

## Changes occurring in vegetable oils composition due to microwave heating

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### RESUMEN

#### Cambios producidos en la composición de aceites vegetales debido al calentamiento en microondas.

Se estudia el efecto del calentamiento en horno de microondas sobre aceites de diferente composición en ácidos grasos. Aceites de girasol, soja, cacahuete y una mezcla de soja y cacahuete al 50%, se calentaron durante 2, 4, 6, 8, 10, 12, 15 y 18 minutos. Los valores de índice de peróxidos, acidez libre y absorbancia a 420 nm fueron proporcionales al tiempo de calentamiento. Otras determinaciones incluyeron el contenido total en tocoferoles mediante cromatografía en capa fina, la composición en ácidos grasos y en epoxiácidos mediante cromatografía gas líquido, y la formación de dienos y trienos conjugados mediante espectrofotometría UV. Los resultados indicaron que el contenido en tocoferoles disminuyó con el tiempo de calentamiento dependiendo del tipo de tocoferol predominante y que los cambios estaban relacionados con los encontrados para el índice de peróxidos. Los ácidos polinsaturados también decrecieron con el tiempo de calentamiento. Finalmente, los resultados obtenidos con la mezcla de aceites de soja y cacahuete fueron muy útiles para interpretar los resultados obtenidos con los aceites individuales.

**PALABRAS-CLAVE:** *Aceite vegetal – Calentamiento – Cambios químicos – Microondas.*

### SUMMARY

#### Changes occurring in vegetable oils composition due to microwave heating.

The effect of microwave heating on three vegetable oils having different lipid compositions was studied. Sunflower, soybean and peanut oils in comparison with oil admixture of soybean and peanut oil (1:1, w/w), were selected for this study. Each oil was heated for 2, 4, 6, 8, 10, 12, 15 and 18 minutes in microwave oven. Peroxide value, free acidity and colour absorbance (at 420 nm) were proportionally increasing with the increase of heating period. Colour absorption threw light on the formation of browning products arising from phospholipids during microwave heating. Total tocopherol contents were determined by preparative thin layer chromatography, whereas the fatty acid compositions and formed epoxy acid were analyzed by capillary gas liquid chromatography. The formed conjugated dienes and trienes were determined by UV spectrophotometry. It was found that the total tocopherols of the microwave heated oils, decreased depending on the type of the predominating tocopherols. Also a relation of peroxide formation, during microwave heating, with changes in total tocopherol composition was discussed. It was found that polyunsaturated fatty acids generally decreased by increasing the heating period. The results obtained from the heated oil admixture helped interpret the results obtained from other heated individual oils.

**KEY-WORDS:** *Chemical changes – Heating – Microwave – Vegetable oil.*

### 1. INTRODUCTION

Microwave energy, with its unique heating ability, offers many advantages for both home and industrial food applications such as baking, cooking, thawing, blanching, dehydration, pasteurization, sterilization and tempering, also effects of microwave on food constituents as well as the formation of flavors and colors in food have been studied (Decareau, 1985; Riley, 1985; Rosenberg and Bogl, 1987; IFT, 1989; Giese, 1992). Microwave ovens are credited with rapid heating rates and high efficiency, especially because of their high penetration power (Riley, 1985; IFT, 1989; Burfoot et al., 1990). Numerous investigations have been done to study the effects of microwave on food constituents and various chemical reactions are induced by microwave energy (Finot, 1995). Lipid components of animal fats and vegetable oils microwave heated were also investigated (Yoshida et al., 1990; Yoshida et al., 1992; Farag, 1994). It was found that the rate of quality deterioration, such as oxidation, depends on the polyunsaturated fatty acid content (Yoshida et al., 1990; Lin et al., 1999).

In vegetable oils exposed to microwave energy, the higher the amount of polyunsaturated fatty acids in the oils, the greater was the rate of quality deterioration of the oils. The levels of free fatty acids also increased in vegetable oil heated in microwave oven (Yoshida et al., 1992). Thus, the changes in olive oil composition due to microwave heating were studied by Cossignani et al. (Cossignani et al., 1998). The traditional parameters, including free acidity, peroxide value and ultraviolet absorbance values, were determined.

Since tocopherols are very important biological and nutritive food components, the effect of microwave heating on the soybean tocopherols was studied by Yoshida and Kajimoto (Yoshida and Kajimoto, 1989). They showed that the effects of microwave treatment on the total tocopherols content of soybean oil gradually decreased by heating time.

In the present work, the objective was to study the influence of microwave heating on the three selected oils, i.e., sunflower, soybean and peanut oils, having different fatty acid and tocopherol compositions. In addition to these oils, a binary oil admixture from

peanut and soybean oils (1:1 w/w) was prepared as a model to help interpret some results concerning the microwave heating of different oils having different contents of polyunsaturase and tocopherols.

## 2. MATERIALS AND METHODS

### 2.1. Materials

Three oil seeds, namely, sunflower, soybean and peanut (Season, 2000) were purchased from Oilseed Crop Department, Ministry of Agriculture, Giza.

### 2.2. Methods

#### 2.2.1. Lipid Extraction

Representative oilseed samples of sunflower, soybean and peanut (500g of each) were ground and soaked in chloroform-methanol (2:1, v/v) with intermittent stirring to extract the oil. The extract of each sample was collected, filtered, dried over anhydrous sodium sulphate and then evaporated under reduced pressure at 50°C in a rotary evaporator and the samples were kept in stoppered dark glass vials in the refrigerator till analysis. In addition to the three extracted oils (sunflower, soybean and peanut), a binary oil admixture of soybean and peanut, in an equal weight ratio, was prepared as a model.

#### 2.2.2. Microwave Heating (MW)

A microwave oven (Samsung model 9245, MB245) having frequency of 2,450 MHz, was used for heating the oil samples. Eight samples of each oil (16 g each) were placed in eight petri-dishes (7 cm diameter). The samples of each oil were heated in the MW oven for 2, 4, 6, 8, 10, 12, 15 and 18 minutes. Temperatures in the oven reached 120°, 160°, 176°, 197°, 210°, 216°, 222° and 227°C at the respective heating periods. Each sample removed from the oven was kept immediately in a vacuum dissicator to cool it down to room temperature. The samples were kept in dark glass vials in a refrigerator till analysis.

#### 2.2.3. Determination of Peroxide and Acid Values

Peroxide value and acid value were determined according to Official American Oil Chemist's Society (AOCS) methods (AOCS, 1985). The values were expressed as meq of peroxide O<sub>2</sub> / kg oil and mg KOH / g oil, respectively.

#### 2.2.4. Color Index (CI)

As an index of colour development during MW heating the spectrophotometric absorbance at 420 nm of a 2.5% w/v solution of oil in isooctane was determined using a Shimadzu Spectrophotometer (Yoshida and Kajimoto, 1989).

#### 2.2.5. Determination of Tocopherol Content

(By preparative thin layer chromatography)

Owing to the difficulties usually encountered in isolating all individual tocopherols for their GLC analysis (Dirks, 1965; Slover et al., 1967), it was advisable to determine total tocopherols quantitatively by preparative thin layer chromatography (PTLC). Thus, a sample of methyl ester of each oil (previously transesterified (Barford et al., 1963) was accurately weighed and applied as streaks on silica gel plates (300μ thickness of silica gel 60 GF<sub>254</sub> nm, E.Merk). Standard tocopherol mixture [alpha, gamma and delta tocopherols, supplied from Nisshin Oil Mills (Japan)] was applied as reference on one side of the plates. The plates were developed with two successive solvent systems, 80/20/1 and 70/30/1 v/v/v hexane/diethyl ether/glacial acetic acid. The tocopherols were detected on the chromatoplates as purple spots under ultra-violet light. The different tocopherol zones (located with the help of the standard tocopherols) were scraped of the plates, extracted twice with moistened diethyl ether, dried over anhydrous sodium sulphate and filtered. The ether was evaporated under reduced pressure and the residue was accurately weighed using microanalytical balance (Mettler M5). The results were the mean value of three determinations and expressed as mg total tocopherols per 100g oil. It is worthy to mention that standard tocopherols mixture was subjected to PTLC and the recovery was found to be more than 94%.

#### 2.2.6. Fatty Acid Methyl Ester

The oil samples were converted to their methyl esters via transesterification using methanol containing 0.04 N sodium methylate as a catalyst (Barford et al., 1963). Hewlett Packard Model 6890 gas chromatograph was employed for the analysis of fatty acid methyl esters under the following conditions: HP- INNOWax capillary column (polyethylene glycol), 30.0 m x 530 μm, film thickness 1.0 μm; column temperature, 120-240°C at a rate of 10°C min<sup>-1</sup>; injection temperature, 250°C; split ratio, 8:1; split flow, 120 ml min<sup>-1</sup>; gas saver, 20 ml min<sup>-1</sup>; and carrier gas, N<sub>2</sub> with flow rate 15ml min<sup>-1</sup>; FID detector temperature, 280°C; hydrogen flow rate, 30 ml min<sup>-1</sup> and air flow rate, 300 ml min<sup>-1</sup>. Peak areas

were determined by electronic integrator and percentage composition of fatty acids was automatically calculated. Standard mixture of fatty acids methyl esters was chromatographed under the same operating conditions.

#### 2.2.7. Preparation of Epoxy Methyl Esters

A sample of mixed fatty acid methyl esters of soybean oil was subjected to partial epoxidation using in-situ formed peracetic acid in the presence of acid-cation exchanger (El-Mallah and El-Shami, 1975). The epoxy components were identified with the help of standard GLC chromatogram (El-Mallah et al., 1983) and thin layer chromatography-picration technique (Stahl, 1966; Fioritti et al., 1966).

#### 2.2.8. Conjugated Fatty Acids

Shimadzu Spectrophotometer UV-240 was used in measuring the conjugated fatty acids (conjugated dienes and trienes) formed through MW heating of oil samples. Absorbance at 234 and 268 nm were used for determining conjugated dienes and trienes, respectively (AOCS, 1985).

### 2.3. Expression of the Results

All the results are the mean values of more than two experimental replications ( $n=2-4$ ) for each experimental analysis.

## 3. RESULTS AND DISCUSSION

The PV was taken as a measure for the degree of oxidation during the MW heating of oil samples. The results are graphically represented in Fig. 1. It is observed that the PVs of the three oils as well as the admixture increased gradually as MW heating is progressing till they reached higher values at 18 min heating. From these results it can be seen that the rate of peroxide formation in sunflower oil during MW heating, is higher than that in other oils. Accordingly, it seems that the rates of peroxide formation in the tested oils are arranged in following descending order: sunflower oil > peanut oil > oil admixture > soybean oil.

With respect to the changes of AV Fig.2, it can be observed that, the AV of the different oils subjected to MW heating at different time intervals, increased generally by time of heating. The increase in acidity is undoubtedly due to the splitting of ester linkages of triglyceride molecules as a result of heating (Yoshida et al., 1992).

Concerning the changes in colour (when checked by naked eye), it was found that with the progress of MW heating of soybean oil, colour changes gradually from yellow-brown at 2-10 min heating to light brown at 15 min heating. Finally-deep brown colour was

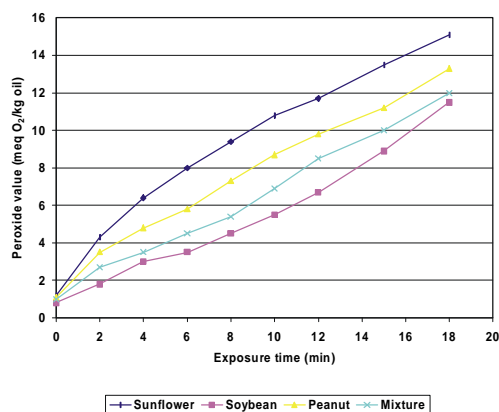


Figure 1

Relationship between peroxide value and exposure time during microwave treatment at frequency of 2,450 MHz. Each value is an average of three to four determinations and is expressed as meq O<sub>2</sub>/kg oil.

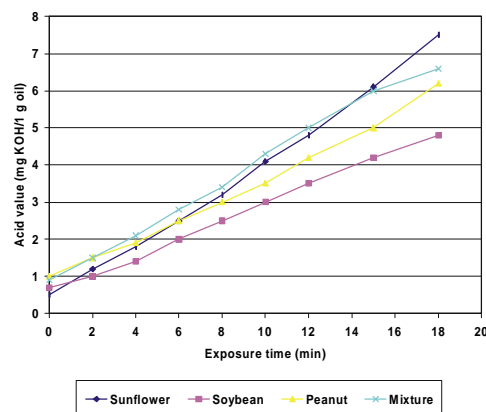


Figure 2

Relationship between acid value and exposure time during microwave treatment at frequency of 2,450 MHz. Each value is an average of three to four determinations and is expressed as mg KOH/1g oil.

observed at 15-18 min heating. It is worthy to mention that a burnt odour appeared after 6 min heating. However, the changes in colour of the other oils are comparatively lower. The absorbance at 420 nm was taken as a measure of colour index (Yoshida and Kajimoto, 1989) during MW heating of oils (Fig. 3), it was found that soybean oil gave higher colour index at longer heating periods, while the other oils gave a light coloured oils even at prolonged heating periods. Thus the intensity of the colour at 420 nm in the oils during different MW heating periods can be arranged in the following order: soybean oil > oil

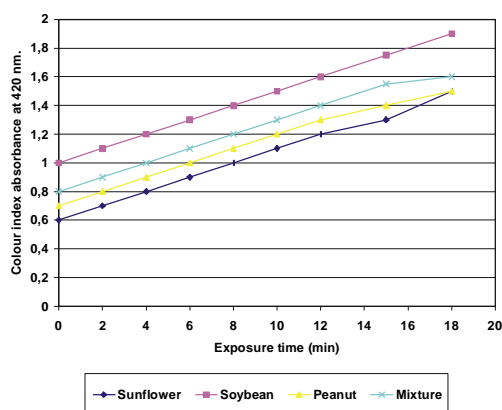


Figure 3  
Effect of microwave treatment on oil colour index.  
Each value is an average of three to four determination and is expressed in terms of absorbance at 420 nm.

admixture > peanut oil > sunflower oil. This shows that the soybean oil colour is more affected by MW heating than the other oils, whereas sunflower oil is less affected than any of the other oils. It is worthy to mention that, the darkening of oils during MW heating may be due to the presence of phospholipids in the oil that develop browning products having antioxidant activity (Dziedzic, 1984; Hudson and Ghavami, 1984; Husain et al., 1986; Frankel, 1993).

Before discussing changes of tocopherols in MW heated oils it is worthy to mention that alpha-tocopherols is the major tocopherol in sunflower oil (Kanematsu et al., 1983; El-Shami et al., 1994) whereas gamma- and delta-tocopherols are predominating in soybean oil (Kanematsu et al., 1983; Minar and Hassanein, 1991). However, in peanut oil alpha- and gamma-tocopherols are the major tocopherol (Kanematsu et al., 1983).

It was found feasible and simple to determine total tocopherol contents of the oils with the help of PTLC and the results are represented graphically in Fig. 4. It was observed that, generally, there is a decrease in total tocopherol content in all oil samples as MW heating was proceeding. Thus in soybean oil the total tocopherol content decreased from 120 mg/100g oil to 114, 100, 91, 80 and 75 mg/100g oil at 0, 2, 6, 10, 15 and 18 min heating respectively. While, the total tocopherol content in sunflower oil decreased slightly from 70 mg/100g oil to 67, 66, 64, 63 and 58 mg/100g oil, whereas, the total tocopherol contents in peanut oil decreased from 25 mg/100g oil to 22, 19, 16, 13 and 8 mg/100g oil. On the other side, the total tocopherol content in oil admixture decreased from 75 mg/100g oil to 71, 67, 63, 55 and 50 mg/100g oil during the selected MW heating periods 0, 2, 6, 10,

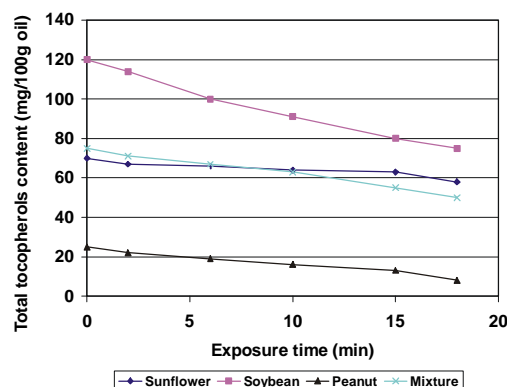


Figure 4  
Changes in the total tocopherol content of oils during microwave treatment at frequency of 2,450 MHz. Each value is an average of three to four determination and is expressed as mg/100g oil.

15 and 18 min respectively. Table III shows the loss percentage in total tocopherol contents of the oil samples and the oil admixture as a result of MW heating. It can be seen that peanut oil is highly affected by MW heating since it exhibits highest percentage loss in tocopherol content during MW heating.

The interpretation of the changes in peroxide value can be attributed to the tocopherol type predominating in the tocopherol composition. Since sunflower oil contains principally alpha-tocopherol, exhibiting lowest antioxidant activity, the rate of peroxide formation is markedly higher than those in other oils. In contrast, soybean oil, containing high antioxidant potency tocopherols (gamma- and delta-tocopherols) resists efficiently the peroxide formation (Herb et al, 1964; Khafizov et al., 1975; Ikeda and Fukuzumi, 1977; Yoshida and Kajimoto, 1989; Yoshida et al., 1992). It can be concluded that the gamma- and delta-tocopherols, as more powerful antioxidants than alpha-tocopherol, can protect oils (Yoshida and Kajimoto, 1989). However, peroxide formation in peanut oil (having the lowest content of tocopherols but nearly equal amounts of alpha- and gamma-tocopherol) is slightly lower than that in sunflower oil. By admixing peanut oil with soybean oil in equal proportions, the stability is improved against oxidation during MW heating as indicated from the peroxide curves in Fig.1. These results, represented graphically, confirmed those obtained from soybean and peanut oils. Thus, the peroxide formation curve of the oil admixture is an intermediate between soybean and peanut oil curves.

Fatty acid composition at selected heating periods was determined to follow any significant changes in

Table I  
Fatty acid composition of oil samples subjected to microwave heating determined by gas liquid chromatography

Oils subjected to MWH*	Sunflower oil				Soybean oil				Peanut oil				Oil admixture (soybean : peanut oil, 1:1 w/w)			
Heating Time (minutes)	0	6	10	18	0	6	10	18	0	6	10	18	0	6	10	18
Fatty acid composition(%)																
Myristic	0.2	0.2	0.2	0.3	0.4	0.3	0.4	0.5	0.0	0.0	0.0	0.0	0.2	0.2	0.3	0.2
Palmitic	9.5	9.2	9.3	9.3	8.5	9.0	9.3	9.3	8.8	8.9	8.8	9.4	9.2	9.3	9.5	9.4
Stearic	0.1	0.1	0.0	0.0	3.6	3.5	3.6	3.9	2.9	2.9	2.8	2.9	3.2	3.3	3.0	3.4
Oleic	55.0	54.2	53.5	53.0	21.9	20.5	20.0	19.5	48.6	46.0	45.7	44.5	35.3	34.0	33.4	32.9
Linoleic	32.1	31.5	30.0	29.6	57.1	55.2	54.0	53.0	32.2	31.0	30.5	30.0	44.9	44.0	43.5	43.0
Linolenic	0.3	0.3	0.3	0.5	6.1	5.8	5.2	5.5	1.5	1.6	1.2	1.4	4.0	3.6	3.4	3.5
C <sub>20</sub> -C <sub>24</sub>	2.8	2.0	3.5	4.5	2.4	2.8	5.1	3.8	6.0	6.9	7.0	8.0	3.2	3.3	3.8	3.7
Others	0.0	2.3	2.9	2.6	0.0	2.4	2.2	4.4	0.0	2.4	3.6	3.6	0.0	2.1	2.9	3.8
Total oxygenated (epoxides)	0.0	0.2	0.3	0.2	0.0	0.5	0.2	0.1	0.0	0.3	0.4	0.2	0.0	0.2	0.2	0.1

\* MWH : Microwave heating.

\* Each value represents an average of three to four determinations.

Table II  
UV Spectrophotometric determination of the formed conjugated dienes and trienes in oils subjected to MW heating

Heating time (minute)	Diene % (234 nm) in MW heated oils				Triene % (268 nm) in MW heated oils			
	Sunflower oil	Soybean Oil	Peanut oil	Oil admixture (soybean : peanut oil, 1:1 w/w)	Sunflower oil	Soybean Oil	Peanut oil	Oil admixture (soybean : peanut oil, 1:1 w/w)
0	0.0525	0.2150	0.3701	0.2512	0.0150	0.0042	0.0738	0.0411
6	0.5733	0.3956	0.5287	0.4040	0.0991	0.0583	0.0791	0.0507
10	0.9810	0.7509	0.7484	0.6241	0.0648	0.1565	0.0972	0.0950
18	1.0698	1.0483	0.8592	0.8720	0.1988	0.2013	0.1250	0.2132

\* Each value represents an average of three to four determinations.

fatty acid profile and to detect the formation of any unusual oxygenated compounds (Table I). It was found that oleic and linoleic acids generally decreased by prolonged heating. On the other hand it was also able to identify two epoxy components as trace amounts that stood for epoxy oleate and epoxy stearate as compared with the chromatograms of the prepared partially epoxidized soybean oil. It is worthy to mention that these epoxy components were firstly identified by chromatographic picration technique (El-Mallah and El-Shami, 1975; El-Mallah et al., 1983).

With respect to conjugated fatty acid formation during MW heating, it was found that as MW heating was progressing, the amount of conjugated dienes and trienes gradually increased (Table II). It is worthy to mention that the conjugated dienes were formed at, somewhat, higher levels than the conjugated trienes. The conjugation of polyunsaturates can be associated with the oxidation process during MW heating (Frankel, 1961; Herb et al., 1964; Hamilton,

1983; Megahed, 2001). From the results obtained, the following conclusions were derived out:

1. The tocopherols are unstable in the presence of unsaturated fats, particularly those rich in polyunsaturates and losses in tocopherols may be attributed mainly to degradation and partly to the peroxidation of the unsaturated oils (Yoshida and Kajimoto, 1989). Accordingly, oils containing the higher polyunsaturates content, cause losses in tocopherol content during MW heating (Nelis et al., 1985; Yoshida and Kajimoto, 1989).

2. MW heating causes the formation of very lower amounts of some oxygenated compounds, particularly epoxides that can be subjected to decomposition at prolonged MW heating.

In addition, it was reported that the phospholipid constituents of the oils, even in lower concentrations, could act synergistically with tocopherols at higher temperatures (Dziedzic, 1984; Hudson and Ghavami, 1984; Husain et al., 1986; Frankel, 1993).

Table III  
Percentage weight loss of total tocopherols of oil samples subjected to microwave heating

Oil subjected to MWH* / Heating time (minute)	2min	6min	10min	15min	18min
Sunflower oil	4.2	5.7	8.5	10.0	17.0
Soybean oil	8.3	16.6	24.1	33.3	37.5
Peanut	12.0	24.0	36.0	48.0	68.0
Admixture	5.3	10.66	16.0	26.66	33.33

\*MWH = Microwave heating.

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Recibido: Diciembre 2001  
Aceptado: Febrero 2003