Changes in Olive Oil Composition Due to Microwave Heating

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ABSTRACT: The effects of microwave heating on some components of extra-virgin olive oil were studied. Traditional parameters, including free acidity, peroxide value and ultraviolet absorbance values at 232 and 268 nm, were determined in six extra-virgin olive oil samples before and after the microwave treatment. Significant differences ($P < 0.01$) were detected for free acidity, peroxide, and ultraviolet absorbance at 268 nm; also, the absorbances at 232 nm showed significant differences ($P < 0.05$) between treated and untreated samples. The glycerolic fractions, triacylglycerols (TAG), diacylglycerols (DAG), and monoacylglycerols (MAG), were isolated by thin-layer chromatography. The respective percentage fatty acid (FA) composition and percentage amount were obtained by high-resolution gas chromatography with an internal standard. For the most abundant TAG fraction, the stereospecific analysis was carried out to obtain the FA percentage compositions of the three *sn*-positions. Small but significant modifications were observed regarding the decrease in the TAG percentage and increases in the DAG and MAG percentage amounts. No significant changes were observed for the FA compositions of TAG, DAG, and MAG fractions before and after the treatment. Nevertheless, the results of TAG stereospecific analysis showed losses of unsaturated FA in all *sn*-positions. Higher percentage changes in the *sn*-1- than in *sn*-2-position of TAG were observed. Regarding the volatile fraction, different profiles were obtained after the treatment.

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KEY WORDS: Acylglycerols, microwave heating, olive oil, stereospecific analysis, triacylglycerols, volatile fraction.

The use of microwave heating, both for home and for institutional meal preparation, has increased over the last few decades because of its advantages, which include speed and convenience (1).

For industrial applications, microwave heating can be used for many processes: tempering, dehydration, blanching, cooking, pasteurization, and sterilization (2). Numerous investigations have been done to evaluate the effects of microwaves on food constituents (3); the effects of microwave and conventional heating on nutrient retention and the formation of flavors and colors in foods have been compared (4). With reference to lipid components, microwave heating was investigated to verify the effect on different animal and vegetable fats (5–7). The rate of quality deterioration, such as oxidative degradation, depends on the amount of polyunsaturated fatty acids in the oil (6).

To evaluate the consequences of microwave heating on lipids, some measurements of lipid oxidation, such as peroxide value, thiobarbituric acid test, carbonyl value, anisidine value and conjugated diene and triene levels, are frequently reported (5,6). Many authors have reported the results of the fatty acid (FA) compositions of total lipids in the oil, sometimes together with those of the different acylglycerol fractions (8); sometimes the effects of microwave treatments on the molecular species percentage composition and FA percentage distribution in triacylglycerols (TAG) were investigated (9,10). Raghuveer and Hammond suggested that acylglycerol structure might affect the relative rates of oxidation of acyl groups in the TAG molecules (11). The influence of the α- and β-positions occupied by unsaturated FA in the glycerol backbone on the oxidation rate was investigated in thermally oxidized oils (12,13).

The objective of this study was to determine the effects of microwave heating on olive oil samples; in particular, the FA composition in the partial and total acylglycerols, as well as in the *sn*-positions of TAG studied. An additional objective of the research was the evaluation of qualitative and quantitative (percentage and absolute) compositional changes in the volatile fraction of the olive oil samples as a consequence of the microwave treatment.

EXPERIMENTAL PROCEDURES

Materials. Six samples of extra-virgin olive oil, produced in Umbria (Italy) in 1996, were used. Each sample (10.0 g) was placed in a 25-mL open glass vessel and then in a Whirlpool Model Vip 27 (Philips, The Netherlands) microwave oven for 10 min, at a frequency of 2,450 MHz and a power of 0.75 kW. The temperature of the treated samples soon after they were taken out of the oven was 210 ± 5°C. After the microwave heating, the samples were stored under N_2 and cooled at room temperature in the dark. Within half an hour, the free acidity, peroxide value, and ultraviolet (UV) absorbance were deter-

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mined. The samples were then stored at 4°C for the successive determination of the other parameters.

Volatile compounds were analyzed with both a thermal desorption cold-trapping injector (TD-CTI) system and with a solid-phase micro extraction (SPME) system. Five milliliters of each oil sample was placed in 10-mL closed vials and then microwave-treated under the above-described conditions. The temperature of the samples, measured as soon as they were taken out of the oven, was 150 ± 5°C.

Analysis of oil samples. Free acidity, peroxide value, and UV absorbance were determined according to EC 2568/91 (14).

The separate glycerolic fractions from each oil sample, TAG, diacylglycerols (DAG), and monoacylglycerols (MAG), were obtained by thin-layer chromatography (TLC) on silica gel plates under the previously reported conditions (15); their respective FA percentage compositions and percentage contents in each sample were determined by high-resolution gas chromatography (HRGC) of the FA methyl esters (FAME) (15) after methyl nonadecanoate was added as an internal standard.

Stereospecific analysis of TAG. The TAG fraction was subjected to the stereospecific analysis by the procedure reported in previous papers (15,16). This method is based on pancreatic lipase hydrolysis to obtain the FA composition of the *sn*-2-position and on stereospecific phosphorylation by *sn*-1,2-DAG kinase of *sn*-1,2-DAG, obtained from TAG by partial hydrolysis with ethyl magnesium bromide. By HRGC analysis of FAME of TAG, *sn*-2-MAG, and *sn*-1,2-phosphatidic acids, it is possible to obtain the FA percentage positional compositions of TAG.

Analysis of volatile compounds. This operation was completed by dynamic headspace analysis; a 5.0-mL olive oil sample was heated at 40°C and swept with N_2 (100 mL/min) for 20 min. An internal standard (acetic acid, *n*-pentyl ester) was added for quantitative analysis. Tenax TA (Chrompack, Middelburg, The Netherlands) was used as a trap. Volatiles were thermally desorbed at 230°C onto a pre-cooled (–120°C by liquid N_2) fused-silica trap for 5 min, and then quickly transferred to the chromatographic column by flash heating of the cold trap at 230°C for 3 min. HRCG was performed with a Hewlett-Packard (Palo Alto, CA) 5890 series II gas chromatograph, equipped with a HP-5971 mass selective detector (MSD) and fitted with a HP-Innowax capillary column (60 m, 0.25 mm i.d., 0.5 µm film thickness). Helium was used as carrier gas at 1 mL/min. The oven temperature was held at 45°C for 6 min, then programmed at 3°C/min to 60°C, then at 6°C/min to a final temperature of 240°C, which was held for 10 min. The transfer line and MSD temperatures were 280 and 180°C, respectively. The compounds were identified by comparison of retention times and mass spectral data with those of authentic reference compounds. When standards were not available, the volatile compounds were tentatively identified by mass spectrum matching with the Wiley 138 mass library collection. The quantitative analyses were carried out without correction of the peak areas with response factors because the reference substances were not available for many compounds.

The volatile fraction of the samples was also analyzed by using solid-phase micro extraction and high-resolution gas chromatography with mass selective detector (SPME-HRGC-MSD). In particular, 5.0 mL of each sample in a 10-mL vial with hole cap and polytetrafluoroethylene/silicone septa was heated at 40°C under stirring; the extraction of volatiles was carried out at the same temperature by SPME 100 µm polydimethylsiloxane fiber (Supelco, Bellefonte, PA), maintained in the headspace for 45 min. After this time, the thermal desorption of the volatile compounds was carried out by placing the fiber in the split-splitless injector at 250°C for 1 min. The injector was maintained in the splitless mode for this time. The separation, detection, and identification of volatiles were performed by the same HRGC-MSD apparatus as previously described. The only change in experimental conditions was the temperature program. The oven temperature was held at 45°C for 6 min, then programmed at 3°C/min to 60°C, then at 10°C/min to a final temperature of 240°C, which was held for 10 min.

Statistical analysis. Analyses were repeated at least twice, and the mean values are reported.

Student's *t*-test (17) of type 1, for paired samples, and two-tailed, was used to determine the differences between treated and untreated samples.

RESULTS AND DISCUSSION

In Table 1, the results of the investigated classical parameters are reported; significant differences were observed for all parameters. Concerning the slightly higher values of free acidity for treated samples in respect to the nontreated samples, it should be understood that the alkali titration determines all acid components. Moreover, during the microwave treatment, at the same time that acid compounds are being produced, either by breakdown due to oxidative processes or by hydrolysis, some of them could have sufficient vapor pressure to evaporate. As expected, higher peroxide values were indicative of the extent of primary oxidation and were detected in the treated samples. Increases in UV absorbance were observed, indicating that oxidation had occurred (6,18).

Evaluation of the changes in the glycerolic fractions of microwave-treated olive oils was one of the most important observations made. Table 2 reports the results of the percentages

TABLE 1
Classical Parameters for Untreated and Microwave-Treated Samples[a]

	NTS[b]	TS[b]	*P*[c]
Acid value, as g oleic acid · 100 g^{-1}	0.30 ± 0.04	0.35 ± 0.03	0.01
Peroxide value, meq $O_2 \cdot kg^{-1}$	9.0 ± 1.8	18.1 ± 3.9	<0.01
UV absorbance, 232 nm	1.80 ± 0.14	2.19 ± 0.41	0.05
UV absorbance, 268 nm	0.11 ± 0.02	0.43 ± 0.20	0.01

[a]Mean values ± standard deviations, $n = 6$.
[b]NTS, untreated samples; TS, treated samples.
[c]*t*-Test probability for the two sets of data. UV, ultraviolet.

TABLE 2
Percentage Amounts of Tri-, Di-, and Monoacylglycerol (TAG, DAG, and MAG) Fractions[a]

	NTS	TS	P^b
TAG	96.8 ± 0.9	93.2 ± 0.6	<0.01
DAG	1.2 ± 0.2	1.4 ± 0.2	<0.01
MAG	0.03 ± 0.01	0.08 ± 0.01	<0.01

[a]Mean values ± standard deviations, $n = 6$.
[b]*t*-Test probability for the two sets of data. See Table 1 for abbreviations.

of TAG, DAG, and MAG fractions in the samples. There were significant decreases ($P < 0.01$) in the TAG fraction and increases ($P < 0.01$) in the DAG and MAG fractions after microwave treatment. Both oxidative and hydrolytic processes could be responsible for these modifications in treated olive oil samples. The results of the FA percentage compositions of MAG, DAG, and TAG are reported together with the absolute contents of each FA in each acylglycerol fraction (Tables 3–5). With the exception of the linolenic acid percentage in the MAG fraction, no significant differences were detected among the FA percentage of the glycerolic fraction in the treated and untreated samples. Nevertheless, the decrease in the percentages of linoleic and linolenic acids was observed as a general trend in the three glycerolic fractions. Obviously, the changes of the FA absolute contents in each fraction after microwave heating depend on the increase or decrease of the considered fraction in the sample. In Tables 6, 7, and 8, the results of the TAG stereospecific analysis (intrapositional FA percentage compositions, respectively for the *sn*-1-, *sn*-2-, and *sn*-3-positions) are reported. The results of the absolute percentages of each FA in each *sn*-position of the TAG fraction are also reported (Tables 6–8), as described in the footnotes of the respective tables. The data reported in Table 6 show that significant decreases (percentage and absolute) of linoleic and linolenic acids occurred in the *sn*-1-position of the TAG fraction of microwave-treated samples. At the same time, the observed increase of the percentage content of oleic acid after the microwave treatment did not correspond to the increment of the absolute contents; the increase in oleic acid is thus an "apparent" increase in the percentage content.

Similar results were observed for the percentage FA composition of the *sn*-2-position of TAG. Significant decreases in the percentages of linoleic and linolenic acids were detected, balanced by an increase in the percentage of oleic acid. The results of the percentage compositions of linoleic and linolenic acids show that lower changes occurred in the *sn*-2- in relation to the *sn*-1-position.

The data reported in Table 8 for the percentage abundances of the two polyunsaturated FA in the *sn*-3-position are probably anomalous. The data should be evaluated with the observation that they represent relatively low absolute contents.

Comparison between the percentage decreases (DEC%) of the linoleic and linolenic acid absolute contents in TAG *sn*-1- and *sn*-2-positions (the only experimental data) shows that the microwave treatment causes greater FA degradation in the *sn*-1-position (18.2% for linoleic and 50.0% for linolenic acids) than in the *sn*-2-position of TAG (8.3% for linoleic and 33.3% for linolenic). The DEC% was calculated according to the following equation: DEC% = (mg NTS − mg TS/mg NTS) · 100 where mg NTS and mg TS are, respectively, the absolute contents of the FA in the untreated and treated sam-

TABLE 3
Percentage and Absolute Fatty Acid (FA) Compositions of MAG from Nontreated and Treated Oil Samples[a]

	FA percentage abundances		μg in MAG from oil (100 mg)[b]	
	NTS	TS	NTS	TS
C16:0	12.7 ± 2.1	13.0 ± 0.7	3.0	8.2
C16:1[c]	2.7 ± 1.2	2.2 ± 2.1	0.6	1.4
C18:0	3.5 ± 0.9	2.9 ± 0.6	0.8	1.8
C18:1[c]	71.7 ± 3.9	74.5 ± 3.3	17.0	47.1
C18:2n-6	7.6 ± 1.8	6.3 ± 0.4	1.8	4.0
C18:3n-3	1.8[d] ± 0.8	1.0[d] ± 0.3	0.4	0.6

[a]Mean values ± standard deviations, $n = 6$.
[b]Data obtained from the mean values of the percentage content of MAG fraction, Table 2, and of the MAG fraction FA percentage abundances, this table.
[c]Sum of positional isomers n-9 and n-7.
[d]*t*-Test probability for the two sets of data: $P < 0.05$. See Tables 1 and 2 for abbreviations.

TABLE 4
Percentage and Absolute FA Compositions of DAG from Nontreated and Treated Oil Samples[a]

	FA percentage abundances		μg in DAG from oil (100 mg)[b]	
	NTS	TS	NTS	TS
C16:0	7.5 ± 0.3	8.1 ± 0.8	81.7	102.9
C16:1[c]	0.6 ± 0.1	0.7 ± 0.1	6.5	8.9
C18:0	1.4 ± 0.2	1.5 ± 0.2	15.3	19.0
C18:1[c]	80.9 ± 1.0	80.4 ± 1.3	881.8	1021.1
C18:2n-6	8.9 ± 0.6	8.6 ± 0.6	97.0	109.2
C18:3n-3	0.7 ± 0.0	0.7 ± 0.1	7.6	8.9

[a]Mean values ± standard deviations, $n = 6$.
[b]data obtained from the mean values of the percentage content of DAG fraction, Table 2, and of the DAG fraction FA percentage abundances, this table.
[c]Sum of positional isomers n-9 and n-7. See Tables 1–3 for abbreviations.

TABLE 5
Percentage and Absolute FA Compositions of TAG from Nontreated and Treated Oil Samples[a]

	FA percentage abundances		mg in TAG from oil (100 mg)[b]	
	NTS	TS	NTS	TS
C16:0	12.9 ± 0.6	12.5 ± 1.0	10.6	9.9
C16:1[c]	0.8 ± 0.1	0.8 ± 0.1	0.7	0.6
C18:0	2.1 ± 0.1	2.0 ± 0.1	1.7	1.6
C18:1[c]	77.7 ± 0.6	78.3 ± 0.9	63.9	62.0
C18:2n-6	6.0 ± 0.4	5.8 ± 0.2	4.9	4.6
C18:3n-3	0.6 ± 0.1	0.5 ± 0.1	0.5	0.4

[a]Mean values ± standard deviations, $n = 6$.
[b]Data obtained from the mean values of the percentage content of TAG fraction, Table 2, and of the TAG fraction FA percentage abundances, this table.
[c]Sum of positional isomers n-9 and n-7. See Tables 1–3 for abbreviations.

TABLE 6
Percentage and Absolute FA Compositions of *sn*-1-Position of TAG from Nontreated and Treated Oil Samples[a]

	FA percentage abundances		mg in *sn*-1-position of TAG from oil (100 mg)[b]	
	NTS	TS	NTS	TS
C16:0	16.7 ± 1.4	16.5 ± 1.0	4.5	4.4
C16:1[c]	1.0 ± 0.3	1.1 ± 0.4	0.3	0.3
C18:0	2.5 ± 0.3	2.5 ± 0.2	0.7	0.7
C18:1[c]	71.0[d] ± 1.3	72.4[d] ± 1.5	19.5	19.1
C18:2 n-6	8.0[d] ± 0.6	7.0[d] ± 0.7	2.2	1.8
C18:3 n-3	0.6[d] ± 0.1	0.4[d] ± 0.1	0.2	0.1

[a]Mean values ± standard deviations, $n = 6$.
[b]Data obtained from the mean values of the percentage content of TAG fraction, Table 2, of the TAG fraction FA percentage abundances, Table 5, and of the intrapositional FA percentage abundances, calculated from the results of TAG stereospecific analysis, Tables 6–8.
[c]Sum of positional isomers n-9 and n-7.
[d]*t*-Test probability for the two sets of data: $P < 0.05$. See Tables 1–3 for abbreviations.

TABLE 8
Percentage and Absolute FA Compositions of *sn*-3-Position of TAG from Nontreated and Treated Oil Samples[a]

	FA percentage abundances		mg in *sn*-3-position of TAG from oil (100 mg)[b]	
	NTS	TS	NTS	TS
C16:0	21.9 ± 1.1	20.4 ± 3.2	5.9	5.4
C16:1[c]	0.9 ± 0.2	0.8 ± 0.4	0.2	0.2
C18:0	3.6 ± 0.3	3.5 ± 0.2	1.0	0.9
C18:1[c]	72.2 ± 1.1	72.8 ± 3.2	19.8	19.2
C18:2n-6	1.1 ± 0.3	2.1 ± 0.7	0.3	0.5
C18:3n-3	0.2 ± 0.1	0.4 ± 0.1	0.1	0.1

[a]Mean values ± standard deviations, $n = 6$.
[b]Data obtained from the mean values of the percentage content of TAG fraction, Table 2, of the TAG fraction FA percentage abundances, Table 5, and of the intrapositional FA percentage abundances, calculated from the results of TAG stereospecific analysis, Tables 6–8.
[c]Sum of positional isomers n-9 and n-7. See Tables 1–3 for abbreviations.

ples. The obtained results are in agreement with those obtained by Raghuveer and Hammond (11), by Wada and Koizumi (12), and by Yoshida and Alexander (13) for FA thermal oxidative degradation. These authors reported that the TAG, with unsaturated FA linked at the *sn*-2-position of glycerol, are more stable toward thermal oxidation than those with the same acid at the *sn*-1- or *sn*-3-positions. The results have important nutritional significance because of the well-known fact that the FA incorporated in the TAG *sn*-2-position are better absorbed than the same FA esterified in the *sn*-1-position (19,20).

Further investigations of this research were conducted to evaluate the changes of the composition of the volatile fraction from the oil samples. In Figure 1, the chromatographic profiles of the oil sample, before and after microwave treatment, are shown. In Table 9, the percentage composition of volatile fractions and the absolute amounts (g/5 mL of oil) of their components are reported. Variable amounts of many volatile compounds in different oil samples were observed. This result is explainable by many factors, such as olive tree growing conditions and oil production technology, which influence the composition of the relative fractions. Nevertheless, increases in some compounds and the formation of new compounds were detected in the microwave-treated samples, which included hydrocarbons, aldehydes, and ketones, both saturated and unsaturated.

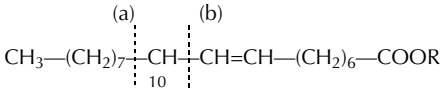
TABLE 7
Percentage and Absolute FA Compositions of *sn*-2-Position of TAG from Nontreated and Treated Oil Samples[a]

	FA percentage abundances		mg in *sn*-2-position of TAG from oil (100 mg)[b]	
	NTS	TS	NTS	TS
C16:0	0.5 ± 0.1	0.6 ± 0.1	0.1	0.2
C16:1[c]	0.6 ± 0.1	0.6 ± 0.1	0.2	0.1
C18:0	0.0 ± 0.0	0.1 ± 0.0	0.0	0.0
C18:1[c]	89.4[d] ± 0.4	89.7[d] ± 0.5	24.5	23.7
C18:2n-6	8.7[d] ± 0.5	8.4[d] ± 0.4	2.4	2.2
C18:3n-3	0.9[d] ± 0.0	0.8[d] ± 0.1	0.3	0.2

[a]Mean values ± standard deviations, $n = 6$.
[b]Data obtained from the mean values of the percentage content of TAG fraction, Table 2, of the TAG fraction FA percentage abundances, Table 5, and of the intrapositional FA percentage abundances, calculated from the results of TAG stereospecific analysis, Tables 6–8.
[c]Sum of positional isomers n-9 and n-7.
[d]*t*-Test probability for the two sets of data: $P < 0.05$. See Tables 1–3 for abbreviations.

Numerous studies, generally on model systems, have been carried out to elucidate the mechanisms of formation of the products from FA thermal/oxidative decomposition (21). Comparison of the compounds identified in this research with those reported in the literature did not show the presence of some compounds (in particular some unsaturated long-chain aldehydes, such as *t*-2-decenal, *t*-2-undecenal, and *t*,*t*-(*t*,*c*-)-2,4-decadienal), probably because the inability of Tenax to absorb and desorb these compounds under the conditions found to be the best for the other volatiles. For this reason, the SPME technique with a nonpolar fiber was used. In Figure 2, the chromatographic profiles for the volatile compounds of the two oil samples are shown. The SPME analysis was used to verify the presence of nonanal and unsaturated long-chain aldehydes.

Concerning the identified compounds, the mechanisms of formation have been elucidated for many of them. As an example, formation of the compounds octane and nonanal, (a) and (b) respectively, has been verified to derive from the oleate 10-hydroperoxide:



The compound 2,4-decadienal is derived from the linoleate 9-hydroperoxide:

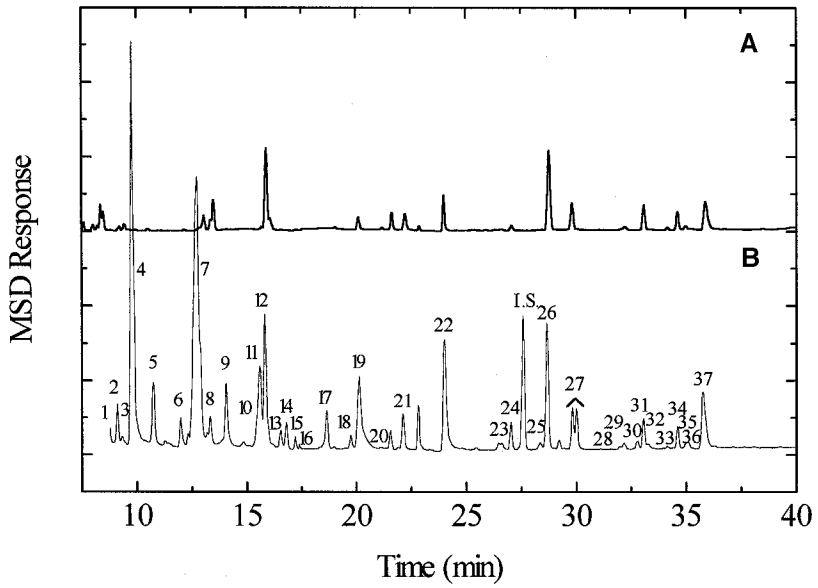
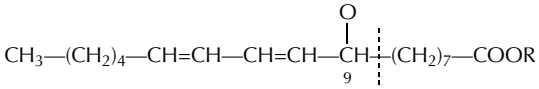
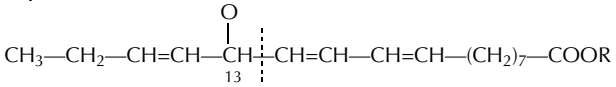


FIG. 1. Chromatographic profiles of volatile compounds trapped on Tenax, (A) before and (B) after microwave treatment. The compounds are listed in Table 9.



and the compound 2-pentenal from the linolenate 13-hydroperoxide:



Among the substances formed after the microwave treatment, an epoxide compound was tentatively identified: 3-ethyl-2,2-dimethyl oxirane. Formation of epoxides in lipids has been reported (21).

In conclusion, microwave heating causes oxidative changes in the glycerolic fractions of olive oil. The results of the stereospecific analysis of TAG fraction show that the unsaturated FA linked at the *sn*-2-position are more stable toward oxidation than those linked to the TAG primary positions. This fact has considerable nutritional significance.

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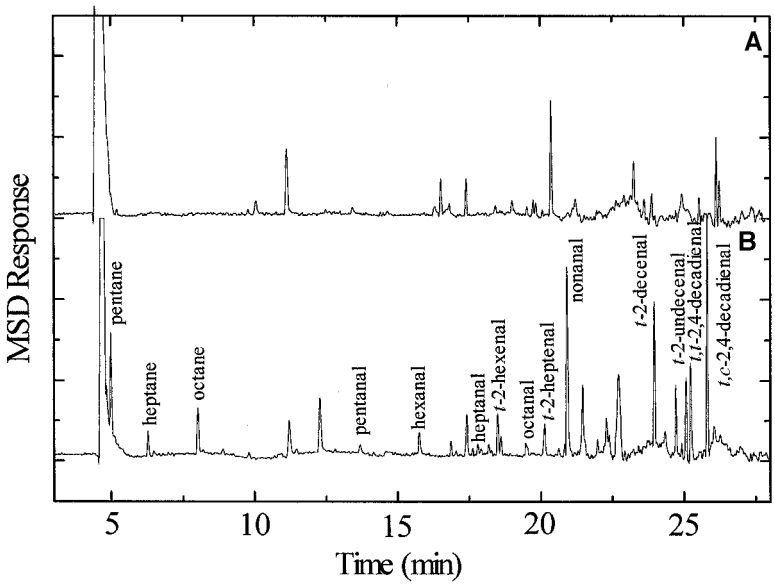

FIG. 2. Chromatographic profiles of compounds adsorbed by SPME, (A) before and (B) after microwave treatment.

TABLE 9
Percentage and Absolute Compositions of Volatile Fractions[a]

	Compounds	Percentage compositions		Amounts (μg/5 mL)[b]	
		NTS	TS	NTS	TS
1	2-Me 1,3-butadiene[c]	0.3 ± 0.4	0.6 ± 0.1	1.0 ± 1.1	3.9 ± 1.1
2	*t*-1,3-Pentadiene[c]	0.5 ± 0.5	1.7 ± 0.1	1.7 ± 1.9	10.8 ± 4.5
3	*c*-1,3-Pentadiene[c]	0.7 ± 0.6	0.7 ± 0.1	2.6 ± 2.2	4.6 ± 2.2
4	Heptane	0.1 ± 0.2	9.9 ± 9.1	0.4 ± 0.7	74.9 ± 93.6
5	1-Heptene[c]	0.0 ± 0.0	1.6 ± 1.8	0.0 ± 0.0	12.0 ± 16.4
6	Propanal[c]	0.5 ± 0.3	2.5 ± 0.6	1.7 ± 1.2	15.8 ± 7.3
7	Octane	0.0 ± 0.0	4.5 ± 5.2	0.0 ± 0.0	34.8 ± 47.7
8	Acetic acid, Me ester	6.6 ± 1.2	2.6 ± 0.5	22.4 ± 3.8	15.4 ± 1.7
9	2-Propenal[c]	0.0 ± 0.0	3.8 ± 0.7	0.0 ± 0.0	24.1 ± 7.8
10	Butanal	0.0 ± 0.0	0.9 ± 0.4	0.0 ± 0.0	6.4 ± 4.9
11	2-Butenal[c]	0.0 ± 0.0	6.3 ± 2.5	0.0 ± 0.0	36.7 ± 5.0
12	Acetic acid, Et ester	17.2 ± 8.5	8.0 ± 4.3	62.0 ± 34.9	50.3 ± 28.2
13	2-Butanone	1.2 ± 1.4	1.2 ± 0.7	4.2 ± 5.0	7.2 ± 3.3
14	3-Et-2,2-diMe oxirane[c]	0.0 ± 0.0	1.2 ± 0.2	0.0 ± 0.0	7.8 ± 1.4
15	2-Me butanal	3.7 ± 3.2	1.6 ± 1.1	11.4 ± 8.5	8.5 ± 4.9
16	3-Me butanal	4.1 ± 3.9	1.6 ± 1.3	12.4 ± 10.8	8.2 ± 6.3
17	3-Buten-2-one[c]	0.0 ± 0.0	2.9 ± 1.3	0.0 ± 0.0	16.7 ± 3.5
18	2,3-Butanedione[c]	0.0 ± 0.0	0.8 ± 0.3	0.0 ± 0.0	5.0 ± 1.2
19	3-Pentanone + pentanal	7.6 ± 3.8	6.4 ± 1.1	24.7 ± 9.8	38.7 ± 5.9
20	1-Penten-3-one	0.7 ± 0.6	0.5 ± 0.3	2.2 ± 1.9	2.7 ± 1.5
21	2-Me 3-buten-2-ol	1.3 ± 2.2	2.8 ± 0.7	5.2 ± 9.0	16.7 ± 0.4
22	Hexanal + 2-Me 1-propanol	5.9 ± 2.2	7.1 ± 0.5	20.9 ± 11.6	44.0 ± 13.8
23	*t*-2-Pentenal	0.3 ± 0.3	0.7 ± 0.2	0.8 ± 0.8	4.1 ± 0.1
24	1-Penten-3-ol	4.2 ± 2.7	4.5 ± 2.8	13.4 ± 6.9	24.2 ± 12.3
25	Heptanal	0.0 ± 0.0	0.1 ± 0.0	0.0 ± 0.0	0.7 ± 0.3
26	3-Me 1-butanol	10.4 ± 10.2	3.8 ± 2.6	40.0 ± 45.2	27.8 ± 27.1
27	*t*-2-Hexenal + 1-pentanol	9.3 ± 4.1	7.4 ± 4.1	30.9 ± 9.0	41.2 ± 13.3
28	*t*-2-Penten-1-ol[c]	0.2 ± 0.2	0.1 ± 0.1	0.5 ± 0.5	0.9 ± 0.3
29	*c*-2-Penten-1-ol	1.5 ± 0.4	0.7 ± 0.2	5.1 ± 0.3	4.5 ± 0.7
30	1-Hydroxy 2-propanone[c]	0.0 ± 0.0	0.2 ± 0.2	0.0 ± 0.0	1.7 ± 2.0
31	1-Hexanol	2.7 ± 3.6	0.9 ± 0.8	10.7 ± 15.3	7.0 ± 7.8
32	*t*-2-Heptenal[c]	0.0 ± 0.0	0.3 ± 0.0	0.0 ± 0.0	1.8 ± 0.2
33	*c*-3-Hexen-1-ol	0.9 ± 0.2	0.4 ± 0.3	3.0 ± 0.1	2.4 ± 1.5
34	*t*-2-Hexen-1-ol[c]	2.3 ± 0.8	1.0 ± 0.2	8.2 ± 4.0	7.0 ± 3.5
35	2-Butoxy ethanol[c]	0.7 ± 0.2	0.3 ± 0.0	2.2 ± 0.4	1.8 ± 0.2
36	Nonanal	0.2 ± 0.1	0.3 ± 0.1	0.8 ± 0.4	1.7 ± 0.2
37	Acetic acid	17.0 ± 2.5	9.8 ± 4.8	58.0 ± 9.0	56.8 ± 23.5

[a]Mean values ± standard deviations, $n = 3$.
[b]These values were obtained considering the response factor = 1 for all components (as reported in the Experimental Procedures section).
[c]Tentatively identified. See Table 1 for abbreviations.

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