltd., Kyto, Japan) on a G-SCOT column, 40 m  $\times$  0.28 mm, coated with Silar 5CP (Gasukuro Kogyo Co. Ltd., Tokyo, Japan). The carrier gas was N<sub>2</sub> at a flow rate of 0.95 mL/min and a split ratio of 1/58. The column was operated isothermally at 190 C; injector and detector temperatures were 230 C.

## Determination of Tocopherol by High Performance Liquid Chromatograph (HPLC)

Tocopherols were separated with an ATTO Liquid Chromatograph HSLC-01-3-S (ATTO Co. Ltd., Tokyo, Japan) on a Spherisorb S5W column, 4.6 × 200 mm (Phase Separations Ltd., UK) and a Unisil Q 30 precolumn, 4.0 × 50 mm (Gasukuro Kogyo Co. Ltd.), and monitored with a Shimadzu RF-500 Spectrofluorophotometer (Ex. 298 nm, Em 325 nm). The mobile phase was *n*-hexane/isopropanol (99.75: 0.25, v/v) at a flow rate of 1.4 mL/min. One gram of lipid was dissolved in *n*-hexane to 10 mL and the supernatant after centrifugation was injected into the column with a sample loop of 20  $\mu$ l. Tocopherols were determined by comparing the peak area with that of external standard which was injected after each analysis. The standard solution contained  $\alpha$ -,  $\beta$ -,  $\gamma$ - and  $\delta$ -tocopherol at a concentration of 50  $\mu$ g/mL.

## **Statistics**

Two-way analysis of variance was carried out to determine whether there were significant differences in various values among the varieties and also among the environmental conditions. Four varieties (Tainan No. 1, IS-903, IS-907 and Peredovik) and seven environmental conditions (planting location and date designated in Table I) were selected for the analysis of variance.

## **RESULTS AND DISCUSSION**

#### Seed Yield and Lipid Content

Yield and lipid content of sunflower seed are shown in Table I. The seed yield varied from 1420 to 4220 kg/ha and the average yield of 48 samples was 2580 kg/ha. There were significant differences in seed yield among the varieties and environment. Tainan No. 1 showed higher seed yield than IS-903, IS-907 or Peredovik. Even in the same location the seed yield varied largely with the planting date. The seed yields of soybean and rape produced in Japan were 1470 and 1720 kg/ha in 1979 (3), respectively, while the average seed yield of sunflower was 2580 kg/ha in the experimental plantings. Therefore sunflower is a superior oil crop with a high seed yield.

The average lipid content of 53 sunflower seed samples was 38.8% and this value was greater than that of soybean (ca. 20%) and comparable to that of rapeseed (ca. 40%). The lipid content varied in the following ranges: Tainan No. 1, 25-47%: IS-903, 31-45%: IS-907, 31-41%: Peredovik, 33-43%. Two-way analysis of variance showed that there was no significant difference in lipid contents either among the four varieties above or among the environmental conditions. There was no correlation between lipid content and average temperature during maturation of seed. Robertson et al. (4) also found that temperature had no significant effect on oil contents. Ganvin (5) reported that the oil content of sunflower seed produced in a growth cabinet was as follows: 37.3% at 16 C, 40.4% at 21 C, 36.4% at 26.5 C. This small variation from 36.4 to 40.4% in a temperature-controlled cabinet seems to be disturbed in the field experiments and to result in no correlation between lipid content and temperature.

#### TABLE I

Yield and Lipid Content of Sunflower Seed

Sample no.	Planting location	Variety	Planting date (1981)	Seed yield (kg/ha)	Lipid content <sup>b</sup> (%)	Temperature <sup>c</sup> (C)		
1	Fukushima	Tainan No. 1	June 2	1850	43.5	24.2		
2	Fukushima	Tainan No. 1	June 10	1690	40.5	23.8		
3	Fukushima	Tainan No. 1	June 20	2740	43.3	22.6		
4a	Fukushima	Tainan No. 1	July 1	2230	46.6	20.8		
5	Fukushima	Tainan No. 1	July 10	2580	43.1	18.8		
6 <sup>a</sup>	Fukushima	IS-903	July 1	2050	40.4	20.8		
7a	Fukushima	IS-907	July 1	2060	38.9	20.8		
8 <sup>a</sup>	Fukushima	Peredovik	July 1	2280	43.2	20.8		
9 <b>a</b>	Gifu	Tainan No. 1	June 30	2230	41.4	23.5		
10	Gifu	Tainan No. 1	July 20	2770	41.6	20.4		
11 <sup>a</sup>	Fukushima	IS-903	June 30	1560	36.8	23.5		
12	Gifu	IS-903	July 20	3230	36.3	20.4		
13 <sup>a</sup>	Gifu	IS-907	June 30	<b>146</b> 0	39.8	23.5		
14	Gifu	IS-907	July 20	2760	34.6	20.4		
15 <sup>a</sup>	Gifu	Peredovik	June 30	1500	41.5	23.5		
16 <sup>a</sup>	Okayama	Tainan No. 1	May 18	3880	42.4	27.4		
17	Okayama	Tainan No. 1	June 26	1990	37.7	25.4		
18	Okayama	Tainan No. 1	July 23	2340	24.5	19.7		
19 <sup>a</sup>	Okayama	IS-903	May 18	4220	45.0	27.4		
20	Okayama	IS-903	June 26	1630	35.2	25.4		
21	Okayama	IS-903	July 23	2290	30.6	19.7		
22 <sup>a</sup>	Okayama	IS-907	May 18	2960	41.1	27.4		
23	Okayama	IS-907	June 26	1420	34.6	25.4		
24	Okayama	IS-907	July 23	1720	31.1	19.7		
25 <sup>a</sup>	Okayama	Peredovik	May 18	2830	39.5	27.4		
26 <sup>a</sup>	Ehime	Tainan No. 1	June 2	2570	38.8	26,5		
27 <sup>a</sup>	Ehime	Tainan No. 1	June 17	3440	40.6	26.5		
28 <sup>a</sup>	Ehime	Tainan No, 1	July 7	3270	38.1	23.5		
29 <sup>a</sup>	Ehime	IS-903	June 2	2420	39.7	26.5		
30 <sup>a</sup>	Ehime	IS-903	June 17	3070	42.2	26.5		
31 <sup>a</sup>	Ehime	IS-903	July 7	2400	37.6	23.5		
32a	Ehime	IS-907	June 2	2420	40.9	26.5		
33a	Ehime	IS-907	June 17	2280	36.1	26.5		
34a	Ehime	1S-907	July 7	2200	37.1	23.5		
35a	Ehime	Peredovik	June 2	2130	39.7	26.5		
36 <sup>a</sup>	Ehime	Peredovik	June 17	2410	38.7	26.5		
37a	Ehime	Peredovik	July 7	2100	33.1	23.5		
38ª	Ibaraki	Tainan No. 1	May 21	3940	45.0	24.2		
394	Ibaraki	15-903	May 21	3770	39.9	24.2		
404	IDaraki Ibaraki	15-907 Dama dana'i	May 21	2230	35.0	24.2		
414	Ibaraki	Claimal	May 21	3100	30.7	24.2		
42	Ibaraki Ibarali	Clairsol	May 21	2510	57.0	24.2		
45	IDaraki Ibarali	Armavirsky	May 21	2050	44.5	24.2		
44	Ibaraki	13-897 SP 254	May 21 May 21	2950	39.0 29.9	24.2		
43	Ibaraki	50-234 Surpred 212	May 21	2850	30.0 42.7	27.2		
40	Ibaraki	SUIDICU 212 SR-212	May 21	2000	43.7	24.2		
	Ibaraki	JD-212	May 21	3300	27.0 22 1	24.2		
10	Ibaraki	155allKa 16-9044	May 21	7700	33,1 40 1	24.2		
77 50	Ibaraki Ibaraki	13-07++ Zelentra 260	May 21	5700	36 5	24.2		
50	Ibaraki	Inro 65-01	May 21	2740	30.5	27.2		
52	Ibaraki	Vorhod	May 21	2/40	20.7	24.2		
52	Ibaraki	1S-7775	May 21	2120	37.4	24.2 24.2		
55	IUaraKi	15-7773	May 21	3130	57.0	27.4		

<sup>a</sup>The values of these samples were utilized for the analysis of variance.

<sup>b</sup>Fresh seed weight basis.

<sup>c</sup>Average temperature during maturation of seed.

On the other hand it was reported that the oil content of sunflower in a climate chamber decreased with the increase of temperature from 10 to 35 C (6). It seems that the effects of temperature on lipid content may be influenced by the other environmental conditions.

#### **Fatty Acid Composition**

Fatty acid composition of 53 samples of sunflower seeds are shown in Table II. Palmitic acid ranged from 4.2 to 6.2% and stearic acid from 2.4 to 6.0%. Linoleic acid ranged from 43.8 to 75.4% and oleic acid from 13.6 to 49.9%. There were significant differences in linoleic acid percentage among Tuinan No. 1, IS-903, IS-907 and Peredovik; IS-907 showed the highest percentage. The combined percentage of oleic acid and linoleic acid was  $90.3 \pm 2.0\%$ , but the ratio of the two acids largely varied with the planting location and date. It seems that the environmental conditions largely influenced the fatty acid synthesis, though the characteristic fatty acid patterns were under genetic control.

The lipid of seed grown at a higher temperature is known to contain less unsaturated fatty acids (4-9, 11), and the same relation was observed in this study. Percentage of linoleic acid of four varieties is plotted in Figure 1 against the average temperature during maturation of seed. The percentage of linoleic acid was negatively correlated with the temperature (r = -0.886) and the percentage of oleic

## TABLE II

#### Tocopherol Content and Fatty Acid Composition of Sunflower Seed Lipid

Sample <sup>a</sup> no.		Tocopherol content (μg/g lipid)			Fatty acid composition (wt %)									
	α	β	γ	δ	14:0	16:0	16:1	18:0	18:1	18:2	18:3	20:0	20:1	22:0
1	648	9	_		0.1	5.5	0.1	3.3	27.5	62,8	tr	0.2	0.1	0,4
2	630	11	-	~~ .	0.1	5.7	0.1	3.0	22.3	68.1	tr	0.3	0.2	0.4
3	561	8	-		tr	5.1	0.2	4.9	21.1	68.0	tr	0.3	tr	0.5
4 <sup>D</sup>	425	8	8		tr	4.8	0.1	5.1	20.1	69.2	tr	0.3	tr	0.5
5	868	24	15	tr	tr	4.3	tr	6.0	24.7	64.1	tr	0.3	tr	0.5
6 <sup>D</sup>	552	20	10		tr	4.8	0.1	5.3	18.7	70.0	tr	0.4	0.2	0,4
70 ab	736	20	17	tr	0.1	5.4	0.1	4.3	15.2	74.1	0.1	0.3	0.1	0.5
80 ob	682	14	~		tr	5.1	0.2	5.1	18.7	70.1	tr	0.3	tr	0.5
10	589	13	2		tr	4.7	tr	3.9	28.3	62.1	0.1	0.3	0.2	0.4
10	724	23	4		tr	4.8	tr	4.5	22.1	67.6	tr	0.3	0.1	0.5
110	723	10	/		tr	4.9	tr	5.1	31.8	57.6	tr	0.3	tr	0.3
12	274	19	3		0 1	4.2	tr ••	4.7	23.8	00. <del>4</del>	UT The	0.3	tr	0.5
130	776	10	5		0.1	5.5	Cr tr	3.4 7 5	21.7	08.0		0.2	0.1	0.4
15b	602	20	5		0.1	5.1	tr	5.5	19.7	70.8 64 1	0,1	0.2	01	0.0
16b	618	11		_	0.1	3.0	0.2	7.2	23.0	4.1	11 +r	0.3	0,1	0.4
17	764	22			0.1	5.0	0.2	3.0 4 3	25 2	54.4	11 tr	0.5	0 1	0.5
18	445	17			0.1	6.1	01	4.5	14.3	74.5	01	0.7	0.1 tr	0.5
19b	548	16	_		0.1	5 1	tr	3.4	45.0	45.8	0.1 tr	0.3	<u><u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u></u></u>	0.5
20	437	16			tr	48	0.2	44	34 3	54 7	0.2	0.5	5.2 fr	0.5
21	546	16	_		tr	5 3	tr	5 1	14.8	73 5	0.1	0.5	0.1	0.7
22b	589	21	_	tr	0.1	5.0	0.3	2.6	44.9	46.4	tr	0.4	03	tr
23	373	16	-		0.1	5.3	0.1	3.4	30.4	59.8	tr	0.4	0.1	0.5
24	737	18	_		0.1	6.2	0.1	3.7	13.6	75.4	0.1	0.4	tr	0.4
25 <sup>b</sup>	304	20	-		0.1	5.1	0.1	4.0	49.9	40.1	tr	0.4	tr	0.4
26 <sup>b</sup>	543	18	_		0.1	5.1	0.1	3.2	44.5	46.2	tr	0.3	0.1	0.5
27 <sup>b</sup>	654	17	_		tr	4.9	0.1	3.2	42.2	48.8	tr	0.2	0.2	0.4
28 <sup>b</sup>	651	18	7	-	tr	4.3	tr	3.9	41.7	49.2	tr	0.3	tr	0.5
29 <sup>b</sup>	608	20	-	-	0.1	5.3	0.1	3.3	37.3	53.2	tr	0.3	0.1	0.4
30 <sup>b</sup>	700	24	3	-	0.1	4.8	0.1	3.1	44.4	46.7	tr	0.2	0.1	0.4
31 <sup>b</sup>	624	21	6	-	tr	4.4	tr	4.1	38.0	52.5	tr	0.3	0.1	0.6
32 <sup>b</sup>	637	25	-		0.1	4.9	0.1	2.6	42.9	48.7	tr	0.2	0.2	0,4
33 <sup>D</sup>	664	21	-		0.1	4.9	0.1	2.8	39.6	51.8	0.1	0.2	0.1	0.4
34 <sup>D</sup>	641	26	-	-	tr	5.1	tr	3.0	29.4	61.3	tr	0.5	tr	0.6
35 <sup>b</sup>	746	35		-	tr	5.6	0.2	3.1	40.0	49.9	tr	0.2	0.1	0.8
36 <sup>D</sup>	772	28	7		tr	5.4	0.2	3.9	45.7	43.9	tr	0.4	0.1	0.4
370	792	28	11	tr	tr	4.4	tr	4.2	43.4	47.0	0.1	0.2	0.1	0,5
38 <sup>D</sup>	835	29	12	tr	0.1	4.9	tr	3.1	41.2	49.9	tr	0.3	0.1	0.6
390	778	19	-		tr	5.0	tr	3.3	38.8	51.9	tr	0.3	0.1	0.5
400	790	20			0.1	5.4	0.1	2.9	36.3	54.4	tr	0.3	0.1	0.4
410	948	45	10	tr	0.1	5.3	0.1	3.2	38.6	51.9	0.1	0.3	0.1	0.5
42	852	34		-	0.1	5.3	0.1	3.0	40.3	50.3	0.1	0.2	0.1	0.5
43	1065	39	7		tr	4.7	tr	3.0	46.0	45.3	tr	0.2	0.1	0.5
44	794	27		-	0.1	5.4	0.1	2.6	34.7	56.7	tr	0.2	tr	0.3
45	848	27	-	-	0.1	5.1	0.1	2.6	33.2	58.1	0.1	0.2	0.1	0.4
40	671	33	10	_	0.1	5.1	tr	3.0	47.2	43.8	tr	0.3	0.1	0.5
47	//5	48	12	9	0.1	5.3	0.1	2.9	43.2	47.7	tr	0.2	tr	0.5
48	800	22	_	-	tr	5.2	0.1	3.1	37.5	53.6	tr	0.2	tr	0.4
47 50	709	20	_	-	tr	5.5	tr	2.7	42.2	49.0	tr	0.2	tr	0.5
51	916	21	_		0.1	5.2	tr	3.1	41.1	49.7	tr	0,2	0,1	0.5
52	610	20	_	_	0.1	3.3	tr	3.3	43.9	40.5	tr	0.3		0.6
53	843	37	_	_	0.1	5.0	0.1	2.1	44.1	47.0	UT	0.2	0.1	0.3
55	073	57	_	-	0.1	5.5	0.1	2.4	43.4	40.0	u	0,2	0.2	0.3

<sup>a</sup>The same number as Table I.

<sup>b</sup>The values of these samples were utilized for the analysis of variance.

acid was positively correlated (r = 0.873). The decrease of 1 C corresponded to the increase of 3.5% of linoleic acid. It was reported that linoleic acid content showed best correlation with average minimum temperature (r = -0.84) (10), while in this study correlations with average temperature, average minimum temperature and average maximum temperature were at the same level (r = -0.866, -0.867 and -0.857, respectively).

The combined percentage of linoleic acid and oleic acid was constant at ca. 90% and was not influenced by temperature. Thus it may be reasonable to speculate that the temperature during maturation of seed influenced the desaturation of oleic acid to linoleic acid. These effects of temperature have accounted for a higher concentration of oxygen available to desaturation and induction of desaturase activity at a lower temperature (12,13). This response to the temperature also has been thought to play an important role in the mobility of lipids (14). The sunflower oil with desired fatty acid composition

The sunflower oil with desired fatty acid composition can be obtained by selecting the planting location and date. The oil from sunflower grown in the hot climate of southern regions or planted early in the season will be rich in oleic acid if it is quite warm during maturation; it will therefore be for a frying oil (8,15). The oil rich in linoleic acid will be obtained from sunflower grown in the cool climate of northern regions or planted late in the season, and will be suitable for a salad oil.



FIG. 1. Effect of average temperature during maturation on fatty acid composition. △, Tainan No. 1; ○, IS-903: ●, IS-907; □, Peredovik.

#### Tocopherol

Tocopherol contents of sunflower seed lipid were successfully determined by HPLC (Fig. 2);  $\alpha$ -tocopherol was predominant and accompanied by small amounts of  $\beta$ -,  $\gamma$ - and  $\delta$ -tocopherol (Table II).

There have been many reports (16-18) on tocopherol content of sunflower seed or oil, but the presence of  $\beta$ tocopherol was not reported except for the study by Müller-Multon (19), probably because the separation of  $\beta$ - and  $\gamma$ tocopherol was inadequate in those studies. a-Tocotrienol was not detected in mature seed in this study, while Dompert et al. (6) reported that  $\alpha$ -tocotrienol was present in developing sunflower seeds and disappeared 40 days after the pollination. It is generally observed that non- $\alpha$ -tocopherols are predominantly contained in storage tissues such as fruits and seeds (20), and that oil seeds usually contain a significant amount of  $\gamma$ - or  $\delta$ -tocopherol in comparison with  $\alpha$ -tocopherol (21). It is interesting that sunflower seed contained predominantly  $\alpha$ -tocopherol and that the same tocopherol pattern is observed in safflower oil and olive oil which belong to the oleic-linoleic oil group (22).

Two-way analysis of variance showed that there was no significant difference in  $\alpha$ -tocopherol content among the four varieties and that  $\alpha$ -tocopherol content was largely dependent upon the planting location and date. There was no correlation between  $\alpha$ -tocopherol content (fresh seed weight basis or total lipid basis) and the following factors: seed yield, lipid content, the percentage of linoleic acid and the temperature during maturation. A positive correlation has been found between tocopherol content and unsaturated fatty acids content, and tocopherol played an important role in preventing the oxidative deterioration of polyunsaturated fatty acid of seeds (20). However, sunflower seeds grown at 28 C contained more tocopherol and less linoleic acid than those grown at 12 C (7). Dompert et al. (6) have investigated the lipids of sunflower seeds grown in a climate chamber and found that the total tocopherols were synthesized parallel to total lipids and were not sig-



FIG. 2. Chromatogram of tocopherols in sunflower seed lipid.

nificantly influenced by the temperature, whereas fatty acid composition was largely dependent on temperature. Considering these reports and the results obtained in this study, it cannot be concluded that tocopherol content positively correlates with the unsaturated fatty acids content.

Marquard et al. (23) investigated the tocopherol content of linseed grown in a phytotron. Each of the climatic factors such as photoperiod, humidity and temperature had an effect on tocopherol content and this effect was varied with the other two factors. Further study is needed to clarify the effects of environmental conditions on tocopherol synthesis in seeds.

It has been thought that the vitamin E requirement of animals increased with the consumption of a diet high in polyunsaturated fatty acids (PUFA). Tocopherol contained in a vegetable oil is an important natural antioxidant. Harris et al. (24) proposed a value of 0.6 mg  $\alpha$ -tocopherol per gram PUFA as a vitamin E requirement of humans. The raw sunflower seed oils of this study contained 0.67-2.67 mg  $\alpha$ -tocopherol per gram linoleic acid and these values satisfied the vitamin E requirement proposed by Harris et al. However, it is necessary to take into account the fact that a significant amount of tocopherol in the raw oils is removed or destroyed during the refining process. It might be important to increase  $\alpha$ -tocopherol content of sunflower seed by plant breeding in order to enhance both the nutritive value and the oxidation stability.

#### ACKNOWLEDGMENT

The National Federation of Agricultural Cooperative Association of Japan supplied sunflower seeds.

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[Received October 26, 1982]

## \*Rapid Extraction of Canola Oil

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#### ABSTRACT

The simultaneous size reduction and solvent extraction of canola seeds were studied using a laboratory blender and a small, pilot-scale Szego mill. The laboratory tests established that over 95% of the oil may be removed from the seed in a single contact stage. The effects of contact time and solvent-to-seed ratio were investigated. The extraction equilibrium favored the extraction of the oil at higher solvent-to-seed ratios. In all cases the extraction reached some 90% of the equilibrium value after 3 min. Runs in the Szego mill, which is a unique orbital mill developed by one of us (O. Trass), confirmed that solvent grinding is an efficient extraction technique. In this equipment, contact times as short as 30 sec give significant extraction, with the system approaching equilibrium in one minute. The Szego mill appears to be suitable for the rapid extraction of edible oil seeds such as rapeseed.

#### **BACKGROUND AND OBJECTIVES**

The current rapeseed extraction technology is based on percolating bed extractors. The oil content of the seed is removed, usually after prepressing, by contact with hexane, which percolates through large voids between "flaked" seed particles. This technology was developed for soy extraction, but unfortunately neither rapeseed nor the new canola varieties form strong flakes. Accordingly, their extraction requires significantly larger equipment than an equivalent soy-crushing plant.

The rate of solvent extraction is controlled by the solid particle size and the solvent-to-seed ratio. The reduction of particle size increases the surface area, and decreases solvent penetration pathlengths; thus resulting in significantly increased oil transfer rates into the miscella. Similarly, increasing the solvent-to-seed ratio provides a larger concentration driving force for extraction. Unfortunately, both of these approaches are energy-intensive: grinding energy must be provided for size reduction, while the increase in solvent-to-seed ratio results in larger heat requirement during solvent recovery.

Solvent grinding is a technique that may take advantage of the high mass transfer rates characteristic of small particle sizes without prohibitive energy expenditure. Complete extraction may be achieved in shorter time resulting in a more compact and, possibly, significantly less expensive equipment. Grinding also eliminates the need for breaking down the cellular structure of the seed by cooking. Thus it may be possible to balance some or most of the grinding energy requirements against the energy required in the cooking. Thus it may be possible to balance some or most of the grinding energy requirements against the energy required in the cooking step. The technique could be applied to some of the recently developed techniques of seed preparation, resulting in improved oil and protein products from rapeseed, such as the FR1-75 process developed by Jones and Holme (1).

The novel slurry mill (the Szego mill) developed by Trass and his group at the University of Toronto (2) seems especially well suited to the solvent grinding of rapeseed.

## **EXPERIMENTAL TECHNIQUES**

#### Laboratory Blender

A 2-L Waring blender was used to determine equilibrium extraction data and the best solvent-grinding conditions. Canola seeds were first ground with a Philips' coffee grinder, then an aliquot was placed in the blender. Freon 113 (200 mL) was added and the mixture blended at low speed for up to 2 min. In runs requiring longer blending times, the vessel was cooled under tap water for one minute after each minute of blending beyond the initial two minutes, in order to remove the heat generated by the blending process.

The resulting slurry was vacuum-filtered on a Buchner funnel. The filtrate was collected, and the solvent removed by a rotary vacuum evaporator. The weight of oil and cake were measured, and corrected for the weight of miscella entrained in the filter cake. Residual oil in the meal was measured by the AOCS standard method (3). Particle size distributions were obtained on a Rotap shaker using standard Tyler sieves.

Blending time and solvent-to-seed ratio were varied. The data obtained were used to derive the equilibrium conditions for wet extraction.

Throughout these runs Freon 113 (1,1,2-trichloro-1,2,2 trifluoroethane) was used as a solvent, since it is similar in polarity and boiling point to hexane, yet it is not flammable, and therefore may be safely used without extensive explosion proofing of equipment.

#### Szego Mill

The Szego mill is a unique orbital mill developed by one of