MICROWAVE HEAT TREATMENT OF PEANUT, SESAME AND SOYBEAN SEEDS AND ITS INFLUENCE ON THEIR LIPID CONSTITUENTS

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ABSTRACT

Microwave heating of oil seeds (peanut, sesame and soybean) was conducted to study the influence of heating on their oil constituents. Each seed sample was microwave heated for certain heating intervals avoiding charring and any damage of its components. Acid value, oxidation parameters (represented by peroxide and para-anisidine value and the calculated total oxidation value) and conjugation of polyunsaturated fatty acids have been followed during the course of microwave heating. Fatty acids, as their methylesters, and tocopherols, as their trimethylsilyl (TMS) derivatives, were determined by GLC. Total tocopherols, isolated by preparative TLC from the unsaponifiable matter, were quantitatively determined. It was found that acid value, oxidation parameters and fatty acid conjugation showed considerable changes during microwave heating.

It was also found that unsaturated fatty acids were decreased as a result of the formation of the primary and secondary oxidation products. Meanwhile, individual tocopherols of sesame and peanut oils showed slight decrease during microwave heating, however marked decrease in individual tocopherols was observed in case of

soybean oil.

In conclusion 2min heating of seeds is more reasonable for obtaining high quality oils from seeds probably having some acceptable taste characteristics. Keywords: Peanut, Sesame, Soybean, Microwave heating, Acid value, Oxidation parameters, Fatty acid, Tocopherol.

INTRODUCTION

Microwave heating has been used for many processes: tempering, dehydration, blanching, cooking, pasteurization and sterilization (Decareau, 1985). Moreover short-time microwave heating of peanut yielded 95% reduction of aflatoxins without measurable changes in protein and lipid contents (Luter et al., 1982). It has been postulated that microwave thermal effects cause microbial destruction or enzyme inactivation (Esaka et al., 1987; Khall and Villota, 1988 and Kermasha et al., 1993). Some reports suggested that nutrient retention, such as vitamins in microwaved foods would be improved because cooking time is shortened (Institute of Food Technologists, 1987 and Gould and Golledge, 1989). However other studies suggested that, nutrient retention in microwave processing is not much greater than that in conventional cooking (Harris and Karmas, 1975 and Thompson, 1982). Yoshida et al., (1997) studied the composition and fatty acid distribution of phospholipids in soybean seeds at different moisture levels when roasted in a microwave oven. Whereas, Daglioglu et al., (2000) studied the effect of microwave and conventional baking on the oxidative stability and fatty acid composition of puff pastry. The effect of microwave heating on olive oil samples, in particular the fatty acid composition in partial

and total acylglycerol was studied by Cossignani *et al.*, (1998) In addition, Yoshida and Kajimoto, (1989) studied the effects of microwave heating on tocopherols of soybean in relation to chemical changes in the lipid. They found that microwave treatment of soybean for 6 min was optimal to prepare full-fat soy flour without a burnt odour.

The objective of this work was to follow chemical changes of the oils of microwave heated seeds of peanut, sesame and soybean. Therefore, the seeds were subjected to microwave heating at different time intervals under carefully controlled conditions. Acid value, oxidation parameters (represented by peroxide and p-anisidine values) and fatty acid conjugation were determined. Meanwhile fatty acids and tocopherols compositions were also periodically determined. The results may help to predict the optimum time necessary to keep the quality of the seeds.

MATERIALS AND METHODS

Materials:

Three seeds, namely, peanut, sesame, and soybean (season, 2001) were obtained from Oilseed Crop Department, Ministry of Agriculture, Giza, Egypt.

Methods:

A microwave oven (MW) (Samsung model 9245,MB 245) having frequency of 2,450 MHz, was used for heating the seeds. Preliminary experiment on determining the maximum exposure time of each seed to MW heating, without charring or burning was carried out. Then, seed samples were placed separately as a single layer in 5 petri- dishes (15 cm diameter) and heated in the MW oven for 2.0,3.0 and 4.0 min for peanuts; 2.0,4.0 and 6.0 min for sesame seeds and 1.0, 1.5, 2.0 and 2.5 min for soybean seeds. Each seed sample removed form the oven was kept immediately in a vacuum dissecator to cool down to room temperature. Then, the microwaved and untreated (control) seed samples, were separately ground and soaked in chloroform- methanol (2:1,v/v) with intermittent stirring. The solvent was decanted and the samples were soaked again in the same solvent to extract any residual lipids. The extracts of each sample were combined, filtered, dried over anhydrous sodium sulphate and evaporated under reduced pressure at 50°C in a rotary evaporator. The extracted oils were then kept in the refrigerator till analysis.

Determination of Acid Value and Some Oxidation Parameters:

Acid value (AV), Peroxide value (PV) and Para-anisidine value (p-AV) were determined according to A.O.C.S (1978).

The totox value or oxidation value (OV) was calculated as follows: OV= 2(PV) +p-AV.

Conjugated Fatty acids:

Shimadzu Spectrophotometer UV-240 was used in measuring the conjugated fatty acids (conjugated dienes and trienes) formed during MW

heating of the oil samples. Absorbancy at 234 and 268 nm were used for determining conjugated dienes and trienes, respectively according to A.O.C.S (1978).

GLC Analysis of Fatty Acids:

The oil samples were converted to their fatty acid methyl esters by transesterification using 5% hydrogen chloride in dry methanol (Chrisite, 1973). Hewelett Packard Model 6890 gas chromatograph was employed for the analysis of fatty acid methyl esters under the following conditions: column, INNO wax capillary column (polyethylene glycol), 30.0 m x 530 μm , film thickness 1.0 μm ; column was operated isothermally at280°C; injection temperature, 280°C; split ratio, 8:1; split flow, 120 ml/min; gas saver, 20 ml/min; and carrier gas, N2 with flow rate 15 ml/min; FID detector temperature 280°C; hydrogen flow rate, 30 ml/min and air flow rate, 300 ml/min. Peak areas were determined by electronic integrator and percentage composition of fatty acids was automatically calculated. Standard mixture of fatty acids methyl esters was also chromatographed under the same operating conditions.

GLC Analysis of Tocopherols:

The unsaponifiable matter of each oil sample was previously prepared according to the A.O.C.S (1978). The unsaponifiable matters were fractionated by preparative TLC (0.500mm thickness, 60 GF254) to isolate tocopherols from the other constituents. Two successive solvent mixtures, hexane/diethylether/acetic acid (80/20/1 and 70/30/1,v/v/v) were used as developing solvent systems. Standard tocopherol mixture was also applied on the plates to help identification of tocopherols. The tocopherols were detected on the chromatoplates as purple spots under ultra-violet light. The zones of tocopherols were scraped off and extracted twice with moistened diethylether, the solvent was removed under reduced pressure and the residue was accurately weighed.

The isolated tocopherols were converted into their trimethyl silyl derivatives (TMS) and injected into the gas chromatograph (Slover et al., 1967).

Gas chromatograph, Hewlett- Packard Model HP6890, was used for analysis of the TMS derivatives according to the following operating conditions: column (HP-1 Methyl siloxan) $30\text{mx}530\mu\text{m}$; film thickness 2.65 μ m; column temperature: 280°C; Flow rates, nitrogen ,30ml / min; hydrogen ,35ml/min and air ,300ml/min.

Expression of the Results:

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

RESULTS AND DISCUSSION

Classical Parameters Analysis:

Acid value, oxidation parameters and fatty acid conjugation of oils of the microwave (MW) heated seeds at different intervals are recorded in Tables (1-3).

Table (1) showed that the acid value (AV) of peanut increased slightly from 0.1 (control seed oil) to 0.4, 0.5 and 0.65 after 2,3 and 4 min heating respectively. Concerning the peroxide value (PV), it was found that, it increased from 0.3 for the control to 2.0, 3.2 and 3.5 for the heated oil samples at the corresponding exposure time intervals. Whereas, para-anisidine value (p-AV) showed a gradual increase at different exposure times and reached 3.0 at 4min of MW heating in comparison with 0.6 for the control. From the totox value or oxidation value (OV), it was found that the values are proportional to exposure time of heating and it reached 10.0 at 4 min of MW heating in comparison with 1.2 for the control. Meanwhile, conjugated dienes were detected at lower levels, and slightly increased during heating.

Table 1: Classical Parameters for Untreated and Microwave - Treated

			D!-!-!	Tatau	Conjugation %		
Sample	Acid Value	Peroxide value	P- anisidine value	Totox value	Diene 234 nm	Triene 268nm	
Control	0.1	0.3	0.6	1.2	0.017		
2 min	0.4	2.0	2.5	6.5	0.133		
3 min	0.5	3.2	2.6	9.0	0.136		
4 min	0.65	3.5	3.0	10.0	0.157		

The results cited in Table (2) showed that AV and PV of the extracted oil from heated sesame seeds exhibited gradual but slight increase as heating process was proceeding. Obviously, p-AV showed extremely higher values after 4 and 6 min heating in comparison with the control. Thus, OV increased from 0.8 (control) to 1.8, 3.4 and 4.3 at 2,4 and 6min heating. On the other side, it was found that conjugated dienes were kept nearly constant at the different heating intervals, but were slightly higher than that of the control.

Table 2: Classical Parameters for Untreated and Microwave -Treated Sesame Seeds.

	1 2 2 2		D	T-4	Conjugation %	
Sample	Acid Value	Peroxide value	P-anisidine value	Totox	Diene 234 nm	Triene 268nm
Control	0.2	0.3	0.2	0.8	0.32	
2 min	0.3	0.5	0.8	1.8	0.42	
4 min	0.5	0.7	2.0	3.4	0.43	
6 min	0.7	0.9	2.5	4.3	0.44	

Acid value, PV and p-AV of the extracted oils of heated soybean are recorded in Table (3). It was observed that the different parameters of the extracted oils from the heated seeds exhibited slight increases at 1.0 and 1.5 min, however marked increases were generally observed at 2.0 and 2.5 min heating. The OV reached higher levels at 2.0and2.5min heating, whereas slight increase in conjugated dienes was observed during exposure to MW heating.

Table 3: Classical Parameters for Untreated and Microwave-Treated

		ybcans.					
	Acid Peroxd		P-anisidine	Totox	Conjugation %		
Sample	Value	Value	value	value	Diene 234 nm	Triene 268 nm	
Control	0.5	0.2	0.3	0.7	0.286		
1 min	0.5	0.5	0.5	1.5	0.308		
1.5 min	0.7	1.0	1.0	3.0	0.334		
2.0 min	1.0	1.4	2.2	5.0	0.363		
2.5 min	1.2	2.3	3.3	7.9	0.367		

In general the classical parameters of oxidation, namely, PV, p-AV and fatty acid conjugation showed significant differences in the three oils. Development of acidity in oils of MW heated seeds may be due to different acid components produced either by breakdown due to oxidative processes or by hydrolysis of triglycerides (Yoshida et al., 1992 and Cossignani et al., 1998). As expected, PV as indication of the extent of primary oxidation and p-AV values representing secondary oxidation, were generally increased by MW heating in the treated samples (Cossignani et al., 1998 and Daglioglu et al., 2000). Detection of conjugated dienes in the oils during MW heating showed that oxidation had occurred (Yoon et al., 1985 and Yoshida et al., 1990).

Fatty Acid Composition:

Fatty acid profiles of MW heated samples at different time intervals are recorded in Tables 4-6.

Oleic acid in peanut oil of MW heated seeds decreased from 43.5% (control) to 42.9, 42.0 and 41.7 % at 2,3 and 4 min heating intervals respectively (Table 4). Also, linoleic acid gradually decreased by MW heating to 33.6, 32.5 and 31.4 % at the corresponding heating periods.

Table (4): Fatty Acid Composition of Oil of Microwave Heated Peanut as

Determined by GLC.

Sample	Myristic C14:0	Palmitic C16:0	Palmito- oleic C16:1	Stearic C18:0	Oleic C18:1		Linolenic C18:3		Other fatty acid
Control	0.9	11.1	0.3	3.3	43.5	34.0	0.3	6.6	-
2 min	0.1	11.5	0.3	3.7	42.9	33.6	0.2	5.2	2.5
3 min	-	12.0	0.3	4.0	42.0	32.5	0.1	5.1	4.0
4 min	45524	12.5	0.2	4.5	41.7	31.4	0.1	5.1	4.5

In Sesame oil (Table 5) a decrease in both unsaturated fatty acids (oleic and linoleic) was also observed, however the decrease in oleic acid was more marked than in case of peanut oil.

Table 5: Fatty Acid Composition of oil of Microwave Heated sesame as

	Deceniii	mod by o				
Sample	Palmitic C16:0	Stearic C18:0	Oleic C18:1	Linoleic C18:2	C20-C24	Other Fatty acid
Control	9.5	5.5	42.0	43.0		
2min	9.6	5.7	41.0	4.1-2	2.0	0.5
4 min	10.0	6.0	39.3	40.2	2.3	2.2
6 min	10.2	6.2	38.5	40.0	2.2	2.9

On the other side, soybean contains linolenic acid beside oleic and linoleic acids (Table 6). Both linoleic and linolenic acids were remarkably affected by MW heating and therefore marked decrease in their quantities was observed. However oleic acid increased as MW heating was progressing, this was due to an apparent increase in the percentage content (Cossignani, et al., 1998).

Table 6: Fatty Acid Composition of oil of Microwave Heated Soybean as Determined by GLC.

Sample	Palmitic C16:0	Stearic C18:0	Oleic C18:1	Linoleic C18:2	Linolenio C18:3
Control	10.7	2.6	22.4	56.0	8.3
1 min	11.4	3.0	24.3	54.3	6.8
1.5 min	12.5	3.4	25.8	52.3	6.0
2.0 min	12.8	3.5	27.0	51.5	5.2
2.5 min	13.2	3.5	27.1	51.2	5.0

It was observed that oleic and linoleic acids of peanut and sesame oils generally, decreased during MW heating due to the thermal oxidative decomposition of hydroperoxides formed from these acids (Nawar 1985 and Cossignani et al., 1998). Whereas, only the polyunsaturated fatty acids, namely, linoleic and linolenic acids in soybean oil (from seeds heated for short time) decreased by MW heating. This may be due to the possibility that polyunsaturated hydroperoxides are formed rapidly at the shorter MW heating periods than that of oleic acid.

Tocopherois:

The total tocopherols of the oil samples were isolated quantitatively from the unsaponfiable matter by preparative TLC and the tocopherol profiles were determined by GLC as their TMS derivatives (Tables 7-9).

The total tocopherol contents of oils of the heated seeds generally showed a decrease in quantity as exposure time increased during MW heating.

With respect to the tocopherol profiles of peanut oil (Table 7) showed that, alpha- and gamma- tocopherols were predominating and they were affected by MW heating. Thus alpha- and gamma- tocopherols showed a gradual decrease during MW heating, whereas beta- and delta – tocopherols showed no significant variation when MW heating was prolonged. The percentage loss of total tocopherol contents reached 10.7, 17.8 and 25.6 at 2, 3 and 4 min MW heating respectively.

Table 7: Tocopherol Profiles of oil of Microwave Heated Peanut as

Sample	Total tocopherol	Tocop				
	mg/ 100g	Alpha	Beta	Gamma	Delta	Loss%
Control	17.08	9.1	0.08	7.4	0.5	
2 min	15.25	8.3	0.05	6.5	0.4	10.7
3 min	14.03	7.6	0.03	6.1	0.3	17.8
4 min	12.7	6.8	0.00	5.6	0.3	25.6

* Total tocopherol loss % = Original total Tocopherol - Found tocopherol content X100

Original total tocopherol

In sesame oil, gamma- tocopherol is predominating and it was slightly affected by MW heating as the other three-tocopherol components (Table 8). Concerning the percentage loss of total tocopherols, it is comparatively lower reaching 4.4,8.8 and 10.8 at 2.0,4.0 and 6.0 min MW heating respectively.

Table 8: Tocopherol Profiles of oil of Microwave Heated Sesame as Determined as TMS by GLC.

	Tocoph				
mg/ 100g	Alpha	Beta	Gamma		Loss%
45	4.2	1.4	34.8		
43	3.8	1.3			4.4
41					8.8
40.1					10.8
	mg/ 100g 45 43 41	mg/ 100g Alpha 45 4.2 43 3.8 41 3.4	mg/ 100g Alpha Beta 45 4.2 1.4 43 3.8 1.3 41 3.4 1.3	mg/ 100g Alpha Beta Gamma 45 4.2 1.4 34.8 43 3.8 1.3 33.9 41 3.4 1.3 32.7	mg/ 100g Alpha Beta Gamma Delta 45 4.2 1.4 34.8 4.6 43 3.8 1.3 33.9 4.0 41 3.4 1.3 32.7 3.6

Soybean oil of the MW heated seeds showed that, gamma – and delta- tocopherols are the predominating constituents and were greatly affected by MW heating (Table 9). These two tocopherol components were subjected to marked decrease by increasing exposure time of MW heating, however the other tocopherol components, namely, alpha-and beta – tocopherols are less affected by MW heating. With reference to the percentage loss of total tocopherols, it was noticed that the percentage loss reached 2.5,10.0,12.2 and 20 at 1,1.5, 2 and 2.5min MW heating respectively.

Table 9: Tocopherol Profiles of oil of Microwave Heated Soybean as
Determined as TMS by GLC

Sample	Total tocopherol	Toco	T			
	mg/ 100g	Alpha	Beta	Gamma	Delta	Loss%
Control	130	6.5	4.0	87.9	31.6	
1 min	126.7	6.2	3.8	86.2	30.5	2.5
1.5 min	117	5.5	2.8	80.3	28.4	
2.0 min	114.1	5.3	2.8	78.0		10.0
2.5 min	104	4.6	1.9		28.0	12.2
	.07	7.0	1.9	72.5	25.0	20.0

Generally, it can be noticed that gamma-tocopherol in sesame oil was less affected by MW heating than that in peanut and soybean oils; this may be due to the presence of other antioxidants (non- tocopherol type) in sesame oil that may protect gamma- tocopherol during MW heating (Bailey, 1979).

It is known that tocopherols are commonly used as antioxidants in vegetable oils, but gamma-and delta- tocopherols are more powerful antioxidants than alpha-tocopherol (Khafizov et al., 1975 and Ikeda and Fukuzumi, 1977)

It was reported by Yoshida and Kajimoto (1989) that, loss of tocopherols may be attributed to their thermal degradation when subjected to MW heating. Also they are unstable in the presence of oxygen, ultraviolet light and highly unsaturated fats (Nelis *et al.*, 1985)

It can generally be concluded that 2 min MW heating is more advantageous for seeds heating and therefore the following criteria, based on this concept, can be derived out:

- 1- Two min MW heating of the seeds, showed that the primary oxidation products of the oil, as determined by PV, are comparatively reasonable at this shorter heating time interval reaching 0.5, 1.4 and 2.0 meq. O₂/kg for sesame, soybean, and peanut respectively. The corresponding oxidation values, representing both primary and secondary oxidation products, were 1.8, 5.0 and 6.5.
- 2- Following the percentage loss of total tocopherols in oils of the three seeds at 2 min heating treatment, reasonable percentage loss were obtained in peanut (10.7%) and sesame (4.4%). However, comparatively higher loss was only recorded for soybean. The loss of tocopherols is mainly attributed to peroxidation of the unsaturated fatty acids during MW heating (Yoshida and Kajimoto, 1989and Yoshida et al., 1992) and accordingly, soybean oil showed higher tocopherol percentage loss due to its higher polyunsaturates content.
- 3- At 2min heating there are different changes in the mono-and polyunsaturated fatty acid components of the oils of heated seeds. Linolenic acid in soybean oil with the other polyunsaturated acid (linoleic) are markedly decreased by MW heating. However, oleic acid in heated soybean oil apparently increased in the oil profile. On the other side, oleic and linoleic acid components of sesame and peanut oils exhibited a decrease in quantity when heated for 2 min. It seems that polyunsaturated fatty acids respond more rapidly to oxidation than oleic acid especially at shorter time of MW heating (Cossignani et al., 1998).

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المعاملة الحرارية بالميكرويف لبذور الفول السوداني والسمسم والصويا وتأثيرها على مكونات الليبيدات لهذة البذور ·

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أجريت دراسة تأثير معاملة الميكرويف الحرارية على بذور الفول السوداني والسمسم وفول الصويا وتأثيرها على مكوناتها الليبيدية بتسخين كل بذرة على حسب نوعها في فرن الميكرويف تجنبا للتفحم الجزئي إضافة إلى عدم تكسير مكوناتها ، وأمكن تتبع رقبم الحموضة وكذلك متابعة حدوث الاكسدة (رقم البيروكسيد ورقم الانيزيدين وكذلك حساب الاكسدة الكلية) وخلاك متابعة حدوث الاكسدة (رقم البيروكسيد ورقم الانيزيدين وكذلك حساب الاهنية ، وأيضا تسم إضافة إلى تقدير الروابط اللزوجية المتبادلة (Conjugation) في الأحماض الدهنية ، وأيضا تسم تقيير التغير أن التعليم التعليم التعليم التعليم التعليم التعليم التعليم الكليم التعليم المسخين بواسطة في المسلم الدهنية عبد المشبعة تقل اثناء تسخين الدهنيم الدهنية غير المشبعة تقل اثناء تسخين الزيوت في فرن الميكرويف كنتيجة لتكوين نواتج الأكسدة الأولية والثانوية ، أما بالنسبة للتغير في التوكوفيرولات فقد وجد أنها تقل بنسبة بسيطة في السمسم وفول السوداني ولكنها تقل بنسبة ملحوظة التعليم المناسلة التعليم المناسلة التعليم ال

في فول الصويا . وقد انتهت الدراسة إلي أن التسخين بالميكرويف لمدة دقيقتين هي فترة مناسبة للحفاظ على مكونات الزيت إضافة إلى احتمالية الحصول على بذور لها طعم و مذاق مستساغ.