

Volatile Oxidation Compounds and Stability of Safflower, Sesame and Canola Cold-Pressed Oils as Affected by Thermal and Microwave Treatments

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Abstract: The goal of this study was to investigate the effect of heating and microwave treatment on the levels of volatile oxidation products and the stability of safflower (*Carthamus tinctorius* L.), sesame (*Sesamum indicum*) and canola (*Brassica napus* L.) cold-pressed oils. Cold-pressed oils were subjected to conventional heating (oven test) using air-forced oven at 60°C and microwave heating for 2 and 4 min. The changes in conjugated diene (CD) and conjugated triene (CT) values were monitored during treatments. As expected, heating generates an increase in CD and CT values. The volatile compounds in treated oils were determined using solid phase micro-extraction-gas chromatography/mass spectrometry (SPME-GC/MS). The obtained GC/MS data were used to characterize volatile compounds of cold-pressed oils during heating and microwave treatments. Under oven conditions, 2-heptenal and 2,4-heptadienal isomers were identified as major components in canola oil, while hexanal and 2-heptenal were found in high levels in safflower and sesame oils. Among volatiles, *p*-cymene was the dominant compound found in microwave-treated canola oil. In addition, hexanal and 2-hexenal were found at high amounts upon microwave treatment especially after 4 min of application.

Key words: *Carthamus tinctorius* L., *Sesamum indicum*, *Brassica napus* L., Shaal oven test, SPME-GC/MS

1 Introduction

Lipid oxidation is a major deterioration problem in oils and fats. Oxidation could alter the flavor of these products by inducing toxic substances and undesirable volatile compounds during oxidation¹⁻³. Hydroperoxides are primary oxidation products which are colorless and odorless. These products are labile and quickly turn into secondary oxidation products such as alkanes, alcohols, aldehydes, and acids⁴. The volatile compounds of secondary oxidation products have an impact on the lipids flavor at extremely low concentrations. Different volatile oxidation products could be derived from various conditions like heat, light and metal⁵.

Oils and fats was subjected to heating (Schaal oven test) to accelerate thermal oxidation. In this test, lipidic samples are usually heated at 60°C in an oven. Hexanal, 2-decenal, 2,4-decadienal, 2-decenal, and pentane were identified as volatile oxidation compounds by SPME methods in thermally oxidized (at 60°C) soybean and corn oils⁶. In addition, hexanal, pentane, propenal and 2,4-decadienal were identified

in high levels in canola oil during heating⁷.

Microwaves are very popular for high speed and convenience, as compared to conventional heating⁸. The utilization of microwave in fast food preparation is increased because of its ease of operation, and low cost⁹. Radiation of Microwave penetrates to the food, causing reheating or cooking. However, there are speculations on the ease of free radical formation when fatty foods are exposed to microwave energy resulting in production of objectionable compounds in microwave-cooked foods¹⁰. Microwave-treated oils formed reactive free radicals that reacted with the atmospheric oxygen to form hydroperoxides and oxidation products¹¹. Unsaturated lipids and fatty acids are susceptible to oxidation upon microwave radiations which result in losses of their organoleptic and nutritional traits¹². Oxidation process increases with increasing microwave power and time. Yoshida and Kajimoto¹³ studied the stability of sesame oils upon microwave treatment. Limited studies were performed on the volatile oxidation compounds in microwave-treated oils and fats. Aldehydes such

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as hexaldehyde, phenylacetaldehyde and nonaldehyde were the major volatile oxidation compounds identified in microwave-treated rapeseed oils¹⁴.

Recently, cold-pressed oils have given an increase in interest as these edible oils have health-promoting properties. Cold pressing is being considered as an interesting substitute for traditional extraction because of consumers' desire for safe food. Cold pressing is a process that involves no thermal or chemical treatments to extract oil. In addition, cold pressing involves no refining process wherein the recovered lipids may contain a high level of natural antioxidants¹⁵⁻¹⁷.

The aim of this study was to investigate the effect of heating and microwave treatment on the levels of volatile oxidation products and the stability of safflower, sesame and canola cold-pressed oils.

2 Materials and methods

2.1 Materials

Cold-pressed safflower (*Carthamus tinctorius* L.), sesame (*Sesamum indicum*) and canola (*Brassica napus* L.) oils (without any added antioxidants) were purchased from a local oil producer in Turkey. All other chemicals and solvents were commercially of the highest grade and used without purification. Hexanal, α -thujene, limonene, *p*-cymene, γ -terpinene and nonanal were purchased from Sigma-Aldrich (St Louis, MO, USA).

2.2 Fatty acid composition of cold pressed oils

The fatty acid methyl ester (FAME) was prepared according to IUPCA¹⁸. The gas chromatograph (GC) analysis of the fatty acid composition was performed using a Shimadzu GC-2010 chromatograph. A DB-23 fused-silica column (30 m, 0.25 mm i.d., 0.25 μ m film thickness, Agilent J&W, USA) was used with helium as a carrier gas at a flow rate of 1 mL/min. The column temperature was isothermal at 190°C wherein the injector and detector temperatures were 230°C and 240°C, respectively. FAME was identified by comparison of their retention times with those of the reference standards.

2.3 Treatments of cold-pressed oils

2.3.1 Thermal treatment (oven test)

Two samples of each oil (10 g) were placed in a series of sealed brown glass bottles (30 mL) and heated for 12 days in a forced-draft air oven at 60°C. Oxidation was monitored in two day intervals during storage.

2.3.2 Microwave treatment

Samples in duplicate (10 g) were separately divided into a 30 mL opened brown bottle. Samples were microwave-treated at a constant frequency (2.450 MHz) and a power of 0.45 kW for two exposure times (2 and 4 min). A domestic

Samsung microwave oven (Model MW71E, Malaysia) was used in these experiments. The temperatures pertaining to the oils in the present work were measured after each microwave-application. Temperatures of canola, safflower and sesame oils in the oven reached 115.2, 169.8; 116.5, 171.8 and 129, 175°C at the two heating periods, respectively.

The volatile compounds as well as conjugated dienes (K_{232}) and trienes (K_{270}) values were used to follow oxidative changes in the heated and microwave-treated oil samples.

2.4 Conjugated dienes (CD), and conjugated trienes (CT)

Coefficients of specific extinction at the absorption wavelengths of 232 and 270 nm (K_{232} and K_{270}) were determined according to method Cd 18-90 of the AOCS Official Methods¹⁹.

2.5 Volatile compounds analysis

Two grams of the sample were placed in 20 mL headspace screw-top vial and allowed to equilibrate for 15 min at the constant temperature (35°C). The headspace was extracted for 45 min at 35°C using a CTC Combi PAL (CTC Analytics AG, Zwingen, Switzerland) autosampler with 75 μ m carboxen/polydimethylsiloxane (CAR/PDMS) solid phase micro extraction (SPME) fiber. Volatile compounds were desorbed by inserting the fiber for 10 min into the injection port of gas chromatography kept at 250°C.

Analysis of volatile compounds was performed with an Agilent model 7890 Series (Agilent Technologies, Santa Clara, CA, U.S.A.) gas chromatographer (GC) in combination with a CTC Combi PAL autosampler and an Agilent 5975 N mass selective detector (Agilent Technologies, Santa Clara, CA, U.S.A.). The compounds were separated in a capillary column, DB-624 (30 m length \times 0.25 mm ID \times 1.4 μ m film thickness, Agilent Technologies, Santa Clara, CA, U.S.A.) with the following temperature program: 40°C, hold for 5 min; 3°C/min up to 110°C; 4°C/min up to 150°C; 10°C/min up to 210°C, hold for 12 min. The temperatures for the injection port, ion source, quadrupole, and interface were set at 250°C, 230°C, 150°C, and 240°C, respectively. Mass spectra were obtained in the electron impact at 70 eV in full scan and a scan range from m/z 41 to 400.

Identification of compounds was detected by comparing mass spectra and Kovats index (KI) with the authentic standards and published data, as well as by comparing their mass spectra with the mass spectrometry library of Nist05 (National Institute of Standards and Technology, Gaithersburg MD, U.S.A.) and Wiley 7.0 (Wiley, New York, NY, U.S.A.). KI parameters were calculated using the series of n-hydrocarbons (C_4 to C_{20}).

3 Results and discussion

3.1 Fatty acid composition

Fatty acid composition of cold-pressed oil samples is presented in Table 1. The identified major fatty acids were unsaturated fatty acids, oleic and linoleic acids, ranged with 38.1% (sesame oil)-61.7% (canola oil) and 21.7% (canola oil)-49.9% (safflower oil), respectively. Palmitic acid was identified as the major saturated fatty acid and accounted for between 4.78% (canola oil) and 9.79% (sesame oil). The results are consistent with the results of Bauer *et al.*²⁰⁾ for canola oil, Gecgel *et al.*²¹⁾ for safflower oil, and Uzun *et al.*²²⁾ for sesame oil.

3.2 Thermal treatment (oven test)

Conjugated diene (CD) and triene (CT) values are good indicators to follow lipid oxidation. Changes in K_{232} and K_{270} values of cold-pressed oils during the storage at 60°C are shown in Figs. 1a and b, respectively. As expected, the K_{232} values of the oil samples increased with storage time. The initial values for canola, safflower and sesame oils increased from 0.38, 0.24 and 1.96 to 11.97, 35.26 and 6.21 at the end of storage time. A little increase in CD values of canola and sesame oils observed under oven test condition. However, higher changes occurred in K_{232} values for safflower oil during storage in comparison to other oils.

CT values of oil samples exhibited an increase trend for canola and safflower oils with storage time. However, a rapid increase observed in sesame oil after 2 days of storage and the K_{270} value of this oil showed stability after that time. The initial K_{270} value of canola, safflower and sesame oils were 0.16, 0.01 and 0.06, respectively. After 12 days of storage, CT values for the mentioned oils rise up to 1.06, 1.28 and 1.10, respectively (Fig. 1b).

The initial K_{232} and K_{270} values of cold-pressed canola oil

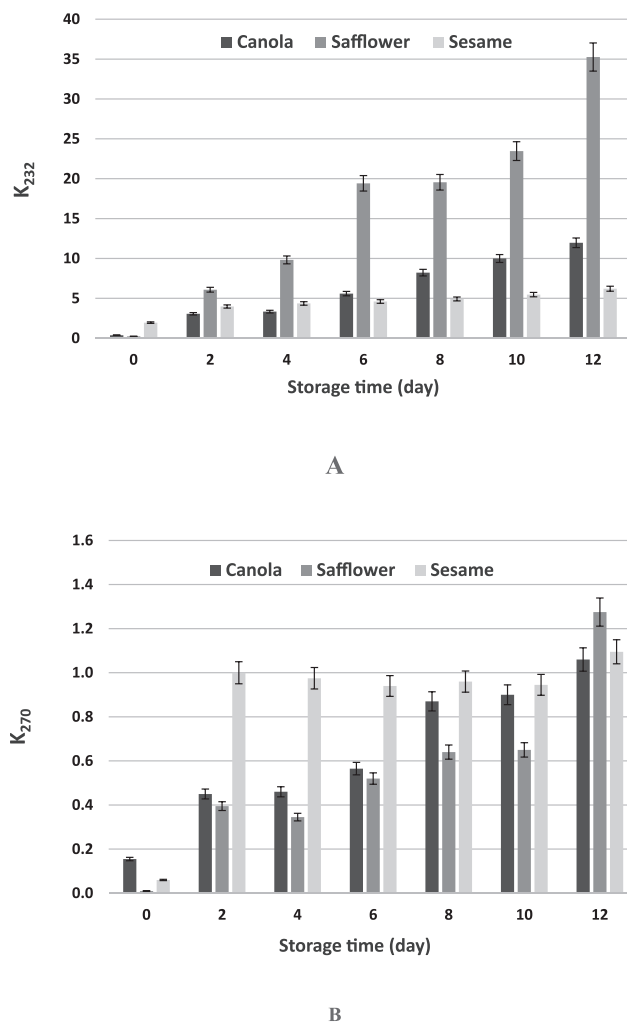


Fig. 1 Changes in K_{232} values (A) and K_{270} values (B) of cold-pressed oils upon heating at 60°C.

Table 1 Fatty acid composition of cold-pressed oils (relative percentage).

Fatty acid	Canola oil	Safflower oil	Sesame oil
Myristic, C14:0	0.05	0.09	0.01
Palmitic, C16:0	4.78	5.62	9.79
Palmitoleic, C16:1	0.19	0.11	0.16
Heptadecanoic, C17:0	0.03	0.03	0.03
Heptadecenoic, C17:1	0.02	0.03	0.02
Stearic, C18:0	1.59	1.84	4.86
Oleic, C18:1	61.78	41.49	38.12
Linoleic, C18:2	21.72	49.93	45.99
Linolenic, C18:3	8.04	0.07	0.37
Arachidic, C20:0	0.45	0.32	0.45
Eicosenoic, C20:1	1.02	0.15	0.11
Eicosadienoic, C20:2	0.15	0.21	0.05
Lignoceric, C24:0	0.17	0.10	0.04

were similar to those observed in cold-pressed canola oil²³⁾. The CD and CT values of sesame and safflower oils were lower than that of sesame oil from Macedonia²⁴⁾ and also that of sesame and safflower oils²⁵⁾.

A few research associated with storage of cold-pressed oils have been reported in the literature. Abuzaytoun and Shahidi²⁶⁾ showed upward tendency for CD value of cold-pressed hempseed oil and flaxseed oil upon heating. Khan and Shahidi²⁷⁾ reported that CD value of cold-pressed borage oil reached a maximum value of 20.1 at 60°C for 120 h and kept this value for 168 h. There was no direct comparison between our data with these mentioned literatures because of the differences of oils and analysis method used in these works. These literature indicated that these differences on CD values among the oils were associated with fatty acid composition especially unsaturated fatty acids. A rapid increase trend for CD values of oil samples observed during storage under oven test condition (60°C) might be because of unsaturated fatty acid composition especially linolenic and linoleic acids which are rapidly oxidized at these conditions. In our study, safflower oil has higher linoleic acid content among the oil samples.

The evolution of the volatile compounds during oven test (60°C) is presented in **Table 2**. The major identified and analyzed aldehydes were hexanal, 2-heptenal and 2,4-heptadienal isomers for canola oil, as well as hexanal and 2-heptenal for safflower and sesame oil. The major volatile compounds were found as aldehydes during thermal oxidation. Hexanal arises from linoleic acid and is a good indicator for lipid oxidation. Hexanal content of canola, safflower and sesame oil increased up to 6.03, 12.05, and 6.27×10^6 AU at the end of storage, respectively. (E)-2-heptenal is well known and important volatile aldehyde induced during linoleic acid oxidation wherein the odor threshold of this compound is very low with 0.001 mg/kg²⁸⁾. The content of this compound in canola, safflower and sesame oil increased to 8.03, 27.10 and 2.30×10^6 AU at the end of storage, respectively. 2,4-heptadienal isomers were not identified according to the isomer structure. 2,4-heptadienal isomers were only detected in canola oil. The first isomer identified at initial time of experiment and the content of the first isomer increased with increasing thermal oxidation time. The second isomer induced in canola oil after 8 days of storage and its content increased with oxidation time.

Jelen *et al.*²⁹⁾ stated that hexanal was the most abundant volatile oxidation compound in fresh cold-pressed oil and also pointed out that 2-heptenal was the major volatile aldehyde after 10 days storage at 60°C. Torres *et al.*³⁰⁾ reported that major volatile compounds were hexanal, 2-heptenal, 2,4-decadienal, nonanal and 2,4-heptadienal in soybean oil after 10 days storage at 60°C. Beltrán *et al.*³¹⁾ reported that hexanal, (E)-2-heptenal, (E)-2-octenal, nonanal, (E)-2-nonanal, (E,E)-2,4-nonadienal and (E,E)-

-2,4-decadienal were the major aldehydes in almond oils during thermal oxidation at 100°C. This work also declared that American almond cultivar Butte had the highest content of (E)-2-heptenal, (E)-2-octenal, (E,E)-2,4-decadienal and (E,E)-2,4-nonadienal in comparison with the Spanish almond cultivars during thermal oxidation. This situation could be explained that linoleic acid content of American almond cultivar were higher than the Spanish cultivars.

At the late stages of oxidation experiment, 2-heptenal was determined as the major volatile oxidation product in the oil samples. This observation was monitored in the cold-pressed oil oxidation at 60°C²⁹⁾ wherein 2,4-hexadienal isomers took place an important part of the volatile oxidation compounds during oxidation of canola oil. These compounds were identified and their content were richer among the volatile oxidation products during thermal oxidation of soybean oil³⁰⁾ and almond oil³¹⁾.

Beside to these major aldehydes, nonanal was found in all samples during oxidation. After 10 days of storage at 60°C, nonanal content reached up to the maximum level in oils. (E)-2-decenal was the other identified aldehyde found in safflower and sesame oils wherein the content of these compounds generally exhibited a rough trend at thermal oxidation conditions. (E)-2-decenal observed in different packaged olive oils under three storage temperatures (15, 30 and 40°C). The similar trend was observed by Kanavouros *et al.*²⁴⁾ who reported that the differences in the levels of this compound could be related the form of singlet or triplet oxygen present in oil. (E,Z)-2,4-decadienal was only found in safflower oil and this compound was present in the maximum level at the end of storage. However, (E,E)-2,4-decadienal found in the fresh canola oil and this compound disappeared with thermal oxidation. Besides, this compound was appeared in safflower oil and the content of this compound reached up to the highest level among oils during storage. (E,E)-2,4-decadienal was detected in the oxidized oils (olive, soybean, sunflower, peanut and rapeseed oils) and its content increased in the oils stored for 5 days at 60°C³²⁾. The note was peculiar to cold-pressed oils and this sensory situation of these oils could be related to the presence of 2,4-decadienal or hexanal^{33, 34)}. The presence of 2,4-decadienals in our fresh oils could be explained by this expression and also the similar behavior observed in our samples for hexanal during storage. Its content in the oils decreased up to a certain time during oxidation. Heptenal was the last identified aldehyde and appeared only in safflower oil at the end of storage.

6-methyl 5-hepten-2-one was identified in canola oil and its content showed a wavy behavior during oxidation. This behavior for canola oil are not in agreement with the results of Jeleñet *et al.*³⁵⁾ who reported that this compound increased in refined rapeseed oil with increasing oxidation time at 60°C. The other identified ketone was 3,5-octadien-

Table 2 Changes in volatile compounds of oils during oven test.

Compounds	KI ^A	Rt ^B	Canola oil												Safflower oil												Sesame oil											
			Storage time (day)												Storage time (day)												Storage time (day)											
			0	2	4	6	8	10	12	0	2	4	6	8	10	12	0	2	4	6	8	10	12															
Hexanal	837	a	6.29	3.21	1.59	2.84	2.85	4.06	6.03	5.31	2.45	3.91	11.53	6.16	7.64	12.05	5.31	2.45	3.91	11.53	6.16	7.64	12.05	3.95	1.91	2.14	3.62	3.51	5.73	6.27								
2-hexenal	905	b	-	-	-	-	-	-	0.66	-	-	-	-	-	1.04	2.61	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
2-heptanone	934	a	-	-	-	-	-	-	nd	-	-	-	-	-	-	0.43	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
Heptanal	942	a	-	-	-	-	-	-	nd	-	-	-	-	-	0.53	0.62	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
2-heptenal	1011	b	0.73	0.57	0.52	1.21	2.28	3.58	8.03	0.60	0.92	2.36	5.49	5.74	11.99	27.10	0.60	0.92	2.36	5.49	5.74	11.99	27.10	4.00	3.06	2.55	3.20	2.51	2.30	2.30								
6-methyl-5-hepten-2-one	1033	b	0.72	0.85	0.66	0.67	0.32	0.39	0.53	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
limonene	1046	a	0.76	1.67	1.49	1.39	0.41	0.61	1.32	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
p-cymene	1051	b	4.42	9.27	9.39	10.98	1.66	2.24	3.38	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
2,4-heptadienal (isomer 1)	1060	b	1.63	1.96	1.31	2.32	3.01	4.01	6.47	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
2,4-heptadienal (isomer 2)	1075	b	-	-	-	-	-	1.73	2.48	5.98	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
3-octen-2-one	1093	b	-	-	-	-	-	-	-	-	-	-	-	-	0.43	0.77	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
3,5-octadien-2-one	1133	b	0.38	0.42	0.37	0.83	1.28	1.50	2.38	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
Nonanal	1151	a	0.66	1.39	1.19	0.80	0.88	1.47	2.37	0.51	1.09	2.17	1.14	0.87	1.58	1.34	0.51	1.09	2.17	1.14	0.87	1.58	1.34	0.49	1.50	0.57	0.64	0.65	1.78	0.64								
Thymoquinone	1324	b	0.40	1.94	3.63	4.98	2.17	2.90	2.85	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
(E)-2-decenal	1326	b	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-								
(E,Z)-2,4-decadienal	1367	b	-	-	-	-	-	-	-	0.20	-	-	-	-	-	-	0.20	-	-	-	-	-	-	-	-	-	-	-	-	-								
(E,E)-2,4-decadienal	1392	b	0.69	-	-	-	-	-	-	0.60	-	-	-	-	-	-	0.60	-	-	-	-	-	-	-	-	-	-	-	-	-								

^A KI = Kovats index calculated for DB-624 capillary column (J&W Scientific; 30 m, 0.25 mm id, 1.4 µm film thickness) installed on a gas chromatography equipped with a mass-selective detector.

^B Rt = reliability of identification; a = mass spectrum and retention time identical with authentic sample; b = mass spectrum and Kovats index from literature in accordance.

^C Results are expressed as means of total ion current (TIC) area units ($\times 10^{-6}$).

2-one which present only in canola oil and its content increased in the oxidized oil during storage. The similar increase trend for canola oil with oxidation was observed in the results of Mildner-Szkudlarz *et al.*³²⁾. 3-octen-2-one and 2-heptanone were found as ketones only in safflower oil at the late of thermal oxidation at 60°C. These compounds were found in refined and cold-pressed oil treated with conventional heating at 60°C²⁹⁾.

The other identified compound group was terpenes including limonene, *p*-cymene and thymoquinone in canola oil. Limonene was found in the volatile fraction of refined and cold-pressed rapeseed oil²⁹⁾. The terpenes were detected in the headspace of sunflower oil and a slightly higher concentrations of these compounds found in the oil samples with different storage conditions (at room temperature in closed receptacles and in the presence of limited amounts of air) as reported by Guillen and Goicoechea¹⁾.

3.3 Microwave treatment

Figure 2a shows the changes in K_{232} values of oil samples during microwave heating. A marked increase was observed in K_{232} values for all oils treated with microwave. The K_{232} values of canola, safflower and sesame oils were reached up to 3.01, 4.22 and 4.49, respectively. Regarding K_{270} values of oil samples, this value for canola and safflower increased gradually but its value for sesame oil raised suddenly from 0 to 2 min of microwave application. K_{270} values of canola, safflower and sesame oil reached up to 0.64, 0.64 and 1.10, respectively (Fig. 2b).

These results obtained for K_{232} and K_{270} values during microwave treatment were in agreement with those obtained by Albi *et al.*³⁶⁾ who reported that these values in edible oil samples increased and raised up to higher values with increasing the microwave treatment time. They also recorded that the effect of microwave treatment on K_{232} and K_{270} values were more efficient than conventional heating in edible oil samples. These results also bear out the observations of Dandjouma *et al.*³⁷⁾ who reported that the extinction coefficients in *Canarium schweinfurthii* Engl. oil increased rapidly according to the increase of microwave power and time. These situation could be explained that the effect of microwave energy on radical formation were greater than the effect of conventional heating¹³⁾.

Regarding the volatile compounds, *p*-cymene was found as the major volatile compound, followed by hexanal in microwave-treated canola oil. 2-heptenal was the other identified aldehyde and increased to the maximum value after 4 min exposure. After 4 min exposure time, 2,4-heptadienal, nonanal and (E,E)-2,4-decadienal were determined among the volatile compounds in canola oil and these aldehydes increased with increasing microwave treatment time. The other identified terpenes were α -thujene, limonene, γ -terpinene and thymoquinone. α -thujene and γ -terpinene were found only in the samples treated with

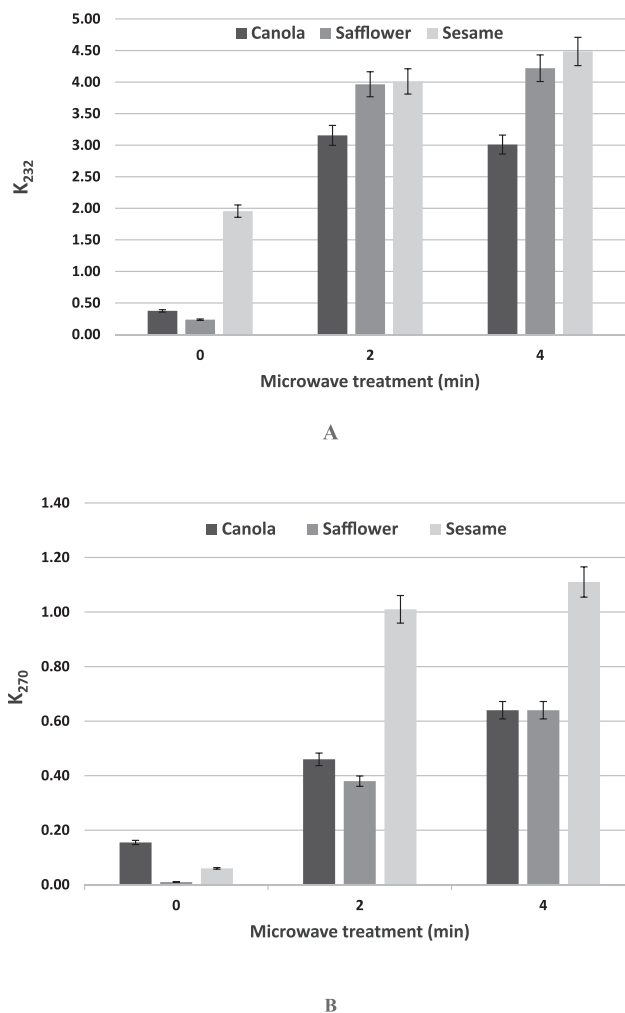


Fig. 2 Changes in K_{232} values (A) and K_{270} values (B) of cold-pressed oils upon microwave treatment.

microwave compared to ones treated with convective heating. 6-methyl-5-hepten-2-one disappeared after 4 min exposure time. Higher levels of hexanal and 2-heptenal were found in the oxidized oils upon microwave treatment. Hexanal content increased with the exposure time and 2-heptenal was determined in only one sample after 4 min application. 2-Hexenal, nonanal, (E,Z)-2,4-decadienal and (E,E)-2,4-decadienal were the other identified volatile compounds and all of them appeared after 4 min. Hexanal and 2-heptenal were the major volatile compounds in sesame oil oxidized with microwave. 2-Hexenal, heptanal, (E)-2-octenal, nonanal, (E)-2-decenal, (E,Z)-2,4-decadienal and (E,E)-2,4-decadienal were the other identified volatile compounds in sesame oil during microwave treatment. *p*-Cymene and γ -terpinene were found in the sesame oil samples as terpenes. (E)-2-octene is a hydrocarbon found in sesame oil and occurred upon microwave treatment.

(E)-2-Decenal, (E)-2-undecenal, (E,E)- and (E,Z)

Table 3 Changes in volatile compounds of oils as affected by microwave heating.

Compound	KI ^A	RI ^B	Canola oil		Safflower oil		Sesame oil	
			Exposure time (min)		Exposure time (min)		Exposure time (min)	
			2	4	2	4	2	4
(E)-2-octene	808	a	–	–	–	–	1.16	0.74
Hexanal	837	a	8.01	5.68	5.59	9.56	4.67	8.32
2-hexenal	905	b	–	–	–	0.86	–	1.23
α -thujene	938	a	2.76	1.20	–	–	–	–
Heptanal	942	a	–	–	–	–	–	0.57
2-heptenal	1011	b	–	3.07	–	7.18	3.60	13.81
6-methyl-5-hepten-2-one	1033	b	0.99	–	–	–	–	–
limonene	1046	a	2.08	1.36	–	–	–	–
<i>p</i> -cymene	1051	b	16.49	10.19	–	–	0.58	–
2,4-heptadienal	1060	b	1.71	3.21	–	–	–	–
γ -terpinene	1075	a	1.27	3.73	–	–	0.31	–
(E)-2-octenal	1117	b	–	–	–	–	–	1.62
Nonanal	1151	a	0.65	2.07	–	1.38	0.33	1.67
Thymoquinone	1324	b	0.99	3.77	–	–	–	–
(E)-2-decenal	1326	b	–	–	–	–	–	0.48
(E,Z)-2,4-decadienal	1367	b	–	–	–	0.87	–	0.25
(E,E)-2,4-decadienal	1392	b	–	0.65	–	1.87	–	0.78

^A KI = Kovats index calculated for DB-624 capillary column (J&W Scientific; 30 m, 0.25 mm id, 1.4 μ m film thickness) installed on a gas chromatography equipped with a mass-selective detector.

^B RI = reliability of identification; a = mass spectrum and retention time identical with authentic sample; b = mass spectrum and Kovats index from literature in accordance.

^C Results are expressed as means of total ion current (TIC) area units ($\times 10^{-6}$).

-2,4-decadienal were identified compounds found upon microwave treatment in olive oil. Beside to these aldehydes, pentanal, hexanal, heptanal, (E)-2-hexenal, octanal, (E)-2-heptenal and nonanal were found in oxidized samples³⁸⁾. Some of identified compounds in our oil samples were consistent with the results of Cossignani *et al.*³⁸⁾. There was no literature information about the effects of microwave treatment on the volatile oxidation compounds of cold-pressed oils, therefore, no comparison could be done with the literatures.

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