Microwave and Conventional Heating Effects on Some Physical and Chemical Parameters of Edible Fats

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Effects of microwave energy and conventional heating on physical and chemical parameters of five edible oils and fats (virgin olive oil, olive oil, sunflower oil, high oleic sunflower oil, and lard) were investigated. These fats and oils were subjected to three well-controlled treatments: heating in conventional electric oven, heating by microwave energy, and exposure to microwave energy, respectively. The effect of microwave heating on the visible spectrum, K_{232} and K_{270} , density, viscosity, and squalene and *trans*-isomer contents of fats and oils was worse than that produced by heating the same fats in a conventional oven at the same temperature, time, surface/volume ratio, and light conditions. Subjecting fats and oils to microwave energy under the same conditions, but below 40 °C, did not produce considerable variations in the same parameters when compared to the original ones.

Keywords: Microwave oven; heating effect; chemical and physical parameters; edible fats and oils

INTRODUCTION

At present, >25% of households, in Europe, have a microwave oven (MO). It is also commonly used in the food industry for potato chip finishing (dryers), poultry cooking, and bacon precooking (Decareau, 1985; Kamel and Stauffer, 1993). There has been an increase in the use of MO in the past decade for food preparation (Tsuyuki, 1982), reflected in an increase in the number of food products at the market prepared specifically to defrost, heat, or cook in MO.

The effects of MO heating on the different components in foods can differ significantly from those produced by heating in a conventional oven (CO). For example, it has been speculated that free radicals can be formed by exposure to microwave energy (Lie-Ken-Jie and Yan-Kit, 1988), especially when high temperature is reached, as is the case when fatty foods are cooked. Fats have a great capacity for storing microwave energy, although they have a small dielectric loss (Mohsenin, 1984; Sowa, 1986). This is due to the fact that, compared with other foods, they also have a low specific conductivity and specific heat at constant pressure (Jowitt et al., 1983).

Few studies have been reported on the possible modifications in the chemical parameters as only a few years have elapsed since MO began to be used routinely in home cooking. Such publications that have appeared deal only with certain aspects related to the fats, such as the tocopherol content and the possible hydrolysis of these compounds (Yoshida et al., 1990, 1991a,b, 1992a, 1993). In other papers only animal fats were investigated (Hear et al., 1987; Yoshida et al., 1992b). In no case are data provided on the physical parameters of the fats and oils subjected to microwave treatment.

Contradictory results have been reported on the possible isomerization (formation of *trans*) in the double bonds of fatty acids as a consequence of exposure to microwave energy (Maga et al., 1977; Mai et al., 1980), and in no study have data appeared relating to the positional isomers.

In all of the studies involving microwave energy with the elevation of the temperature of fats, in no case have heat and microwave energy been treated separately to elucidate the reason for chemical and physical changes.

The objective of this work was to determine physical and chemical characteristics of edible fats and oils when heated in a microwave or conventional oven.

EXPERIMENTAL PROCEDURES

Samples. Five different edible fats and oils were used in this study. Commercially available sunflower oil (SO), high oleic sunflower oil (HOSO), olive oil (OO), and lard (LA) were purchased at a local market. Extra virgin olive oil (VOO) (EEC, 1991, Annex I), extracted from the Pajarera olive variety, was obtained from an industrial plant (Cano e hijos, Córdoba, Spain). The samples were treated in a microwave or conventional oven as follows:

(1) SO-MH. One hundred milliliters of sunflower oil was divided into 20 mL in five open Sovirel flasks (4 cm diameter), which were placed at equal distances on the rotatory plate of the microwave oven. They were heated for 120 min at half power (i.e. they were subjected to total microwave energy for 60 min). The temperature was determined every 30 min with a chromel–alumel thermocouple and remained at 170 \pm 10 °C.

(2) SO-CH. These samples were prepared as in the preceding paragraph but they were heated to 180 ± 2 °C by air convection in an electric oven for 120 min.

(3) SO-M. These samples were distributed as described for SO-MH and were subsequently exposed to microwave energy for 60 min, at intervals of 50 s below 40 $^{\circ}$ C.

The treated HOSO, OO, VOO, and LA were obtained in the same way as for SO as described for SO-MH, SO-CH, and SO-M to give HOSO-MH, HOSO-CH, HOSO-M, OO-MH, OO-CH, OO-M, VOO-MH, VOO-CH, VOO-M, LA-MH, LA-CH, and LA-M, respectively. All samples were completed in duplicate.

The study was completed two times using the methods described. Each value obtained is the mean of two measurements from two replicates.

Reagents and Solvents. All reagents and solvents used were of analytical grade.

Equipment. The microwave oven used was a Moulinex, Micro Chef FM B745 AGS (France). The full power was 1 kW, and the frequency of radiation was 2450 MHz.

A gas chromatograph, HP-5890 (Hewlett-Packard, Avondale, PA), was used with a flame ionization detector and a capillary column (60 m \times 0.25 mm i.d.). For analysis of *cis*– *trans* isomers of fatty acid methyl esters, the column was coated with a 0.20 μ m film thickness of stabilized liquid phase

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 Table 1. Fatty Acid Composition (Percent) in the Fats

 and Oils before Treatment

	sample				
fatty acid	voo	00	SO	HOSO	LA
14:0	nd ^a	nd	nd	nd	1.1
16:0	9.8	8.0	5.5	4.2	22.6
16:1 <i>n</i> -8	0.6	0.3	< 0.1	<0.1	1.8
17:0	nd	< 0.1	< 0.1	<0.1	0.4
17:1 <i>n</i> -8	nd	< 0.1	< 0.1	<0.1	0.4
18:0	3.5	3.8	4.9	4.5	13.8
18:1 <i>n</i> -9	74.8	79.7	34.1	78.8	42.9
18:1 <i>n</i> -7	2.2	1.7	0.6	0.8	3.4
18:2 <i>n</i> -6	7.6	5.1	52.9	9.3	11.5
18:3 <i>n</i> -3	0.6	0.4	0.1	0.1	0.7
20:0	0.5	0.5	0.4	0.4	0.2
20:1 <i>n</i> -9	0.3	0.3	0.2	0.3	1.1
22:0	0.1	0.2	1.0	1.2	nd
24:0	nd	<0.1	0.3	0.4	0.1

^a nd, not detected.

SP-2380 [poly(90% bis(cyanopropyl)–10% cyanopropylphenylsiloxane)]. A capillary column 60 m \times 0.25 mm i.d., coated with a 0.25 μm film thickness of liquid phase TR-140262 [poly-(ethylene glycol)] was used for analysis of the positional isomers of fatty acid methyl esters.

Analytical Procedures. Determination for Chemical Properties. Determination for *trans* isomers of fatty acid methyl esters, fatty alcohols, and sterols was carried out with the analytical methods described in Regulations of the European Union Commission [EEC, 1992; EEC, 1991 (Annexes IV and V)]. The squalene contents were determined by a gas-liquid chromatography according to the method of Lanzón et al. (1995).

Determination for Physical Properties. The value of absorptivity at 232 or 270 nm (K_{232} and K_{270}) as well as the visible spectrum (400–700 nm) was determined by spectrophotometry (spectrophotometer UV–vis, Beckman DU 640, Fullerton, CA). The values of K_{232} and K_{270} were determined according to recommendations in the Regulations of European Community Annex IX (EEC, 1991). To obtain the visible spectrum, the samples were dissolved in *n*-hexane (50% v/v).

For the determination of density, picnometer of 25 mL was used (IUPAC, 1992, No. 2101) that had been previously calibrated with distilled water. The viscosity measurement was carried out with a temperature-controlled falling ball viscosimeter (Höppler Type BH VEB MLW Leipzig, Germany) (UNE, 1973). Refractive indices were determined by a Abbe refractometer (Hilger & Watts Ltd., London, U.K.) equipped with a thermostat, using white light for the measurement (IUPAC, 1992, No. 2102). These three parameters were determined at 25 °C, with the exception of the LA measured at 60 °C.

Statistical Analysis of Experimental Data. Each reported value is the mean of two measurements from two replicates. Duncan's multiple range test (Duncan, 1955) was applied to determine significance between means, at a level of p < 0.05.

RESULTS AND DISCUSSION

Table 1 shows the values of the methyl esters of the untreated samples. Fatty alcohol and sterol compositions of untreated samples are shown in Table 2. There were no significant ($p \le 0.05$) differences in the methyl ester, fatty alcohol, and sterol compositions of the untreated and treated samples (data not shown). This was true for all five fats studied and for all treatments.

The amounts of squalene in the original samples were 4700, 3559, 224, and 175 ppm for VOO, OO, SO, and HOSO, respectively. However, no squalene was detected in the LA. Table 3 shows the losses of squalene after treatments. These losses ranged from 16 to 27% in the samples subjected to heating in a MO; this loss

 Table 2.
 Composition (Percent) and Content (Parts per

 Million) of Sterols and Fatty Alcohols in the Fats and
 Oils before Treatment

	sample				
	VOO	00	SO	HOSO	LA
sterols					
campesterol	3.2	2.9	10.4	11.0	nd ^a
stigmasterol	1.2	1.1	9.2	8.2	nd
β -sitosterol	85.3	84.7	65.8	68.3	nd
Δ^5 -avenasterol	10.1	11.2	1.8	1.2	nd
Δ^7 -stigmasterol	0.2	0.1	12.8	11.3	nd
cholesterol	nd	nd	nd	nd	100
total sterols (ppm)	1328	1245	3085	3115	982
alcohols					
22:0	17.5	19.1	14.1	13.4	nd
24:0	34.1	35.0	38.4	40.2	nd
26:0	32.2	31.8	29.3	31.1	nd
28:0	16.2	14.1	18.2	15.3	nd
total alcohols (ppm)	265	276	1115	1207	340 ^b

 $^a\,\mathrm{nd},$ not detected. b Other different fatty alcohols from those of the vegetable oils.

Table 3. Squalene Content (Parts per Million) in the Fats and Oils before and after Treatments^a

sample	total squalene	loss %	sample	total squalene	loss %
V00	4700	0	SO	224	0
VOO-MH	3478	26 ± 0.5	SO-MH	177	21 ± 0.4
VOO-CH	4230	9 ± 0.2	SO-CH	199	11 ± 0.2
VOO-M	4700	0	SO-M	244	0
00	3559	0	HOSO	175	0
OO-MH	2598	27 ± 0.5	HOSO-MH	147	16 ± 0.3
OO-CH	3203	10 ± 0.2	HOSO-CH	163	7 ± 0.1
OO-M	3559	0	HOSO-M	175	0

^{*a*} Mean value of four determinations.

was greater in the olive oil than in the sunflower oil. Conventional heating produces losses ranging from 7 to 11%; no differences in the squalene losses among the oils were observed. No losses of squalene were observed when fats and oils were subjected to microwave energy under the same conditions but below 40 $^{\circ}$ C.

Mean values of density, viscosity, refractive index, K_{270} , and K_{232} are shown in Table 4. For all samples analyzed (30 treated and 10 untreated), the coefficients of variation (CV %) were less than 1, 2.5, 0.1, 5, and 5%, respectively.

The patterns of change shown by the first two parameters that appear in Table 4, density and viscosity, were similar. There was a small increase in the values as a consequence of the conventional heating, a more pronounced increase when the effects of temperature and microwave energy were combined, and, finally, values similar to those of the original samples when the fats were subjected only to microwave energy without any increase of temperature.

The greater increase in the viscosity is directly related to the formation of dimers and polymers (increase in the length of the carbon chain). The increase in the density is due to oxygen incorporation through the oxidation compounds (oxidized triglycerides) according to Dobarganes (1980). It is to be supposed, therefore, that these compounds were generated in greater quantity in the samples heated in the microwave oven.

No variations were observed in the refractive index in any of the samples for any of the fats.

The unheated samples showed no increase of K_{270} , demonstrating that exposure of a fat to microwave



Figure 1. Changes in the absorption of the visible spectrum between 400 and 700 nm: (a) virgin olive oil (VOO); (b) VOO exposed to microwave energy (VOO-M); (c) VOO heated in a conventional oven (VOO-CH); (d) VOO heated in a microwave oven (VOO-MH).

 Table 4. Physical Parameters of the Original and

 Treated Fats and Oils^a

sample	density, ^b g/mL	viscosity, ^c mPa•s	refractive index ^d	K ₂₇₀ ^e	K_{232}^{e}
VOO	0.9094	68.19	1.4681	0.15	1.98
VOO-CH	0.9096	70.09	1.4682	0.60	2.49
VOO-MH	0.9106	77.90	1.4683	1.36	5.07
VOO-M	0.9091	68.49	1.4681	0.15	2.13
00	0.9087	71.64	1.4681	0.21	2.05
OO-CH	0.9088	71.91	1.4682	0.63	2.36
OO-MH	0.9109	78.17	1.4686	0.87	4.47
OO-M	0.9088	71.34	1.4678	0.22	2.08
SO	0.9145	51.00	1.4730	3.15	4.70
SO-CH	0.9161	52.83	1.4733	4.25	8.75
SO-MH	0.9172	54.35	1.4739	5.16	17.43
SO-M	0.9152	50.95	1.4730	3.20	4.79
HOSO	0.9087	70.24	1.4690	0.83	2.32
HOSO-CH	0.9105	70.39	1.4691	1.24	3.05
HOSO-MH	0.9107	77.72	1.4692	1.70	4.78
HOSO-M	0.9093	71.07	1.4690	0.83	2.35
LA	0.8875	20.50	1.4609	0.23	3.62
LA-CH	0.8881	20.58	1.4610	0.79	4.36
LA-MH	0.8888	20.85	1.4611	1.26	6.57
LA-M	0.8876	19.78	1.4609	0.23	3.65

^{*a*} Mean value from four samples. ^{*b*} CV \leq 1%. ^{*c*} CV \leq 1.5%. ^{*d*} CV \leq 1.5%. ^{*e*} CV \leq 5%.

energy at low temperature (40 °C) does not affect this parameter. However, with heating (170 °C) their increases were far greater compared to those produced in a conventional oven.

We justified the greater values of K_{270} , supposing that in the case of MO heating the formation of trienes and unsaturated ketones or aldehydes is greater due to the internal friction of the molecules.

To check this hypothesis, two of the samples heated in a MO, VOO-MH and OO-MH, were passed through an alumina column (EEC, 1991, Annex IX). The values of K_{232} and K_{270} obtained after purification were 1.36 and 0.13 for OO-MH and 1.55 and 0.12 for OO-MH, respectively, much lower than those in Table 4. It can be deduced that the values obtained in the impure samples were higher fundamentally as a consequence of the content in oxidation products, these being removed from the purified samples as they passed through the column. This would be in agreement with the results obtained for the other parameters that present variation in Table 4.

The variation of K_{232} , due to the formation of peroxides or conjugated dienes, presented a pattern similar to that of K_{270} .

Table 5 shows the *trans* isomer contents of the fatty acids of the initial and treated samples. The error of the determination of the area corresponding to peak 18: 1(9t) was worse than the value obtained in all of the determinations, due to overlapping of this peak with an artifact. It is not, therefore, possible to draw conclusions with respect to this isomer. The 18:2(9c,12t) isomer content did not show variations due to the treatment in any of the samples. Increases in the 18:2(9t,12c) isomer content greater than the error for the determination were only observed in SO and HOSO, heated in a MO and in a CO. This increase was greater in the SO because the isomer content of the initial sample was superior. The 18:2(9t,12t) isomer was only detected in LA, and the differences between the initial and the treated samples are within the confidence limits.

Figure 1 shows the changes in the absorption spectra between 400 and 700 nm (vis) of the four samples that are indicated in the caption, all of them corresponding to VOO. OO has some spectra similar to those of VOO. SO and LA are not considered, since both of the original samples presented flat spectra, as occurs with fats that have been subjected to a decoloration during refining and have had the colored pigments removed. From Figure 1b, it can be seen that MO treatment, without any elevation of the temperature, did not alter the

 Table 5. trans
 Fatty Acid Content (Percent) in the Total

 Fatty Acid Methyl Esters in the Fats and Oils before and
 after Treatment

	<i>trans</i> fatty acid				
sample	18:1(9t) ^a	$18:2(9C, 12t)^{b}$	18:2(9t,12c) ^b	18:2(9t,12t) ^b	
VOO	0.03	0.02	0.01	nd ^c	
VOO-CH	0.03	0.02	0.01	nd	
VOO-MH	0.06	0.02	0.01	nd	
VOO-M	0.04	0.02	0.01	nd	
00	0.20	0.02	0.03	nd	
OO-CH	0.17	0.02	0.02	nd	
OO-MH	0.21	0.03	0.06	nd	
OO-M	0.14	0.03	0.04	nd	
SO	0.02	0.05	0.10	nd	
SO-CH	0.04	0.06	0.24	nd	
SO-MH	0.08	0.06	0.24	nd	
SO-M	0.04	0.05	0.15	nd	
HOSO	0.05	0.03	0.03	nd	
HOSO-CH	0.08	0.04	0.07	nd	
HOSO-MH	0.09	0.04	0.07	nd	
HOSO-M	0.07	0.03	0.04	nd	
LA	0.50	0.06	0.07	0.04	
LA-CH	0.60	0.08	0.07	0.02	
LA-MH	0.60	0.09	0.09	0.05	
LA-M	0.60	0.07	0.08	0.06	

^{*a*} Error = ± 0.1 . ^{*b*} Error = ± 0.02 . ^{*c*} nd, not detected.

spectrum, which was identical to that of the oil before treatment. This indicates, then, there is no effect on the carotenoids (415, 455, and 485 nm), flavonoids (535 and 560 nm), and chlorophylls (610 and 670 nm). In contrast, heating in a CO (Figure 1c) produced a decrease in the chlorophylls and carotenoids, the decrease in the latter being more pronounced. Finally, when the increase of the temperature was a consequence of the microwave energy, the loss of the pigments was more generalized, suggesting a synergistic effect between temperature and microwave energy.

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LITERATURE CITED

- Decareau, R. V. Microwaves in the Food Processing Industry, Academic Press: London, 1985; pp 28–32.
- Dobarganes, M. C. Aspectos generales de las grasas calentadas. Grasas Aceites 1980, 31, 417–422.
- Duncan, D. B. Multiple range and multiple F-tests. *Biometrics* **1955**, *11*, 1–42.
- EEC. *Diario Oficial de las comunidades Europeas*; Brussels, Belgium, 1991; Annexes I, IV, V, and IX.
- EEC. *Diario Oficial de las comunidades Europeas*; Brussels, Belgium, 1992; pp 17–20.
- Hearn, T. L.; Sgoutas, S. A.; Sgoutas, D. S.; Hearn, J. A. Stability of polyunsaturated fatty acids after microwave cooking of fish. J. Food Sci. 1987, 52, 1430–1431.

- IUPAC. Standard Methods for the Analysis of Oils, Fats and Derivatives, 7th revised ed., 1st Supplement; Blackwell: Oxford, U.K., 1992; No. 2101, p 31, No. 2102, p 34.
- Jowitt, R. Physical Properties of Foods; Applied Science: London, 1983; pp 249–269.
- Kamel, B. S.; Stauffer, C. E. Advances in Baking Technology, Chapman & Hall, London, 1993; pp 292–313.
- Lanzón, A.; de la Osa, C.; Albi, T.; Guinda, A. Método rápido para la determinación de escualeno en aceites vegetales. *Grasas Aceites* **1995**, *46*, 276–278.
- Lie-Ken-Jie, M.; Yan-Kit, C. The use of a microwave oven in the chemical transformation of long chain fatty acid esters. *Lipids* **1988**, *23*, 367–369.
- Maga, J. A.; Twomey, J. A.; Cohen, M. Effect of baking methods on the fatty acid composition of potatoes. J. Food Sci. 1977, 42, 1669–1670.
- Mai, J.; Tsai, C.; Armbruster, G.; Chu, P.; Kinsella, J. Effects of microwave cooking on food fatty acids: no evidence of chemical alteration or isomerization. *J. Food Sci.* **1980**, 45, 1753–1755.
- Mohsenin, N. N. Electromagnetic Radiation Properties of Foods and Agricultural Products; Gordon & Breach: New York, 1984; pp 41–52.
- Sowa, T. The coefficient of dielectric loss tan delta in the quality evaluation of animal fats undergoing aging processes. *Acta Aliment. Pol.* **1986**, *12*, 221–235.
- Tsuyuki, H. Utilization of high frequency and microwave heating in food industry. J. Jpn. Soc. Food Sci. Technol. **1982**, 29, 123–130.
- UNE. Una Norma Española. Materias Grasas, 1973; No. 55.105.73.
- Yoshida, H.; Hirooka, N.; Kajimoto, G. Microwave energy effects on quality of some seed oils. J. Food Sci. 1990, 55, 1412–1416.
- Yoshida, H.; Tatsumi, M.; Kajimoto, G. Relationship between oxidative stability of vitamin E and production of fatty acids in oils during microwave heating. J. Am. Oil Chem. Soc. 1991a, 68, 566–570.
- Yoshida, H.; Hirooka, N.; Kajimoto, G. Microwave heating effects on relative stabilities of tocopherols in oils. J. Food Sci. 1991b, 56, 1042–1046.
- Yoshida, H.; Tatsumi, M.; Kajimoto, G. Influence of fatty acids on the tocopherol stability in vegetable oils during microwave heating. J. Am. Oil Chem. Soc. 1992a, 69, 119–125.
- Yoshida, H.; Kondo, I.; Kajimoto, G. Effects of microwave energy on the relative stability of vitamin E in animal fats. J. Sci. Food Agric. **1992b**, 58, 531–534.
- Yoshida, H.; Kajimoto, G.; Emura, S. Antioxidant effects of d-Tocopherols at different concentrations in oils during microwave heating. J. Am. Oil Chem. Soc. 1993, 70, 989– 995.

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