Performance of Sunflower Oil with High Levels of Oleic and Palmitic Acids During Industrial Frying of Almonds, Peanuts, and Sunflower Seeds

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ABSTRACT: High-oleic, high-palmitic sunflower oil (HOHPSO) is a seed oil from a new mutant sunflower line characterized by increased levels of both oleic acid (>50%) and palmitic acid (>25%) and a high oxidative stability. In this study, its performance at frying temperature was compared with that of palm olein in thermoxidative assays (4 h, 180°C). Also, industrial discontinuous frying of almonds, peanuts, and sunflower seeds (200 kg of each product) was carried out to define both the performance of HOHPSO and the main changes undergone by the foods. The evaluation of polar compounds and their distribution in the main groups, i.e., polymers, oxidized monomers, and DAG, as well as changes in tocopherols and oxidative stability, demonstrated the excellent behavior of HOHPSO during thermoxidation and frying. The increase in polar compounds and the loss of tocopherols and stability were much lower for HOHPSO than for palm olein under identical heating conditions. Only 1.3% polar compounds were formed during industrial discontinuous frying for 4 h and the oil stability increased, probably due to the formation of antioxidant compounds. As for the foods, the FA composition of the surface oil was clearly different from that corresponding to the internal oil, the former denoting the presence of HOHPSO in high concentration, particularly in fried sunflower seeds. Changes in oil stability of the foods attributable to the frying process clearly demonstrate the interest in using a highly stable oil such as HOHPSO to protect the surface against oxidation during food storage.

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KEY WORDS: Almonds, frying, high-oleic oil, high-palmitic oil, peanuts, sunflower oil, sunflower seeds.

The development of genetically modified oils in the last decade has contributed significantly to an increase in the production of natural oils with enhanced oxidative and thermal stability (1). The application of these stable oils in the frying process has the advantage of making the consumer preference for natural oils compatible with the thermal stability required by food preparation processes involving high temperatures (2).

Recently, a new sunflower mutant was developed that contains high levels of oleic and palmitic acids and a very low level of linoleic acid (3) The high-oleic and high-palmitic traits were fixed in the third generation from mutagenized seeds (M3) and in subsequent generations, and an open-pollinated pure line with high agronomical productivity was obtained and multiplied in the field. The characterization of the lipid classes in this mutant (4) and the chemical and physical properties of the oil (5,6) have been reported. The high oxidative and thermal stability found suggests a very good performance in discontinuous frying operations or in industrial frying with a high turnover period, in which the oil must be replaced frequently.

A frying process is used in the industrial preparation of an increasing number of foods. Among them, the production of snack nuts by frying is an alternative to dry-roasting. These high added-value products are the preferred snacks for exporting (7). The advantage of frying compared with dry-roasting is that the surface of the food can be protected against oxidation during storage by using a frying fat with higher stability than that of the nut or seed oil. However, from the point of view of frying fat degradation, two negative characteristics have to be considered. On one hand, in foods with a high lipid content such as almonds, peanuts, and sunflower seeds, a significant lipid interchange between the food and the frying fat is expected because the food surface is unprotected during frying. This is opposite to what happens in other lipidic foods, which are battered or breaded before being fried. On the other hand, because of the physical structure of these products and their noncontinuous oil distribution, frying fat absorption is low; hence, a highly stable frying fat or oil is needed to maintain frying oil quality and increase the stability of the fried product during storage. Surprisingly, studies giving information on changes during frying of these products are unavailable. Previous related studies focused on differences in composition and quality attributable to processing conditions (8,9) and on oxidation during storage (10–14).

The objective of this study was to determine the performance of high-oleic, high-palmitic sunflower oil (HOHPSO) during the industrial frying of almonds, peanuts, and sunflower seeds given the need for a stable oil in their preparation, and also to define the main changes these foods undergo.

EXPERIMENTAL PROCEDURES

Materials. Five tonnes of sunflower (*Helianthus annuus* L.) seeds from the mutant line CAS-12 (3) were obtained from plants grown in the field (southern Spain); the seeds were supplied by Advanta Seeds (Advanta, Marchena, Spain). Almonds, peanuts, sunflower seeds, and palm olein were supplied by SALYSOL, S.A. (Sevilla, Spain).

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Oil extraction and refining. HOHPSO was extracted and refined in the pilot plant of the Instituto de la Grasa. The seed oil was extracted using a continuous oil screw press (Mod. Lion-Expeller, Anderson Int., Cleveland, OH) with a processing capacity of 400 kg/h. The crude oil obtained was refined in a discontinuous pilot plant, 200 kg per batch. The oil was degummed in an agitation tank reactor with 0.2% (w/w) phosphoric acid for 30 min at 25°C. Neutralization was performed in the same reactor using 18°Baumé (4.18 M) lye, at room temperature for 40 min. Once the gums and soapstocks were separated, the oil was water-washed three times at 80°C and then bleached with 0.1% (w/w) silica (Trisyl; Grace, Columbia, MD) and 1% (w/w) bleaching earths (Fulmont; Süd-chemie, Munich, Germany), at 70°C under vacuum for 10 min. Deodorization was performed with 3% (w/w) steam at 240°C for 3 h under a vacuum of less than 3 mmHg.

Thermoxidation assay. Thermal oxidation was carried out in triplicate under strictly controlled conditions using a Rancimat apparatus (Metrohm Ltd., Herisau, Switzerland). Briefly, 8 ± 0.01 g of samples were weighed in Rancimat reaction vessels and inserted in the heating block previously heated at 180 ± 1°C. After heating for a period of 4 h, final samples were removed and maintained at –30°C until analysis. The Rancimat instructions were carefully observed for temperature correction. No bubbling of air was applied during heating, and the tubes were left open. This procedure was described in detail, including reproducibility data, in a previous publication (15).

Industrial frying. Industrial discontinuous frying was carried out under the conditions of temperature and time normally applied in the industry for the preparation of these products. Briefly, 425 L of oil was heated at 170°C in a discontinuous fryer (2×1 m^2 surface). A scheme of the fryer is shown in Figure 1. First, two frying operations with almonds (100 kg and 4-min frying each); second, two frying operations with peanuts (100 kg and 10-min frying each); and finally, two frying operations with sunflower seeds (100 kg and 4-min frying each) were carried out. Between frying operations, the oil was maintained for around 30 min without food, and the total oil heating period was 4 h. Samples of oil and fried product were removed after each frying operation and maintained at –20°C until analyses.

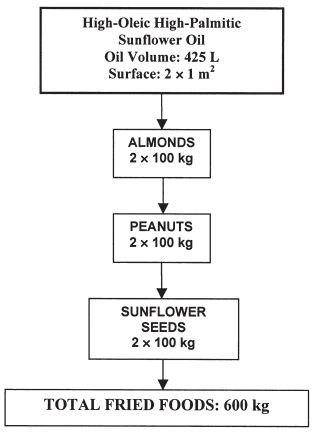

FIG. 1. Scheme of industrial frying experiments.

Analytical methods. Moisture was determined gravimetrically, starting from 20 g of raw and fried seeds, after grinding and freeze-drying the samples. Total food lipids were obtained by a 6-h Soxhlet extraction (16) using diethyl ether as the solvent. Dry lipid-free matter was calculated by subtracting the moisture and lipid content of each sample from 100.

The free oil fraction or surface oil was determined according to Sankarikutty *et al*. (17). Thus, 10 g of sample was added to 100 mL of light petroleum (60–80°C) and stirred for 15 min at 25°C with a magnetic stirrer. The extraction procedure was repeated twice. After filtration through anhydrous Na_2SO_4, the solvent was evaporated under reduced pressure and samples were then dried to a constant weight using a stream of nitrogen. After eliminating the surface oil, the samples were ground, and the internal oil was then determined as for the total food lipids.

Polar compounds and their distribution in polymeric TAG (dimers plus higher oligomers), oxidized TAG monomers, DAG, and FA were quantified by a combination of adsorption and high-performance size-exclusion chromatography (HPSEC) (18). Conditions applied for HPSEC of polar compounds were as follows: Sample solutions of 10–15 mg polar compounds/mL in THF were used for the analysis. A Rheodyne 7725i injector with a 10-μL sample loop and a Waters 510 HPLC pump (Waters Associates, Milford, MA); two 100- and 500-Å Ultrastyragel columns (Waters Associates), 25×0.77 cm i.d., connected in series and packed with a porous, highly cross-linked styrenedivinylbenzene copolymer (<10 μm); and a refractive index detector (Hewlett-Packard, Palo Alto, CA) were used. The procedure was standardized by the IUPAC Commission on Fats, Oils and Derivatives (19).

Natural tocopherols were determined by HPLC with fluorescence detection, following the IUPAC Standard Method (20).

FA composition was determined by GLC after derivatization to FAME with 2 N KOH in methanol, according to the IUPAC Standard Method (20).

FFA, PV, and oil stability index (OSI) were determined following AOCS standard methods (21).

Statistical methods. Data for thermoxidized samples are expressed as the mean of three experiments and the SEM. Data for initial oils are expressed as the mean of two analytical determinations and the SEM. Data for oil stability are expressed as the mean of samples extracted from two frying operations and the SEM. Differences between samples were assessed with Student's *t*-test, with probability values of 5% being statistically different.

RESULTS AND DISCUSSION

Changes in frying oils. Table 1 shows the main characteristics of the initial oil used in this study compared with those of a typical palm olein. Palm olein is becoming the oil of choice for major food manufacturers in many European countries because

TABLE 1
Physicochemical Characteristics of the Initial Oils

		HOHPSO	Palm olein
Oil stability index (h, 120°C)		15.3	12.6
FFA (%)		0.10	0.08
PV (meq/kg)		4.7	5.4
Tocopherols (mg/kg)		554	685[a]
Major FA (%)	C16:0	25.5	38.2
	C16:1	7.3	0.2
	C18:0	1.9	3.9
	C18:1	59.7	41.5
	C18:2	2.2	11.1

[a]Also includes tocotrienols. HOHPSO, high-oleic, high-palmitic sunflower oil.

of its good performance and naturally high oxidative stability (2). It is normally used for frying almonds, peanuts, and sunflower seeds in the industry, where the assays were carried out. As one can observe in Table 1, the oxidative stability was even higher for HOHPSO than for palm olein (15.3 vs. 12.6 h at 120°C) in spite of its significantly lower content of saturated FA (27.4% for HOHPSO vs. 42.1% for palm olein), which implies it has better nutritional properties. This was in agreement with the results of Guinda *et al*. (6), obtained with a HOHPSO with a similar FA composition but even higher stability (19 h at 120°C), probably due to its higher tocopherol content (870 mg/kg). The results seem to indicate the positive influence of the low content of linoleic acid (2.2% for HOHPSO vs. 11.1% for palm olein) on stability against oxidation. The levels of tocopherols and FFA, and the PV were typical of good-quality refined oils.

Table 2 summarizes the changes undergone by both oils under thermoxidative conditions, as described in the Experimental Procedures section. The themoxidative assays were intended to compare the performance of both oils under temperature and heating conditions similar to those applied in industrial frying. The initial level of polar compounds was high in palm olein because of its characteristically high content of DAG (22). Consequently, to define the performance of both oils at a high temperature, we considered the increase in polar compounds after heating. The results showed that HOHPSO had better performance at a high temperature, as its increase in polar compounds after 4 h of heating was significantly lower than that found for palm olein (2.5 vs. 7.3% for HOHPSO and palm olein, respectively). As expected, heating for 4 h did not modify the levels of DAG and FA because of the absence of moisture during thermoxidation. The main differences we observed between the initial and heated oils were in the increase of polymerization and oxidation compounds. In parallel, the loss of tocopherols and oxidative stability also reflected the better behavior of HOHPSO. Around one-third of the initial tocopherols remained in HOHPSO after heating, and oil stability was close to the initial value. In contrast, tocopherols and tocotrienols had practically disappeared in palm olein, and their stability against oxidation had decreased considerably.

Table 3 summarizes the results obtained for the frying oil during the industrial frying experiments. Each value corresponds to the average of those obtained for two frying operations of each product. As one can observe, the increase in polar compounds was very low in spite of the oil being unprotected by food during most of the heating period (23). Considering the present regulations in many European countries limiting the level of polar compounds to 25% in frying fats and oils (24), the increase of just 1.3% in 4 h is an indication of the high thermoxidative stability of HOHPSO and, consequently, of the potentially long time period for use without replacement. In contrast, the content of tocopherols remained close to the initial level for most of the heating period, and only a slight decrease was observed at the end of the experiment. Interestingly, the oil stability increased after frying, which seems to indicate the formation of antioxidant compounds soluble in the oil during frying. In this respect, this activity has been attributed to the compounds formed through interaction between proteins and either carbohydrates from the food or oxidized lipids from the frying oil (25–27).

Changes in food lipids. Table 4 shows quantitative changes in the major food components after frying. The results for fried products are expressed in terms of the food weight obtained from 100 g of prefried food by holding constant the dry, lipid-free matter. Thus, from the values in Table 4, both the loss of moisture and the net gain in lipids from each food can be determined directly by subtraction. As shown, in the case of almonds and peanuts, there was practically no change in total

TABLE 2
Changes During Thermoxidation for 4 h at 180°C[a]

	HOHPSO		Palm olein	
	Initial	Final	Initial	Final
Total polar compounds (%)	3.3 ± 0.10[a]	5.8 ± 0.14[b]	7.8 ± 0.15[c]	15.1 ± 0.21[d]
Polymeric TAG (%)	0.3 ± 0.03[a]	1.7 ± 0.06[b]	0.8 ± 0.10[c]	4.7 ± 0.12[d]
Oxidized TAG monomers (%)	0.5 ± 0.05[a]	1.6 ± 0.05[b]	1.1 ± 0.05[c]	4.8 ± 0.07[d]
DAG (%)	2.1 ± 0.05[a]	1.9 ± 0.03[a]	5.3 ± 0[b]	5.1 ± 0.05[b]
Tocopherols (mg/kg)	554 ± 2.8[a]	181 ± 3.6[b]	685 ± 4.9[c]	14 ± 1.2[d]
Oil stability index (h, 120°C)	15.3 ± 0.15[a]	12.9 ± 0.52[b]	12.6 ± 0.05[b]	4.4 ± 0.10[c]

[a]Final values are mean ± SEM (n = 3 experiments); initial values are mean ± SEM (n = 2 analysis). Values in rows with different superscript roman letters differ significantly ($P < 0.05$). For abbreviation see Table 1.

TABLE 3
Changes in HOHPSO After Frying Almonds, Peanuts, and Sunflower Seeds

	Initial	After frying almonds[a]	After frying peanuts[a]	After frying sunflower seeds[a]
Tocopherols (mg/kg)	554	537	551	431
Stability (h, 120°C)	15.3	19.5	18.2	17.1
FFA (%)	0.08	0.18	0.27	0.40
Total polar compounds (%)	3.3	3.6	4.1	4.6
Polymeric TAG (%)	0.3	0.5	0.7	0.8
Oxidized TAG monomers (%)	0.6	0.7	1.0	1.1
DAG (%)	2.0	1.9	2.0	1.9

[a]Mean values of oil samples extracted from the two lots of fried products. For abbreviation see Table 1.

TABLE 4
Major Changes During Frying of Almonds, Peanuts, and Sunflower Seeds (wt% on raw food basis)

	Almonds		Peanuts		Sunflower seeds	
Analysis	Raw	Fried	Raw	Fried	Raw	Fried
Moisture (%)	4.5	1.7	3.6	0.8	5.1	0.6
Lipids (%)	45.5	48.7	43.8	47.9	39.5	57.8
Dry lipid-free matter (%)	50.0	50.0	52.6	52.6	55.4	55.4
Total weight (g)	100	100.4	100	101.3	100	113.8

weight; consequently, the loss of moisture was parallel to the increase in lipids. However, the increase in lipids was substantially higher in sunflower seeds, possibly due to their high surface/weight ratio.

Table 5 shows a quantification of the major FA in the initial frying oil and in the total lipids extracted from the foods before and after frying. The final composition of the frying oil is omitted, as no differences in FA composition between the initial and final frying oils were found, thus indicating that contamination by oil from the food was not appreciable under the conditions applied. On the contrary, clear differences were found in the lipids of fried foods with respect to the raw products, showing the considerable uptake of frying oil by the product. This was clearly denoted, particularly by the increases of palmitic acid and also of palmitoleic acid in all fried samples For example, palmitoleic acid was absent from raw peanuts and sunflower seeds, whereas 7.3% was found in the frying oil. Consequently, its content in the fried foods is a quantitative indication of the proportion of frying oil in the fried foods and can easily be calculated (28). Thus, the contributions of frying oil were around 6, 8, and 18% for almonds, peanuts, and sunflower seeds, respectively.

A more detailed evaluation was obtained by differentiating the surface oil, accessible to organic solvents, in the intact seed and the internal oil extracted after milling. The distribution of lipids in the raw and fried foods is listed in Table 6, which shows that major quantitative changes took place in sunflower seeds. Table 7 shows the FA composition of the total, surface, and internal oil in both raw and fried foods. One can easily observe that, as expected, the FA composition of the internal oil was very similar to that found for raw products (Table 5). On the contrary, the composition of the surface oil was similar to that of the HOHPSO, particularly for sunflower seeds. From the content of palmitoleic acid, we deduced that the proportions of frying oil in the surface were 32, 48, and 84% for almonds, peanuts, and sunflower seeds, respectively.

The high proportion of frying oil on the surface of these foods could be a guarantee of the stability of fried foods during later storage, given the high stability of HOHPSO. In this respect, Figure 2 summarizes the changes in oil stability as a consequence

TABLE 5
Major FA (%) in the Frying Oil and in the Seeds Before and After Frying

Major FA	Frying oil	Almonds		Peanuts		Sunflower seeds	
(%)	(HOHPSO[a])	Raw	Fried	Raw	Fried	Raw	Fried
C16:0	25.5	6.4	7.7	10.9	12.9	4.7	8.8
C16:1	7.3	0.5	0.9	< 0.1	0.6	< 0.1	1.3
C18:0	1.9	1.1	1.1	3.9	3.8	4.0	3.6
C18:1	59.7	59.9	61.1	38.9	42.1	38.8	39.6
C18:2	2.2	27.8	26.0	35.7	34.0	48.2	43.5

[a]For abbreviation see Table 1.

TABLE 6
Distribution of Lipids in Almonds, Peanuts, and Sunflower Seeds: Total, Surface, and Internal Oil (%)

Lipids (%)		Almonds	Peanuts	Sunflower seeds
Total	Raw	45.5	43.8	39.5
	Fried	48.5	47.3	50.4
Surface	Raw	1.2	2.2	1.3
	Fried	3.4	5.9	7.0
Internal	Raw	44.5	40.8	37.7
	Fried	45.3	41.1	40.1

of the frying process. The OSI values obtained by the Rancimat apparatus at 120°C for the total oil and for the internal oil after eliminating the oil from the surface are reported for both raw and fried products. Two interesting results can be deduced from Figure 2. First, in the raw seeds, the OSI values for the total oil were lower than those obtained for the internal oil, the differences being significant for almonds and peanuts ($P < 0.05$). The opposite was found for fried products, for which OSI values in the three products were significantly higher for the total oil. In the case of raw products, the lower stability of the total oil might be explained by the presence of surface oil with the same composition as that corresponding to the internal oil but being more accessible to air. Even if the history of the raw almonds, peanuts, and sunflower seeds is unknown, a lower stability is to be expected in the unprotected surface oil, contributing to a decrease in the stability of the total oil. On the contrary, the high stability of the surface oil in fried foods, similar to that of HOHPSO, contributed to the increase in stability of the total oil. Second, the stability of oils after frying was significantly higher ($P < 0.01$) than that obtained for raw products. This difference cannot be explained only by the high stability of HOHPSO, because similar values should have been expected for the internal oils before and after frying. This enormous difference seems to indicate the formation of antioxidants soluble in the oil, probably due to interactions between carbohydrates and/or oxidized lipids and proteins at the high temperature of the frying process (25–27), as already found for the frying oil.

In sum, HOHPSO showed excellent performance during the frying of these products. Also, frying these products in high-stability oils could be very useful in protecting the food surface against oxidation during the storage period. Even more, the high stability of the HOHPSO increased after frying, probably because of the formation of compounds with antioxidant activity at the high temperature of the frying process.

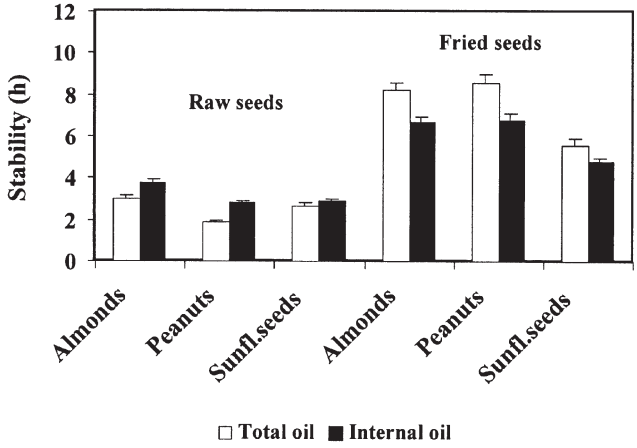

FIG. 2. Changes in oxidative stability attributable to the frying process.

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TABLE 7
Major FA (%): Differences Between Surface and Internal Oil Fractions in Fried Almonds, Peanuts, and Sunflower Seeds

	Almonds			Peanuts			Sunflower seeds		
Major FA (%)	Total	Surface	Internal	Total	Surface	Internal	Total	Surface	Internal
C16:0	7.7	13.8	7.1	12.9	19.2	11.7	8.8	22.9	6.3
C16:1	0.9	2.7	0.6	0.6	3.5	0.2	1.3	6.1	0.6
C18:0	1.1	1.3	1.3	3.8	2.8	3.7	3.6	2.2	4.0
C18:1	61.1	57.6	62.6	42.1	45.7	41.8	39.6	54.9	43.0
C18:2	26.0	20.5	27.8	34	22.2	35.2	43.5	10.5	43.1

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