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The influence of microwave roasting on bioactive components and chemical parameters of cold pressed fig seed oil

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SUMMARY: The effect of microwave roasting process on the compositional parameters and bioactive contents of fig seed oil were investigated. Fig seeds were ground and roasted in a microwave oven at 350, 460 and 600 Watt for 5 and 10 minutes and the roasted seeds were processed to obtain oil. The results showed that peroxide, K_{232} and K_{270} values were adversely affected by roasting. Fig seed oil was a prosperous source of γ-tocopherol and significant losses were observed due to microwave pre-treatment. The major fatty acids in fig seed oil were linolenic, linoleic and oleic acids; whereas the major triacylglycerols were LnLO, LnLnL, LnLnLn and LnLnO, according to fatty acid profile. The most abundant sterol in the fig seed oil samples was β-sitosterol with 3235.90 to 3625.62 mg/kg, followed by Δ5- and Δ7-avenasterols. The principal component analysis and agglomerative hierarchial clustering served to differentiate between intense and mild microwave-treated oils as well as the unroasted samples.

KEYWORDS: *Fig seed oil; Microwave; Sterols; Tocopherols; Triacylglycerols*

RESUMEN: ***Influencia del tostado por microondas en los componentes bioactivos y los parámetros químicos del aceite de semilla de higo prensado en frío.*** Se investigó el efecto del proceso de tostado por microondas sobre los parámetros de composición y contenido bioactivo del aceite de semilla de higo. Las semillas de higo se molieron y tostaron en un horno de microondas a 350, 460 y 600 vatios durante 5 y 10 minutos a continuación se obtuvo el aceite. Los resultados han demostrado que los valores de peróxido, K_{232} y K_{270} se vieron afectados negativamente por el tostado. El aceite de semilla de higo es una buena fuente de γ-tocoferol y se observaron pérdidas significativas mediante el pretratamiento con microondas. Los principales ácidos grasos del aceite de semilla de higo fueron los ácidos linolénico, linoleico y oleico; mientras que los principales triacilgliceroles fueron LnLO, LnLnL, LnLnLn y LnLnO que ratificaron el perfil de ácidos grasos. El esterol más abundante de las muestras de aceite de semilla de higo fue el β-sitosterol que varió de 3235,90 a 3625,62 mg/kg, acompañado de Δ5-avenasterol y Δ7-avenasterol. El análisis de componentes principales y la agrupación jerárquica aglomerativa permitieron la diferenciación de aceites tratados con microondas intensos y suaves, así como las muestras sin tostar.

PALABRAS CLAVE: *Aceite de semilla de higo; Esteroles; Microondas; Tocoferoles; Triacilgliceroles.*

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1. INTRODUCTION

Ficus carica L., generally known as fig, is one of the oldest cultivated fruit trees and is a member of the Moraceae botanical family. It is grown in warm and dry climates, and its natural areas are the Mediterranean coast and western Asia. Major producers are Turkey, Egypt, Morocco, Spain, Greece, California, Italy, Brasil, Algeria and Iran (Nakilcioğlu-Taş, 2018). The tree is mainly cultivated for its fig fruit, which is consumed either as fresh, dried, canned or as jam, juice and puree. Fresh and dried fruits are principal sources of minerals, vitamins, dietary fiber, amino acids, organic acids and sugar (Solomon *et al*., 2006). Fig fruits have also been denoted to be fine sources of phenolics that contribute to the nutritional quality and antioxidant capacity of the fruit (Veberic *et al*., 2008).

The fig holds a vast number of small seeds that vary greatly in amount depending on the volume and the maturity of the fruit. The seeds are located within the interior part of the fruit as a mass with a jelly-like flesh (Badgujar *et al*., 2014). A considerable amount of waste containing large amounts of seeds is generated when figs are used for the production of juice and puree. The obtained by product is commonly utilized for recovering fig seed oil. The seeds contain notable amounts of oil, which may reach up to 30% in dried figs (İçyer *et al*., 2017).

Fig seed oil is distinguished by its high linolenic acid content, while oleic, linoleic, stearic and palmitic acids were also described to be present in the oil (Joseph and Raj, 2011). The replacement of saturated fatty acids in the human diet with polyunsaturated ones was recommended by WHO/FAO (WHO, 2008). Fig seed oil can be incorporated into common dietary items as a good source of plant-based polyunsaturated fatty acid. Apart from the fatty acid profile, fig seed oil is also a valuable resource of γ-tocopherol and other bioactive compounds, such as α-tocopherol and phytosterols (Güven *et al*., 2019).

Microwave pre-treatment is a desirable technique used to produce oil from seeds. The technique has several advantages such as short processing time, low energy consumption, higher yield and better retention of nutraceuticals in the resultant oil (Azadmard-Damirchi *et al.*, 2011) when compared to conventional methods. A number of works have been performed to observe the influence of microwave pre-treatment before the extraction of oil from pumpkin (Ali *et al.*, 2017), sunflower (Goszkiewicz *et al*., 2020), chia seed (Ozcan *et al*., 2019), pomegranate (Đurđević *et al*., 2017), black cumin (Bakhshabadi *et al*., 2017), Camellia (Ye *et al*., 2021) and other seeds. Of course, each oil obtained from various oilseeds has its own characteristic triacylglycerol profile and all oils respond differently to thermolytic and oxidative reactions that take place during heating processes due to their different chemical composition. There is no information available on the impact of microwave roasting prior to oil extraction on the quality, stability or chemical composition of fig seed oil. Some reports have been published on the chemical profile of fig seed oil (Duman and Yazıcı, 2018; Güven *et al*., 2019), although the information regarding the impact of microwave pre-treatment on the oil composition is lacking and needs to be investigated. Because no research on oil extraction from fig seeds with the use of microwave application wase detected by the authors, The effective parameters including microwave power and time were determined in the present work. Hence, the aim of the present study was to investigate the changes in composition (fatty acid and triacylglycerol profiles) and bioactive compounds (sterols and tocopherols) in fig seed oil due to microwave roasting before mechanical extraction. Since the intensity of heat affects the composition of oil, various power settings (350, 460 and 600 W) and radiation times (5 and 10 min) were taken into consideration. The results of this study may be used to evaluate the feasibility of using microwave pre-treatment as an improvement method for the manufacture of cold-pressed fig seed oil.

2. MATERIALS AND METHODS

2.1. Materials

Fig seeds were kindly supplied by Egesia (Aydın, Turkey). Potassium hydroxide, β-sitosterol, pyridine, sodium hydroxide, ethyl alcohol, and chloroform were purchased from Merck (Darmstadt, Germany). Diethyl ether, methanol, isopropyl alcohol, isooctane, acetonitrile, methyl orange, *n*-hexane, hydrochloric acid, acetone, 5α-cholestan-3β-ol, silica gel, α-, β-, δ- and γ-tocopherol standards were purchased from Sigma-Aldrich (St- Louis, ABD). 37 fatty-acid methyl ester mix, N,O-Bis (trimethylsilyl) and trifluoroacetamide with trimethylchlorosilane were purchased from Supelco (Bellefonte, USA).

2.2. Methods

2.2.1. Microwave pre-treatment and pressing of the fig seeds

Fig seeds were first ground in a grinder (Sinbo, Turkey) and then roasted at 350, 460 and 600 Watt for 5 and 10 minutes in a microwave oven (Arçelik, Turkey). The seeds were cooled to room temperature after roasting. The pressing process was performed with a laboratory scale (single head, 2 hp, 12 kg seed/h capacity, 1.5 kW power) screw press (Koçmaksan KMS 10, Turkey). After pressing, centrifugation was applied to obtain a clearer oil. The fig seed oil samples were kept at 4 ºC in nitrogen atmosphere until analyses.

2.2.2. Fat content

The oil content in the seeds was measured by soxhlet extraction in accordance with AOCS Official Methods Am 2-93. *n*-hexane was the extraction solvent.

2.2.3. Peroxide value and spectrophotometric extinction coefficients at 232 and 270 nm (K_{232} and K_{270})

Peroxide value, spectrophotometric extinction coefficients at 232 and 270 nm were measured by AOCS Official Methods Cd 8-53 and Ch 5-91 (AOCS, 2003), respectively.

2.2.4. Fatty acid profile

The percentages of fatty acids were determined by preparing their corresponding methyl esters according to the method established by International Union of Pure and Applied Chemistry (IUPAC, 1987). The esters were analyzed with a gas chromatography instrument (GC 2010, Shimadzu, Japan) fitted with a flame ionization detector. The separation of the peaks was achieved with a DB-23 column (60 m length x 0.25 mm internal diameter and 0.25 µm film thickness) (J&W Scientific). The column, injector and detector temperatures were 195, 230 and 240 °C, respectively. Nitrogen was the carrier gas (1.0 ml/min).

2.2.5. Triacylglycerol profile

The triacylglycerol profile of the fig seed oils was determined using AOCS Official Method Ce 5b-89 (AOCS, 2003). The oil sample (0.5 g) was dissolved in acetone and analyzed using HPLC (Shimadzu, Japan) fitted with a differential refractometer detector (RID). Chromatographic elution was achieved using an ACE 5 C18 column (4.6 mm ×250 mm, 5µm particle size, ACE, Scotland). The mobile phase consisted of acetone/acetonitrile (1:1) at a flow rate of 1.5 ml/min. The column temperature was 30 °C and volume of injection was 10 µl. Triacylglycerol peaks were defined by matching with those in the literature (Holčapek *et al*., 2005).

2.2.6. Sterol profile

The sterol profile of oil samples was determined in accordance with AOCS Ch 6–91 (2003). The sterols were first silanized and then quantitatively detected with a gas chromatography instrument (GC 2010, Shimadzu, Japan) fitted with a flame ionization detector. Chromatographic seperation was performed using a HP-5 column (Chrom Tech., USA) with 30 m length, 0.25 µm film thickness and 0.25 mm internal diameter. Nitrogen was the carrier gas at a flow rate of 0.8 mL/min. The injector, detector and column temperatures were 280, 290 and 260 °C, respectively.

2.2.7. Tocopherol profile

The tocopherol composition of the fig seed oil was detected using an HPLC instrument (Shimadzu, Kyoto, Japan) equipped with an InertSustain NH_2 column. The column was 250 mm in length, with 4.6 mm internal diameter and 5 µm particle size (GL Sciences, Japan). The mobile phase was composed of *n*-hexane:isopropyl alcohol (99.5:0.5) in an isocratic system. The injection volume was 20 µl and the flow rate was 1.2 ml/min. Tocopherol homologues were determined at 290 nm. α-, and γ- tocopherol standards were used to prepare the standard curves.

2.2.8. Statistical analysis

The SPSS software, version 15.0 (SPSS Inc., Chicago, USA) was used for statistical evaluation. Differences were calculated by one-way ANOVA procedure and Duncan's multiple range test was used to compare the significance of differences at $p < 0.05$. Data were also analyzed with multivariate tests (PCA and AHC) using XLSTAT, version 2020 (Addinsoft, USA).

3. RESULTS AND DISCUSSION

The oil content in the fig seeds was 22.18% on wet weight basis, similar to the findings of former works (Nakilcioğlu-Taş, 2019).

The changes in lipid oxidation parameters (peroxide value and specific ultraviolet absorbances) as a result of the microwave process are presented in Table 1. Peroxide value, indicator of primary oxidation products, increased by ascending microwave power. Significant differences were determined in peroxide values of increasing radiation times at 460 and 600 W power settings. The K_{232} value increased significantly due to microwave roasting, although no significant differences were determined among the three power settings and two process times. The K_{270} value, which shows the secondary oxidation products (aldehydes and ketones), was detected to increase with ascending power settings.

Tocopherols are antioxidants which are naturally present in edible oils and play important roles as Vitamin E for human health. The influence of microwave power settings on the tocopherol composition of the fig seed oil is given in Table 1. Fig seed oils contained α- and γ-tocopherols varying in 101.62-114.07 mg/kg and 3888.22-4132.09 mg/kg, respectively. Güven *et al*. (2019) reported 4267 mg/kg of γ-tocopherol; and Baygeldi *et al*. (2021) described 314.61±51.53 mg/100 g of γ-tocopherol, 7.40±0.26 mg/100 g of d-tocopherol and 3.71±0.62 mg/100 g of α-tocopherol for fig seed oil. Microwave pre-treatment caused significant losses in both tocopherols, possibly due to thermo-induced oxidation and degradation of tocopherols (Ji *et al.*, 2019), similar to the findings reported for poppy seeds (Ghafoor *et al*., 2019) and pumpkin seeds (Yoshida *et al*., 2006). In the current work, the highest tocopherol loss was determined at the highest microwave power setting (600 W) at the longer period of roasting (10 min). Moreover, roasting time was found to be statistically important on both α- and γ- tocopherol content at 600 W power setting.

The changes in fatty acid composition due to microwave roasting are shown in Table 2. The major fatty acid was linolenic acid, ranging from 45.06-46.01%, slightly higher than the findings of previous works (İçyer *et al*., 2017; Duman and Yazıcı, 2018; Baygeldi *et al.*, 2021). Extended roasting time seemed to decrease the linolenic acid content in the samples. Linoleic acid was the other leading fatty acid, and varied from 27.99-28.75% and found to be the lowest at 600 W and 10 min of microwave heating. Oleic acid was the predominant monounsaturated fatty acid (16.63-16.98%) and it slightly decreased due to the microwave process. Modest changes were observed in palmitic, stearic and arachidic acids; whereas myristic, palmitoleic, heptadecanoic, heptadecenoic, gadoleic acids were determined to remain unchanged. C 16:1 and C 18:0 were high and negatively correlated (r = -0.93). Previous works have reported either decreases in unsaturated fatty acids due to degradation (Fathi-Achachlouei *et al*., 2019; Suri *et al*., 2020), or statistically constancy in fatty acid profile (Güneser and Yılmaz, 2017) for different types of oils.

Table 1. Peroxide value, UV spectrophotometric indices and tocopherol contents of oils obtained from fig seeds roasted at different microwave setting and times

Microwave power (W)	Time (min)	Peroxide value (meqO_2/kg oil)	K_{232}	K_{270}	α-tocopherol (mg/kg)	γ- tocopherol (mg/kg)
Control		1.06±0.00^{A}	1.86±0.31^{A}	0.36±0.03^{A}	114.07±0.89^{A}	4057.08±102.74^{BC}
350	5	1.06±0.00^{A}	2.27±0.14^{B}	0.41±0.08^{AB}	108.46±2.91^{B}	4079.05±157.60^{BC}
	10	1.06±0.00^{A}	2.30±0.16^{B}	0.41±0.01^{AB}	108.99±3.82^{B}	4132.09±57.57^{C}
460	5	1.41±0.50^{B}	2.60±0.18^{B}	0.49±0.09^{BC}	107.14±1.96^{B}	3955.39±141.13^{AB}
	10	2.13±0.00^{C}	2.46±0.15^{B}	0.47±0.03^{AB}	108.09±1.61^{B}	4125.55±71.23^{BC}
600	5	1.42±0.00^{B}	2.41±0.15^{B}	0.58±0.12^{C}	108.86±1.51^{B}	4070.05±92.80^{BC}
	10	3.02±0.25^{D}	2.58±0.28^{B}	0.51±0.03^{BC}	101.62±2.29^{C}	3888.21±87.35^{A}

The results are presented as mean±standard deviation (n=4). Means in the same column with different letters are significantly different according to Duncan's multiple range test ($p < 0.05$).

Table 2. Fatty acid composition of oils obtained from fig seeds roasted at different microwave settings and times (%)

Fatty acids	Control	350 W		460 W		600 W	
		5 min	10 min	5 min	10 min	5 min	10 min
C 14:0	0.01±0.00^{A}	0.01±0.01^{A}	0.01±0.00^{A}	0.01±0.00^{A}	0.01±0.00^{A}	0.01±0.00^{A}	0.01±0.00^{A}
C 16:0	6.46±0.04^{A}	6.53±0.16^{AB}	6.46±0.09^{A}	6.49±0.13^{A}	6.44±0.07^{A}	6.50±0.05^{A}	6.66±0.09^{B}
C 16:1	0.33±0.12^{A}	0.38±0.10^{A}	0.47±0.25^{A}	0.33±0.03^{A}	0.48±0.21^{A}	0.43±0.06^{A}	0.35±0.12^{A}
C 17:0	0.03±0.00^{A}	0.03±0.01^{A}	0.03±0.01^{A}	0.03±0.01^{A}	0.03±0.01^{A}	0.03±0.01^{A}	0.03±0.00^{A}
C 17:1	0.02±0.01^{A}	0.02±0.00^{A}	0.02±0.00^{A}	0.02±0.01^{A}	0.02±0.00^{A}	0.02±0.00^{A}	0.02±0.01^{A}
C 18:0	2.23±0.10^{B}	2.19±0.16^{AB}	2.13±0.09^{AB}	2.24±0.10^{B}	2.04±0.17^{A}	2.15±0.03^{AB}	2.26±0.08^{B}
C 18:1	16.98±0.25^{B}	16.71±0.11^{AB}	16.65±0.15^{A}	16.86±0.24^{AB}	16.63±0.13^{A}	16.98±0.26^{B}	16.89±0.03^{AB}
C 18:2	28.53±0.18^{BCD}	28.28±0.19^{ABC}	28.10±0.49^{AB}	28.75±0.06^{D}	28.21±0.45^{ABC}	28.59±0.09^{CD}	27.99±0.07^{A}
C 18:3	45.22±0.49^{A}	45.66±0.37^{AB}	45.95±0.52^{B}	45.06±0.14^{A}	46.01±0.64^{B}	45.10±0.36^{A}	45.60±0.15^{AB}
C 20:0	0.07±0.02^{AB}	0.05±0.02^{AB}	0.04±0.02^{A}	0.08±0.01^{A}	0.04±0.04^{A}	0.05±0.02^{AB}	0.05±0.01^{AB}
C 20:1	0.13±0.03^{A}	0.13±0.01^{A}	0.14±0.05^{A}	0.14±0.02^{A}	0.11±0.03^{A}	0.14±0.01^{A}	0.12±0.01^{A}

The results are presented as mean±standard deviation (n=4). Means in the same line with different letters are significantly different according to Duncan's multiple range test ($p < 0.05$).

Triacylglycerols are the major components and represent 95-98% of edible oils. The determination of the triacylglycerol composition has critical importance to understanding the characteristics of oils. The change in triacylglycerol composition in fig seed oils by microwave application is given in Table 3. The major triglycerides identified were LnLO (oleolinoleolinolenin), LnLnL (linoleodilinolenin), LnLnLn (trilinolenin) and LnLnO (oleodilinolenin). In addition, LnLL (dilinoleolinolenin), LnLP (palmitolinoleolinolenin), SLLn (stearolinoleolinolenin), LLO (oleodilinolein), LnLnP (palmitodilinolenin), LLP (palmitodilinolein), LOP (palmitooleolinolein), LOO (dioleolinolein), LnOO (dioleolinolenin), LLL (trilinolein), LnLnS (stearodilinolenin), SLO (stearooleolinolein), OOO (triolein) and SOLn (stearooleolinolenin) were determined in descending order. The triacylglycerol composition of fig seed oils was in good agreement with their fatty acid profile. The major triglyceride, LnLO, varied from 13.00-13.54% and the increase in process time caused a slight decrease in the LnLO ratio at 460 and 600 W power settings. The other three crucial triacylglycerols, namely LnLnL, LnLnLn and LnLnO, ranged from 13.07-13.29%, 10.82-11.19% and 10.32-10.74%, respectively. Various results have been reported for the triacylglycerol composition of different matrices in response to microwave roasting, prior to oil extraction (Yoshida *et al*., 2006, Ali *et al*., 2017). In the current study; LOP, LLO and LnLL were determined not to be affected significantly by microwave pre-treatment. Although remarkable discrepancies were determined between the triacylglycerol percentages of unroasted and microwave roasted fig seed oils, different microwave power settings and radiation times did not have a clear influence on the rates of the remaining triacylglycerol. LnLnLn was determined to be highly correlated with C18:3 ($r = 0.947$).

Phytosterols are valuable components not only for their ability to lower serum cholesterol levels but also for their anti-ulcerative, anti-inflammatory, anti-bacterial and antitumour properties in humans (Moreau, 2003). The changes in the sterol distribution of fig seed oils by microwave roasting are shown in Table 4. The major sterols were β-sitosterol, Δ7-avenasterol and Δ5-avenasterol. In addition, 24-methylene-cholesterol, campestanol, campesterol, Δ7-campesterol, stigmasterol, sitostanol, clerosterol, Δ7-stigmastenol, Δ5-24-stigmastadienol were detected in small amounts. The total sterol contents in oil samples varied between 4859.96-5281.90 mg/kg and the microwave pre-treatment was found to be statistically insignificant on total sterols. Güven *et al*. (2019) reported 6516.20 mg/kg of total sterols for fig seed oils. A number of works have been published indicating the enrichment in phytosterol content in the oil due to microwave roasting (Azadmard-Damirchi *et al*., 2010), however, there are also reports showing a reduction in

Table 3. Triacylglycerol profile of oils obtained from fig seeds roasted at different microwave settings and times (%)

Triacylglycerols	Control	350 W		460 W		600 W	
		5 min	10 min	5 min	10 min	5 min	10 min
LnLnLn	10.99±0.16^{AB}	11.00±0.16^{ABC}	11.17±0.09^{BC}	10.82±0.06^{A}	11.19±0.17^{A}	10.82±0.03^{A}	11.03±0.11^{BC}
LnLnL	13.07±0.13^{A}	13.13±0.08^{A}	13.29±0.04^{B}	13.10±0.07^{A}	13.22±0.15^{AB}	13.11±0.10^{A}	13.07±0.07^{A}
LnLL	8.13±0.32^{A}	8.07±0.33^{A}	8.06±0.43^{A}	8.27±0.19^{A}	8.28±0.26^{A}	7.96±0.20^{A}	8.36±0.18^{A}
LnLnO	10.61±0.45^{A}	10.74±0.47^{A}	10.59±0.23^{A}	10.69±0.23^{A}	10.32±0.34^{A}	10.35±0.19^{A}	10.36±0.05^{A}
LnLnP	5.36±0.13^{BC}	5.31±0.07^{ABC}	5.41±0.05^{C}	5.36±0.05^{BC}	5.23±0.11^{AB}	5.19±0.10^{A}	5.40±0.02^{C}
LLL	2.23±0.38^{BC}	1.84±0.06^{A}	1.95±0.32^{AB}	1.98±0.17^{AB}	2.40±0.06^{C}	2.09±0.25^{ABC}	2.37±0.13^{C}
LnLO	13.16±0.40^{AB}	13.54±0.13^{B}	13.41±0.29^{B}	13.46±0.18^{B}	13.00±0.19^{A}	13.54±0.21^{B}	13.01±0.13^{A}
LnLP	7.59±0.63^{AB}	8.05±0.17^{B}	7.84±0.61^{B}	7.68±0.42^{AB}	7.38±0.27^{AB}	7.63±0.53^{AB}	6.99±0.23^{A}
LnLnS	1.64±0.66^{AB}	1.24±0.17^{A}	1.48±0.42^{A}	1.62±0.42^{AB}	1.90±0.28^{AB}	1.76±0.55^{AB}	2.23±0.16^{B}
LLO	5.85±0.09^{A}	5.87±0.14^{A}	5.86±0.14^{A}	6.00±0.19^{A}	5.98±0.18^{A}	6.02±0.07^{A}	5.86±0.08^{A}
LnOO	2.93±0.23^{BC}	2.54±0.08^{A}	2.64±0.23^{A}	2.71±0.11^{AB}	2.77±0.10^{ABC}	2.65±0.28^{A}	3.05±0.09^{C}
LLP	3.28±0.09^{A}	3.49±0.16^{AB}	3.43±0.13^{AB}	3.44±0.12^{AB}	3.47±0.30^{AB}	3.58±0.15^{B}	3.35±0.16^{AB}
SLLn	6.21±0.24^{AB}	6.35±0.04^{B}	6.22±0.25^{AB}	6.20±0.19^{AB}	6.04±0.35^{AB}	6.20±0.10^{AB}	5.93±0.09^{A}
LOO	2.93±0.11^{AB}	2.95±0.03^{AB}	2.89±0.08^{A}	2.96±0.01^{AB}	2.93±0.05^{AB}	3.04±0.12^{B}	2.89±0.06^{A}
LOP	3.28±0.16^{A}	3.29±0.09^{A}	3.21±0.11^{A}	3.19±0.09^{A}	3.21±0.12^{A}	3.31±0.17^{A}	3.29±0.09^{A}
SOLn	0.79±0.04^{A}	0.69±0.05^{AB}	0.61±0.07^{B}	0.67±0.12^{AB}	0.69±0.07^{AB}	0.75±0.10^{A}	0.74±0.09^{A}
OOO	0.66±0.02^{AB}	0.67±0.01^{AB}	0.68±0.02^{AB}	0.66±0.04^{A}	0.69±0.01^{AB}	0.69±0.04^{AB}	0.70±0.03^{B}
SLO	1.28±0.11^{AB}	1.22±0.05^{A}	1.27±0.07^{AB}	1.20±0.07^{A}	1.29±0.02^{AB}	1.31±0.05^{AB}	1.38±0.08^{B}

The results are presented as mean±standard deviation (n=4). Means in the same line with different letters are significantly different according to Duncan's multiple range test ($p < 0.05$).

Table 4. Sterol content of oils obtained from fig seeds roasted at different microwave settings and times (mg/kg)

Sterols	Control	350 W		460 W		600 W	
		5 min	10 min	5 min	10 min	5 min	10 min
24-methylene cholesterol	2.93±0.04^{A}	2.73±0.28^{A}	2.81±0.07^{A}	2.91±0.63^{A}	2.88±0.49^{A}	2.85±0.41^{A}	2.92±0.71^{A}
Campesterol	162.46±20.97^{A}	156.25±4.22^{A}	151.02±12.57^{A}	144.04±5.31^{A}	160.54±14.06^{A}	147.96±12.50^{A}	144.61±7.38^{A}
Campestanol	0.84±0.31^{A}	0.78±0.25^{A}	0.87±0.17^{A}	1.00±0.08^{A}	0.98±0.09^{A}	1.09±0.22^{A}	1.21±0.39^{A}
Stigmasterol	127.28±18.29^{B}	121.31±4.98^{AB}	117.53±10.77^{AB}	109.90±4.82^{A}	122.52±10.23^{AB}	113.03±8.15^{AB}	108.72±6.37^{A}
Δ-7-campesterol	5.01±2.23^{A}	3.71±0.61^{AB}	3.66±0.11^{AB}	4.07±0.41^{AB}	3.65±0.18^{AB}	3.47±0.70^{A}	4.25±0.62^{AB}
Clerosterol	20.34±2.04^{A}	22.36±1.94^{A}	21.03±3.46^{A}	24.42±0.71^{A}	20.44±0.92^{A}	20.89±3.95^{A}	20.98±5.50^{A}
β-sitosterol	3538.03±290.18^{A}	3522.73±95.20^{A}	3395.31±320.98^{A}	3235.90±98.36^{A}	3625.62±301.81^{A}	3300.68±264.15^{A}	3250.98±153.31^{A}
Sitostanol	7.87±2.21^{A}	10.58±4.11^{A}	8.66±0.72^{A}	15.09±9.66^{A}	11.49±4.18^{A}	11.19±2.08^{A}	12.48±3.15^{A}
Δ-5-avenasterol	1041.41±52.49^{A}	1053.03±18.89^{A}	1007.04±86.23^{A}	969.62±39.14^{A}	1078.90±87.42^{A}	981.80±68.97^{A}	999.73±55.94^{A}
Δ-5,24 stigmastadienol	87.44±8.78^{A}	85.31±1.06^{A}	84.91±4.98^{A}	80.44±2.40^{A}	90.69±11.76^{A}	85.34±7.70^{A}	83.52±4.76^{A}
Δ-7-stigmastenol	68.46±27.44^{A}	52.81±1.96^{B}	48.48±3.18^{B}	47.34±1.64^{B}	58.19±6.67^{AB}	47.97±2.42^{B}	54.04±3.20^{B}
Δ-7-avenasterol	219.06±2.88^{A}	236.18±4.74^{AB}	223.64±19.59^{AB}	225.24±16.40^{AB}	249.14±20.81^{B}	218.91±9.07^{A}	237.03±15.56^{AB}
Total sterols	5281.90±427.17^{A}	5267.78±117.03^{A}	5064.94±454.57^{A}	4859.96±172.21^{A}	5425.04±451.54^{A}	4935.18±368.04^{A}	4920.47±245.30^{A}

The results are presented as mean±standard deviation (n=4). Means in the same line with different letters are significantly different according to Duncan's multiple range test ($p < 0.05$).

sterol amount (Zhou *et al*., 2016) for various types of oils. β-sitosterol, which accounted for 66.08-67.02% of all sterols, showed a similar trend to total sterols and was not affected by microwave radiation. Δ5-avenasterol was the second most abundant sterol and accounted for 19.74-20.31%. Different microwave power settings and roasting periods were found to be ineffective on Δ5-avenasterol content. The third major sterol was Δ7-avenasterol, ranging from 4.16-4.81% of the sterol fraction and the highest value was obtained at 460 W power and 10 min duration. Campesterol and stigmasterol were the other two sterols and varied from 2.94-3.07% and 2.21-2.40%, respectively. Campesterol was not affected by microwave roasting as in 24-methylene cholesterol, sitostanol, campestanol, clerosterol and Δ5,24 stigmastadienol. Stigmasterol and the remaining individual sterols were found to be only slightly affected by radiation.

In the current work, the principal component analysis (PCA) was performed to provide an overview of the microwave pre-treatment, oil quality and chemical parameters. To perform PCA, the data were displayed in a matrix. The variables which had Kaiser–Meyer–Olkin measurement of sampling adequacy indexes lower than 0.5 were eliminated and the remaining variables were PV, K_{232}, K_{270}, α-tocopherol, γ- tocopherol, C20:0, C18:2, C18:1, C18:3, C18:0, LnLnLn, LnLL, LnLnO, LLL, LnLO, LnLP, LnLnS, LnOO, SLLn, OOO, SLO, campesterol, stigmasterol, campestanol, β-sitosterol, Δ-7-avenasterol, Δ-5,24-stigmastadienol, Δ-7 campesterol and total sterols. The factor score plot is shown in Figure 1. The first two principal components explained 67.59% of the variance (Factor 1: 38.60%, Factor 2: 28.99%). F1 showed high and positive correlations with PV, LnLnS, campestanol and negative correlations with α-tocopherol, LnLP and SLLn. F2 is positively correlated with LnLnLn, β-sitosterol, Δ-5,24-stigmastadienol, total sterols and negatively correlated with LnLO. The factor score plot showed that oils obtained by microwave radiation at 460 and 600 W for 5 and 10 minutes showed a positive correlation; whereas the oils obtained from treatment at 350 W (5 and 10 min) and unroasted seeds had negative correlations with F1. Additionally, fig seeds that were roasted for 10 min at 460 W power setting had a positive correlation and seeds that were processed for 5 min at 460 and 600 W power had negative correlations with F2.

Agglomerative hierarchial clustering (AHC) is an unsupervised method that is used to acquire clusters in terms of their closeness. In the current work, AHC was carried out to reveal the discrimination of the fig seed oils according to microwave treatment. A dendrogram derived from AHC, where 2 main clusters can be observed, is given in Figure 2. In the first cluster, 350 W- 5 min and 350 W- 10 min formed a couple and were sur-

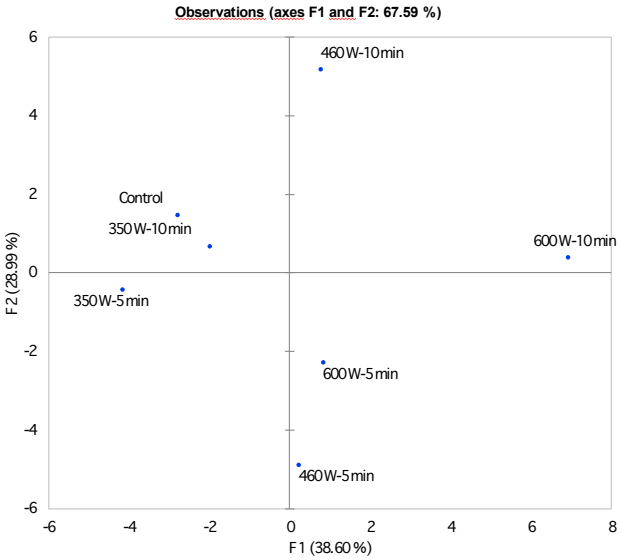

FIGURE 1. PCA score plot presenting the relations between unroasted and MW roasted (350, 460 and 600 W for 5 and 10 minutes) fig seed oils

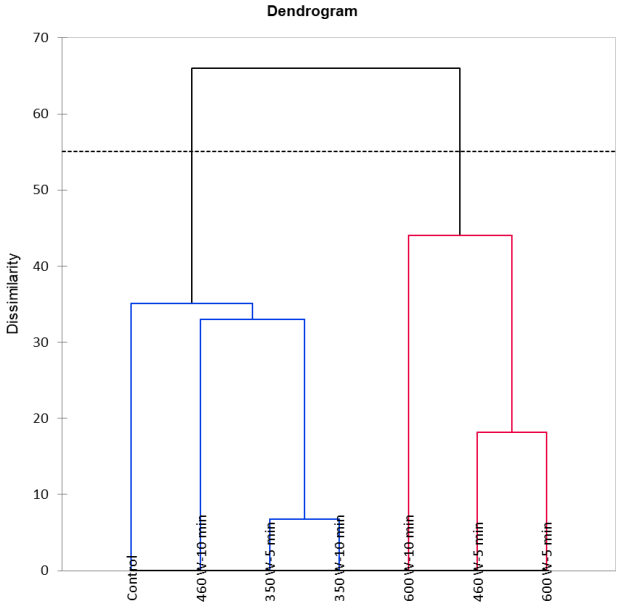

FIGURE 2. AHC dendrogram presenting the closeness of unroasted and MW roasted (350, 460 and 600 W for 5 and 10 minutes) fig seed oils

rounded by 460 W-10 min. This group was conjoined with the unroasted sample, verifying their position on the PCA score plot. In the second cluster 460 W- 5min and 600 W- 5 min were determined to be closely related and surrounded by 600 W- 10 min.

4. CONCLUSIONS

This work reports the changes in chemical composition and bioactive contents in fig seed oil due to microwave pre-treatment. Fatty acid and triacylglcerol profiles were significantly affected by microwave application, however, a certain pattern was not observed due to increasing power and radiation times. Phytosterols were determined to be protected in the oil rather than a possible degradation; whereas significant losses were detected for both α- and γ- tocopherols. PCA and AHC analyses served to differentiate intense and mild microwave-treated oils as well as the unroasted samples. Although a considerable number of works have been published about the effect of microwave pre-treatment on the composition of different vegetable oils, this is the first report evaluating the influence of roasting on cold-pressed fig seed oil. Fig seed oil was determined to be an excellent source tocopherol and the proper selection of processing method can significantly improve the nutritive value of the resultant oil. The findings of the work can contribute to further projects on fig seeds and oils for better evaluation of these nutrient-rich underused seeds. Also, more work should be performed to clarify the other compositional parameters of fig seed oil.

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