Thermal Stability and Frying Performance of Genetically Modified Sunflower Seed (*Helianthus annuus* L.) Oils

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High-oleic sunflower seed oils containing varying levels of oleic acid were evaluated in comparison to conventional sunflower and olive oils, under both thermoxidative and frying conditions. Analytical determinations included quantitation of triglyceride species and polar compound level and distribution. Total polar compounds were significantly lower in high-oleic sunflower oils as compared to conventional sunflower oil. This was essentially attributable to oxidized and polymeric compounds. No differences were found between high-oleic sunflower oils and olive oil, which could not be anticipated on the basis of fatty acid composition but might be otherwise related to differences in triglyceride distribution. After drying, oils and lipids extracted from fried potatoes did not differ significantly in polar compounds. Overall, results showed an excellent behavior of high-oleic sunflower oils with regard to thermoxidation and frying.

INTRODUCTION

Vegetable fats and oils are widely used for deep frying and are among the most important materials in the food service industries (Orthoefer, 1987). The maximum fry-life of a food service fat requires the selection of the proper oil or fat having a high stability, and in consequence, it is common to use partially hydrogenated vegetable oils, with low contents of polyunsaturated fatty acids (Haumann, 1987). However, fatty acid profiles of various deep-fat fried foods show appreciable amounts of trans unsaturated fatty acids which should be reduced because of the suspected negative nutritional and health effects of such acids (Smith et al., 1985).

An interesting alternative for frying is the use of genetically modified seed oils with low contents of polyunsaturated fatty acids, high-oleic oils being one of the most interesting possibilities to take into account (Orthoefer, 1988). High-oleic sunflower seeds have been grown commercially in the United States since 1984 (Purdy, 1986). Their nutritional and chemical characteristics have been defined (Purdy, 1985, 1986; Yodice, 1990), the latter ones being typical of conventional sunflower oils, except for those analytical characteristics associated with fatty acid composition.

The purpose of this study is to determine the behavior of high-oleic sunflower oils at high temperature both in the absence of foods and in frying conditions. Objective analytical methods were used to define total alteration, oxidation, and polymerization.

MATERIALS AND METHODS

Samples. Three samples of refined and genetically modified high-oleic sunflower seed (*Helianthus annuus* L.) oil (HOSO) identified as A, B, and C were supplied by industrial manufacturers. These oils were obtained and used without additives. In addition, conventional refined sunflower (SO) and olive oil (OO) samples were used for comparative purposes. Chemical characteristics of the initial oils are presented in Table I. Potatoes (*Solanum tuberosum* L. cv. Kennebec) used for frying experiments were purchased locally.

Treatment of Samples. (*a*) *Thermal Oxidation.* Thermoxidative treatment of samples was carried out under strictly controlled conditions. Oil samples of approximately 10 g were heated in beakers at 180 ± 1 °C for 5 and 10 h in triplicate. The surface/volume ratio established was 0.7 cm^{-1}. To minimize differences in the treatment, heating operations were effected simultaneously in all samples.

(*b*) *Frying Procedure.* Frying experiments were carried out in commercial electric fryers. Potatoes were peeled, cut into homogeneous strips, and washed with water. Fryers were filled with 600 mL of each of the frying oils (surface/volume ratio of 0.4 cm^{-1}). Fifteen batches each of 100 g of potato strips were fried for 6 min, and intervals of 15 min were established between frying operations. Thus, oils were heated over a total period of 5 h resembling one of the thermoxidative treatments mentioned above. Temperature was monitored during the experiment, being always below 200 °C during intervals and higher than 140 °C right after potatoes were immersed in the frying oil. No replenishments of oils were made during frying. Samples of frying oil and fried potatoes were taken out after the 15th frying operation and were stored at −30 °C for further analyses.

Lipid Extraction. Fried potato lipids were obtained by Soxhlet extraction using diethyl ether as solvent (Asociación Española de Normalización, 1991).

Analytical Procedures. Free fatty acid content (FFA, acidity expressed as oleic acid) was determined by titration (AOCS, 1988a) with 0.02 N KOH in alcohol.

Oxidative stability was determined according to the active oxygen method (AOCS, 1988b).

Fatty acid composition was carried out by gas–liquid chromatography following transesterification of the samples with CH_3ONa and $HCl–CH_3OH$ (Metcalfe and Schmitz, 1961).

Triglyceride composition was carried out by gas–liquid chromatography. A Chrompack CP 9000 chromatograph fitted with a fused silica column coated with Crompack Tap and a flame ionization detector was used. The temperature of the detector was held at 365 °C, and the injection port temperature was 360 °C. The carrier gas was helium. The column temperature was programmed to start at 350 °C for 1 min, increase from 350 to 360 °C at a rate of 0.5 °C/min, and hold at 360 °C for 6 min. The sample injected was 1 μL (5 mg/mL in hexane). Triglycerides were quantitated from percentual triglyceride composition and values of nonaltered triglycerides determined by column chromatography, as indicated hereafter.

Total polar compounds were determined by silica column chromatography, following the method proposed by the IUPAC (Waltking and Wessels, 1981) with two slight modifications: petroleum ether–ethyl ether 90:10 was used to elute the nonpolar fraction (nonaltered triglycerides) to obtain a sharper separation, and a final elution of the column was made with CH_3OH to improve recovery of the sample. Polar compounds were evaluated in triplicate, and significant differences among oils were analyzed by Student's *t*-test ($P \leq 0.05$).

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Table I. Chemical Characteristics of the Initial Oils

sample[a]	induction time, h	free fatty acids, % oleic acid	fatty acid composition,[b] %				
			$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	others
SO	8.9	0.12	6.5	5.1	22.3	65.0	1.1
HOSO-A	21.5	0.09	5.6	4.3	60.3	28.3	1.5
HOSO-B	25.8	0.11	4.5	4.3	75.2	15.2	0.8
HOSO-C	31.1	0.07	4.7	4.0	80.8	9.5	1.0
OO	28.5	0.16	10.1	2.9	75.7	9.5	1.8

[a] Abbreviations: SO, sunflower oil; HOSO, high-oleic sunflower oil; OO, olive oil. [b] Major fatty acids are listed.

Table II. Quantitation of Polar Compounds before and after Heating for 5 and 10 h in the Absence of Food (Weight Percent of Oil)

sample[a]		polar compounds[b]				
		total	TGD	OX TG	DG	FA[c]
SO	initial	5.8 ± 0.26[d]	1.5 (25.5)[f]	2.2 (39.0)	1.6 (27.2)	0.5 (8.3)
	5 h	24.2 ± 2.60[e]	13.9 (57.5)	8.2 (34.0)	1.6 (6.5)	0.5 (2.0)
	10 h	39.7 ± 1.17	26.3 (66.1)	11.0 (27.8)	1.8 (4.6)	0.6 (1.5)
HOSO-A	initial	3.3 ± 0.18	0.3 (8.5)	1.3 (38.5)	1.0 (32.1)	0.7 (20.9)
	5 h	18.4 ± 1.87	9.5 (51.7)	7.0 (38.3)	1.3 (6.8)	0.6 (3.2)
	10 h	29.4 ± 2.44	16.9 (57.6)	10.3 (35.0)	1.5 (4.9)	0.7 (2.5)
HOSO-B	initial	3.5 ± 0.14	0.3 (8.9)	1.0 (27.6)	1.5 (42.1)	0.7 (21.4)
	5 h	17.0 ± 2.42	8.3 (48.6)	6.4 (37.4)	1.6 (9.9)	0.7 (4.1)
	10 h	27.7 ± 1.54	15.6 (56.4)	9.3 (33.6)	2.1 (7.5)	0.7 (2.5)
HOSO-C	initial	3.5 ± 0.17	1.3 (35.8)	0.8 (22.9)	1.2 (35.2)	0.2 (6.1)
	5 h	11.1 ± 1.25	5.6 (50.3)	4.1 (36.8)	1.2 (10.7)	0.2 (2.2)
	10 h	28.9 ± 1.45	16.3 (56.5)	10.0 (34.4)	2.1 (7.3)	0.5 (1.8)
OO	initial	4.0 ± 0.12	0.5 (11.2)	0.6 (15.0)	2.5 (61.5)	0.5 (12.3)
	5 h	19.0 ± 1.39	8.9 (46.8)	7.2 (37.7)	2.6 (13.7)	0.3 (1.8)
	10 h	28.8 ± 1.13	14.6 (50.6)	10.9 (38.0)	2.7 (9.5)	0.6 (1.9)

[a] Abbreviations as in Table I. [b] Abbreviations: TGD, triglyceride dimers; OX TG, oxidized triglycerides; DG, diglycerides; FA, fatty acids. [c] Also includes a part of unsaponifiable matter. [d] Means ± SEM of three determinations. [e] Means ± SEM of three heated samples. [f] Values within parentheses are expressed as wt % on total polar compounds.

Table III. Changes in Major Triglycerides before and after Heating for 5 and 10 h in the Absence of Food (Weight Percent of Oil)

triglyceride	SO[a]			HOSO-A			HOSO-B			HOSO-C			OO		
	initial	5 h	10 h	initial	5 h	10 h	initial	5 h	10 h	initial	5 h	10 h	initial	5 h	10 h
PPO[b]	0.3	0.4	0.3	0.6	0.5	0.5	0.4	0.4	0.3	0.6	0.5	0.6	2.9	3.0	2.7
PPL	1.3	1.2	1.0	0.6	0.4	0.4	0.4	0.3	0.2	0.5	0.4	0.4	0.9	0.7	0.7
PPO + PLS	3.3	3.0	2.4	9.4	8.5	7.8	10.6	9.7	9.0	10.0	9.4	8.7	23.2	21.2	18.9
POL	6.4	5.4	4.9	4.9	4.1	3.6	1.9	1.5	1.3	1.7	1.5	1.1	5.7	4.4	3.6
PLL	11.5	8.6	7.6	3.8	3.0	2.4	2.0	1.6	1.1	0.8	0.6	0.4			
SOO	1.3	1.3	0.9	6.7	6.1	5.6	10.5	8.4	7.1	10.6	8.9	8.2	5.4	4.7	4.5
OOO + OSL	4.6	4.2	3.0	39.7	34.5	31.2	54.9	50.6	45.8	60.4	57.8	45.9	44.3	37.0	32.8
OOL + SLL	16.8	13.5	11.5	12.4	9.5	7.8	4.7	3.3	2.2	5.9	4.9	2.6	9.8	6.9	5.7
OLL	22.5	17.8	14.4	11.0	7.8	6.1	4.5	2.5	1.6	2.4	1.6	0.8	2.0	1.2	0.7
LLL	23.3	16.0	12.0	5.7	4.0	2.7	4.6	2.4	1.4	2.3	0.9	0.4	0.1		
others	2.8	3.8	1.6	1.8	2.8	1.9	1.8	1.9	1.6	1.0	1.9	1.2	1.5	1.2	0.9
nonaltered triglycerides[c]	94.1	75.2	59.6	96.6	81.2	70.0	96.3	82.6	71.6	96.2	88.4	70.3	95.8	80.3	70.5

[a] Abbreviations of oils as in Table I. [b] Abbreviations: P, palmitic acid; O, oleic acid; L, linoleic acid; S, stearic acid. [c] Gravimetric determination by silica column chromatography (Waltking and Wessels, 1981).

Distribution of polar compounds was performed by HPSEC (Dobarganes et al., 1988). The samples were analyzed in a Konik 500 A chromatograph with a 10-μL sample loop. A Hewlett-Packard 1037 A refractive index detector, and two 100- and 500-Å PLGel columns (Hewlett-Packard) connected in series were used. The columns were 30 cm × 0.75 cm i.d., with polystyrene–divinylbenzene highly cross-linked macroporous spherical packing (5 μm). HPLC grade tetrahydrofuran served as the mobile phase with a flow rate of 1 mL/min, and the sample concentration was between 15 and 20 mg/mL in tetrahydrofuran.

RESULTS AND DISCUSSION

Table I shows the general chemical characteristics of the initial oils. Genetically modified sunflower seed oils differed in levels of both oleic and linoleic acid, whereas there was no appreciable variation in the combined level of oleic acid and linoleic acid. Olive oil, used for comparative purposes, had a similar oleic acid concentration to high-oleic sunflower oil B and the same concentration of linoleic acid as in high-oleic sunflower oil C. As expected, induction times were closely related to the unsaturation degree of oils; hence, conventional sunflower oil showed the significantly lowest value.

Polar compound concentration and distribution in initial oils are presented in Table II, which also shows the values obtained after heating for 5 and 10 h in the absence of food. Reproducibility was excellent for total polar compound determination as shown in initial samples evaluated in triplicate, where the coefficient of variation was always lower than 5%. Nevertheless, when three samples of each oil were heated under strictly controlled conditions, standard deviations were generally greater, the coefficient of variation of the means ranging from 7.2% to 14.2%.

Table IV. Comparison of Experimental and 1,3-Random-2-Random Triglyceride Concentrations (Weight Percent of Oil)

	OOO + OSL[b]		LLL	
oil[a]	exptl	1,3-random, 2-random	exptl	1,3-random, 2-random
SO	4.6	5.7	23.3	26.7
HOSO-A	39.7	26.8	5.7	2.0
HOSO-B	54.9	45.9	4.6	0.3
HOSO-C	60.4	55.0	2.3	<0.1
OO	44.3	44.9	0.1	<0.1

[a] Abbreviations as in Table I. [b] OOO elutes at GLC retention time equal to those of OSL.

Similar results were obtained for the different groups of polar compounds.

Among initial oil samples, total polar compounds were present in similar levels except for conventional sunflower oil, with a significantly higher value than the others. In these cases, it was most important to examine the differences found in the distribution of the main groups of polar compounds. Thus, a distinct feature of olive oil vs sunflower oil was the diglyceride content, substantially higher in the former. This fact is probably a consequence of the refining process most crude olive oils are commonly subjected to because of their considerable hydrolytic alteration. During refining, free fatty acids are removed from crude oil, whereas diglycerides, in contrast, mostly remain in the oil due to their lower volatility. Dimer levels basically depend on the conditions used during the deodorization step and the oil unsaturation degree (Dobarganes et al., 1990). Therefore, from the values obtained it could be deduced that refining conditions were more drastic for high-oleic sunflower oil C than for the rest of the oils. On the other hand, oxidized triglyceride monomers, indicative of the total oxidation level (Pérez-Camino et al., 1990), were closely related to the unsaturation degree of oils.

Thermoxidative treatment in the absence of food during 5 and 10 h significantly increased total polar compounds in all samples. After 5 h of heating, the percentages of polar compounds in conventional sunflower oil (24.2%) and high-oleic sunflower oil C (11.2%) were significantly higher and lower, respectively, than that in the other samples. Nevertheless, after 10 h, conventional sunflower oil showed an exclusively significant highest value (39.7%), clearly beyond the polar compound level (25–30%) suggested as the upper limit for frying fats and oils in a number of Western countries (Firestone et al., 1991). As to polar compound distribution, it is clearly observed that the rise of total polar compounds is essentially attributable to oxidized and polymeric compounds. In contrast, diglycerides and free fatty acids, which are indicative of hydrolytic alteration, practically remained at initial levels. Interestingly, dimers plus oxidized triglycerides were by far much higher in conventional sunflower oil than in any of the other samples. Overall, unexpected results were found for high-oleic sunflower oils when compared to olive oil on the basis of their fatty acid compositions. After either 5 or 10 h, no differences were found between olive oil and those high-oleic sunflower oils with higher total unsaturation degree and greater linoleic content (A and B). In the case of high-oleic C, significantly lower values were obtained after a 5-h treatment, regardless of its containing the same level of linoleic acid as in olive oil (9.5%) plus the highest total unsaturation degree (91.3% unsaturated fatty acids). These results cannot be predicted from the initial quality characteristics, shown in Table I, though might be otherwise associated with differences in triglyceride composition. Although controversial results have been obtained in this respect, some interesting studies regarding the influence of triglyceride composition and structure on oxidative stability of oils have been reported (Wada and Koizumi, 1983; Park et al., 1983a–c; Tautorus and McCurdy, 1990; Neff et al., 1992).

Table III shows major triglyceride concentrations (weight percent of oil) and nonaltered triglyceride levels (last row) in initial oils and oils heated for 5 and 10 h in the absence of food, assuming an equal GLC-FID response factor for all molecular species. Triglyceride profiles of initial oils showed relevant differences between conventional olive or sunflower oils and those genetically modified. As expected, triglycerides of higher unsaturation degree showed a greater decrease after the thermoxidative treatment. As to initial oils, the most notable finding was the markedly high concentrations of monoacid triglycerides (OOO and LLL) in genetically modified oils, which were significantly higher than those theoretically expected on the basis of the 1,3-random-2-random distribution model (Vander Wal, 1960; Coleman, 1961). To illustrate this point, Table IV lists theoretical and experimental concentrations of unsaturated monoacid triglycerides. As can be observed, concentrations of the most unsaturated triglyceride (LLL) were approximately 2-, 15-, and 20-fold higher than expected in genetically modified sunflower oils A, B, and C, respectively, which involve an overall reduced level of triglycerides containing linoleic acid. Given that alterations take place in the unsaturated acyl groups of the triglyceride molecule and linoleic acid is the most susceptible fatty acid to undergo degradation in the oils under study, it might occur that a substantial proportion

Table V. Quantitation of Polar Compounds in Frying Oils and Fried Potato Lipids after 15 Frying Operations (Weight Percent of Oil)

		polar compounds[b]				
oil[a]	sample	total	TGD	OX TG	DG	FA
SO	frying oil	25.7	14.3 (55.8)[c]	9.3 (36.1)	1.6 (6.3)	0.5 (1.8)
	potato lipids	25.4	15.1 (59.5)	8.2 (32.4)	1.8 (7.0)	0.3 (1.1)
HOSO-A	frying oil	19.6	9.9 (50.3)	7.6 (38.8)	1.5 (7.8)	0.6 (3.1)
	potato lipids	19.9	10.0 (50.4)	7.5 (37.5)	1.7 (8.5)	0.7 (3.6)
HOSO-B	frying oil	18.2	9.1 (50.3)	7.1 (38.8)	1.5 (8.2)	0.5 (2.7)
	potato lipids	18.7	9.4 (50.1)	6.9 (37.0)	1.8 (9.4)	0.6 (3.5)
HOSO-C	frying oil	16.5	8.5 (51.2)	6.4 (39.0)	1.4 (8.4)	0.2 (1.4)
	potato lipids	16.8	8.7 (51.7)	6.4 (38.1)	1.4 (8.5)	0.3 (1.7)
OO	frying oil	18.3	7.9 (43.1)	7.3 (39.8)	2.6 (14.2)	0.5 (2.9)
	potato lipids	19.0	8.2 (43.1)	7.6 (39.8)	2.7 (14.2)	0.5 (2.9)

[a] Abbreviations as in Table I. [b] Abbreviations as in Table II. [c] Values within parentheses are expressed as wt % on total polar compounds.

of altered triglycerides in high-oleic sunflower oils contained more than one oxidized acyl group. However, evaluation of polar compounds does not permit differentiation of altered triglycerides with either one or more oxidized fatty acyl groups. Therein, similar levels of oxidized acyl groups in olive oil and high-oleic sunflower oils could result in comparatively lower percentages of polar compounds in the latter. Although differences in fatty acid triglyceride distribution could explain in part the results obtained, other compounds may also be involved. Thus, it has been reported that addition of certain vegetable oil unsaponifiables protects safflower oil from oxidative polymerization (Sims et al., 1972).

Table V shows quantitation of polar compounds in frying oils and potato lipids after 15 frying operations. Even though the total period of heating (5 h) was similar to that used for thermoxidative treatment, a lower alteration level might be expected due to the protective effect of the food and the decrease of temperature during the frying operation. However, frying oils surprisingly showed similar results to those found for samples subjected to thermoxidative alteration in the absence of food during 5 h (Table II). Additionally, as the surface/volume ratio was lower in frying operations than in thermoxidative treatments (0.4 vs 0.7 cm^{-1}), which would presumably involve lower alteration in the former, the results obtained may be associated with the higher temperature reached between frying operations. On the other hand, degradation appears to have taken place following a similar pattern given that oxidation and polymeric compounds significantly increased, while diglycerides and fatty acids remained at the initial levels. This seems to indicate that foodstuff moisture did not affect frying oils to an appreciable extent under the conditions used in this study. As occurred under thermoxidative conditions, high-oleic oils clearly showed better behavior toward alteration during frying than did conventional sunflower oil.

Similarly to previously reported results for deep-frying of frozen prefried foods (Sebedio et al., 1990; Pérez-Camino et al., 1991), no significant differences were found between frying oils and food lipids in either total polar compounds or polar compound distribution, which indicates that there was no preferential adsorption of altered oil compounds on the potato surface.

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