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Impact of Roasting on Quality and Compositional Characteristics of Fig Seed Oil

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ABSTRACT

This study investigates the effect of roasting time and temperature on the chemical parameters and bioactive compound content of fig seed oil. For this purpose, fig seeds were ground and roasted in an electrical oven at 100, 150 and 200 °C for 10 and 20 minutes. After roasting, the seeds were processed into oil using a laboratory scale mill. The obtained fig seed oil samples were evaluated for their peroxide value, K232 and K270 spectrophotometric indices; fatty acid, triacylglycerol, sterol and tocopherol profiles. That results show that peroxide values, spectrophotometric indices, α- and γ-tocopherol levels were negatively affected by the roasting process. The major tocopherol was γ-tocopherol and ranged in 3914-4016 mg kg-1. The main fatty acid was linolenic acid constituting 40% of the fatty acids; followed by linoleic (31.76-31.97%), oleic (17.06-17.30%) and palmitic (7.00-7.11%) acids. The major triglycerides were LnLO (12.27-12.46%), LnLnL (12.18-12.40%), LnLnLn (9.32-9.52%) and LnLL (9.34-9.50%) confirming the fatty acid profile. β-sitosterol was the predominant phytosterol ranging in 2985.07-3623.24 mg/kg followed by Δ-5-avenasterol (19.52-20.60%) and Δ-7-avenasterol (4.16-4.81%). The principal component analysis results enabled a clear discrimination between unroasted, mildly-roasted and intense-roasted oil samples.

Keywords: γ-tocopherol, Fig seed oil, Roasting, sterol, Triacylglycerol

1. Introduction

Fig (*Ficus carica* L.) is a world-famous fruit tree from the Moraceae family and has been largely cultivated for its fruits (Solomon et al. 2006) because figs have a higher nutritional value than most other fruits (Varhan et al. 2019). The fruit has been consumed both in fresh and dry forms as a dietary food product since at least the earliest civilizations. The various parts of *Ficus carica* L. can be used for a number of different purposes (Palmeira et al. 2019). Fig syrup has been used for therapeutic purposes (Khare 2007), the plant latex is used as curdling agent in the production of various dairy products, the leaves are traditionally used as animal feed and the wood is used for ornamental purposes (Badgujar et al. 2014).

The fruit from fig trees provide high concentrations of biochemicals and antioxidants (Hssaini et al. 2020). Phytochemical studies have shown that the fruit contains phenolic compounds, fatty acids, amino acids, anthocyanins, aliphatic alcohols, organic acids, volatile components, phytosterols, hydrocarbons and a number of secondary metabolites (Badgujar et al. 2014). The fruit is a rich source of phenolics that affect the antioxidant capacity, sensory features and health-promoting properties (Caro and Piga, 2008). Rutin has been reported to be the major phenolic of the figs, followed by (+)-catechin, (−)-epicatechin, chlorogenic acid, syringic and gallic acid (Veberic et al. 2008). The fruit has also been reported to contain iron, calcium, potassium and vitamin K (Joseph & Raj 2011). Due to its prosperous composition, the health attributes of the fig have been the subject of numerous studies and various studies have noted the antispasmodic, antiplatelet (Gilani et al. 2008), anticonstipationon (Kim et al. 2010) and hypoglycemic effects (El-Shobaki et al. 2010) of the fruit. In addition, the anthocyanin content of figs have been shown to prevent obesity, cardiovascular disease and certain cancers in addition to diabetes (Wojdyło et al. 2016). For this reason, consumer demand has increased in recent years for figs and fig-based products (Teruel-Andreu et al. 2021).

Figs contain a broad number of tiny seeds that give the characteristic nutty taste of the fruit. The number of the seeds may be up to 1600 and vary depending on the size and the maturity of the fruit. Fig seeds have been shown to be good sources of carbohydrate (52.62-53.66%), protein (14.74-15.07%), ash (2.99%) and oil (23.06-23.67%). Moreover, the seeds are important reserves of minerals including Mg, Mn, Zn, Fe, Ca, Cu, Na, K and P (Nakilcioğlu-Taş 2018). A reasonable amount of waste is produced during pureeing and juice production from the figs. The obtained by-product is appreciably rich in terms of fig seeds and typically used for obtaining fig seed oil, which is characterized by its high linolenic acid content as well as by its oleic, linoleic, stearic and palmitic acid levels (İçyer et al. 2017). Dietary fat composition is known to play fundamental roles in biological activities and the replacement of saturated fatty acids with polyunsaturated acids has shown to have the potential to eliminate the risk of type-2 diabetes and cardiovascular disorders (Lenighan et al. 2019). Fig seed oil is a viable ingredient for common dietary items as a good source of plant based polyunsaturated fatty acids. In addition to its fatty acid composition, fig seed oil has been designated as a very rich source of γ-tocopherol (Güven et al. 2019). γ-tocopherol has recently gained considerable interest due to its possible complementary effects to those of α-tocopherol with respect to health issues (Wagner et al. 2004).

Oilseeds from diverse sources have gained increased interest with increasing demand in vegetable oils for use as a food ingredient or for cooking purposes. Fats & oils are staple foods in many countries as lipids are critical nutrients that affect the taste & structure of the foods (Issaoui & Delgado 2019). The oils are extracted from the seeds via processing techniques that include cleaning, drying, size reduction, roasting, solvent extraction or pressing steps. Roasting is a key step for assisting the release of oil from the seeds and affects the oxidative stability, quality, nutritional characteristics and shelf life of the oil. Various studies have analyzed the influence of pre-roasting prior to the extraction of oil from sunflower (Goszkiewicz et al. 2020), safflower (Taha & Matthäus 2018), rapeseed (Jing et al. 2020), sesame (Ji et al. 2019), pumpkin (Potočnik et al. 2018), black cumin (Suri et al. 2019a), chia (Ghafoor et al. 2018) and other seeds. However, there is no study showing the effects of the roasting process before the extraction on the stability, quality and chemical composition of fig seed oil. For this reason, the aim of this study is to monitor the changes in composition (fatty acid and triacylglycerol profiles) and bioactive compound content (sterols and tocopherols) of fig seed oil by roasting process before mechanical extraction. Since the heat intensity affects the composition of the oil, different roasting temperatures (100, 150, 200 °C) and periods (10, 20 min) were considered.

2. Material and Methods

2.1. Materials

Fig seeds were obtained from a local supplier (Egesia Natural Products, Aydın/Turkey). Sodium chloride, potassium hydroxide, sodium thiosulphate, acetic acid, chloroform, ethyl alcohol, acetone, acetonitrile, pyridine, chloroform and phenolphthalein were from Merck (Darmstadt, Germany). n-hexane, isooctane, diethyl ether, hydrochloric acid, methanol, sodium hydroxide, 2,2-diphenyl-1-picrylhydrazyl, α-,β-, γ- and δ-tocopherol standards, 5α-cholestan-3β-ol and β-sitosterol were purchased from Sigma-Aldrich (St-Louis, USA). N,O-Bis(trimethylsilyl)trifluoroacetamide-BSTFA with trimethylchlorosilane and 37 fatty acid methyl ester mix were from Supelco (Bellefonte, USA). The reagents used for high-performance liquid chromatography (HPLC) analyses were of HPLC grade.

2.2. Methods

2.2.1. Roasting of fig seeds and oil extraction

The fig seeds were first milled in an electrical coffee miller (Sinbo, Turkey) and then roasted at 100, 150 and 200 °C for 10 and 20 minutes in an electrical oven (Nüve, Turkey). After the roasting process, the milled seeds were cooled to room temperature. The oil samples were obtained by pressing with a laboratory scale (12 kg seed h-1 capacity, single head, 1.5 kW power, 2 hp) screw press (Koçmaksan KMS 10; Izmir, Turkey). After the extraction, the oils were centrifuged and then stored in dark glass bottles at 4 ºC in nitrogen atmosphere until analyses.

2.2.2. Peroxide value

The peroxide values of the samples were determined according to AOCS Official Method Cd 8-53. Briefly, 1 g of oil was weighed in a 250 mL erlenmayer flask with glass stopper and dissolved with 30 mL of acetic acid-chloroform solution (3:2 v/v). Saturated potassium iodide solution (0.5 mL) was poured into that solution and agitated gently for 1 min followed by the addition of 30 mL of distilled

water. The mixture was then titrated with 0.01 N $Na_2S_2O_3$ until the yellow color faded. Starch indicator was added and the titration was continued until the blue color disappeared. The peroxide value was calculated using the following equation:

PV (meq O_2/kg oil)=(S-B)xNx100/mass of sample (g)

B: volume of titrant, mL of blank

S: volume of titrant, mL of sample

N: normality of sodium thiosulfate solution

2.2.3. UV spectrophotometric indices (K232 and K270)

The K232 and K270 values were obtained according to American Oil Chemists' Society (AOCS) Official Methods Ch 5-91 (AOCS 2003). Each oil sample was weighed in a 10 mL volumetric flask and diluted in isooctane. The resulting solution was filled in quartz cuvette (optical light path of 1 cm) and the extinction values were determined using a ultraviolet-visible (UV-VIS) spectrophotometer (UV-1800, Shimadzu, Japan).

2.2.4. Antioxidant activity

The antioxidant activity of fig seed oils was determined utilizing the free radical diphenylpicrylhydrazyl (DPPH) method of Atoui et al. (2005). For this purpose, 2.5 g of fig seed oil was diluted with 2.5 mL of methyl alcohol. Fifty microliters of the extract was mixed with 1.950 mL of DPPH solution (6x10-5 M). The mixture was kept in dark cabinet at 25 °C for 60 minutes. After the incubation period, the absorbance was measured at 515 nm with a UV-VIS spectrophotometer (UV-1800, Shimadzu, Japan), using methyl alcohol as blank. The antioxidant activity was determined using the equation given below and expressed as the percentage inhibition of the DPPH radical:

AA (%)=[(Abscontrol – Abssample) x 100]/(Abscontrol)

2.2.5. Tocopherol composition

The oil sample of 1.0 g was dissolved in 10 mL of n-hexane. The tocopherol profile was determined with an HPLC instrument (Shimadzu, Kyoto, Japan) equipped with an InertSustain NH2 column (250 mm x 4.6 mm, 5 µm particle size, GL Sciences, Tokio, Japan). The mobile phase was was n-hexane:isopropyl alcohol (99.5:0.5) with a flow rate of 1.2 mL min-1. The peaks were detected with a UV detector and the detection wavelength was 290 nm. The injection volume was 20 µl. Tocopherol homologoues were identified and quantified using external standards. The results were given as mg kg-1.

2.2.6. Fatty acid composition

The methyl esters of fatty acids were prepared using the method of the International Union of Pure and Applied Chemistry (IUPAC) (IUPAC 1987). The oil sample of 0.4 g was dissolved in 4 mL isooctane and later mixed with 0.2 mL of 2 N methanolic KOH. The mixture was agitated and kept in the dark for 6 min. 0.45 mL of 1 N HCl solution was added to the mixture with a few drops of methyl orange. The mixture was left for 30 min and the upper layer was collected for chromatographic analyses. The esters were then analysed with a gas chromatograph (GC 2010, Shimadzu/Japan). DB-23 fused silica capillary column (60 m x 0.25 mm i.d. and 0.25 µm film thickness) (J&W Scientific) was used for the elution of individual fatty acids. A FID was used to detect the peaks. The carrier gas was nitrogen (99.99% purity) with a flow rate of 1 mL min-1 and the split ratio was 80:1. The detector, column and injector temperatures were 240 °C, 195 °C and 230 °C, respectively. The fatty acid peaks were identified by comparing the retention times with the ones obtained for the respective standards. The results were expressed as a percent of the total methylated fatty acids.

2.2.7. Triacylglycerol composition

The triacylglycerol composition of the fig seed oils was determined according to the AOCS Official Method Ce 5b-89 (AOCS 2003). 0.5 g of oil was dissolved in 10 mL acetone. A chromatographic analysis of the oil solution was carried out using a HPLC instrument (Shimadzu, Kyoto, Japan) equipped with a differential refractometer detector (RID). An ACE 5 C18 column (250×4.6×5µm particle size, ACE, Aberdeen, Scotland) was employed for the elution of the peaks. The mobile phase was acetone:acetonitrile (100:100)

with a flow rate of 1.5 mL min-1 and the injection volume was 10 µl. The oven temperature was 30 °C. Triacylglycerol peaks were identified through comparison with literature data (Holčapek et al. 2005). The results were expressed as area % of the total triacylglycerols.

2.2.8. Sterol composition

The sterol composition of the oils was determined according to AOCS Official method Ch 6-91 (AOCS 2003). The sterol fraction was obtained through saponification with ethanolic potassium hydroxide solution, then extracted three times with diethyl ether and separated by thin layer chromatography. Afterwards, trimethyl silyl ether derivatives of the sterols were analyzed using a gas chromatograph (GC 2010, Shimadzu, Japan) equipped with a flame ionisation detector (FID). Then, the sterol fractions were separated with a HP-5 fused silica capillary column (30 m, 0.25 mm i.d. and 0.25 micrometer film thickness, Chrom Tech., Apple Walley, M, USA). Injector, column and detector temperatures were 280 °C, 260 °C and 290 °C, respectively. Nitrogen was the carrier gas with a flow rate of 0.8 mL min-1. The split ratio was adjusted as 50:1. 5α-cholestan-3β-ol was used as the internal standard for quantification.

2.2.9. Statistical analysis

The statistical analysis was carried out using SPSS 15 packaged software (SPSS Inc., Chicago, USA). Data were evaluated by one-way analysis of variance (ANOVA) using Duncan's multiple range test to check if there were any significant differences among the analysed parameters. A p value of less than 0.05 was considered as significant. Data were also processed by principal component analysis (PCA) using XLSTAT 2021 version (Addinsoft, New York, NY, USA).

3. Results and Discussion

The changes in peroxide values, spectrophotometric indices, tocopherol contents and antioxidant activities of the oils obtained from seeds roasted at different temperatures and times were given Table 1. The peroxide value, which is a measure of the hydroperoxides (primary products of the lipid oxidation) in the oil, increased with the ascending roasting times and temperatures. A significant difference was observed in the peroxide value when the roasting time was extended from 10 to 20 minutes at 200 °C ($p<0.05$). The K232 value, an indicator of the formation of the conjugated dienes, statistically remained unchanged at 100 and 150 °C of roasting temperatures; however, a significant increase was determined with increasing process time at 200 °C ($p<0.05$). K270, a marker of secondary oxidation products namely aldehydes and ketones, had a similar trend with K232 and increased statistically at the most intense roasting conditions ($p<0.05$).

Table 1- Peroxide value, UV spectrophotometric indices and tocopherol contents of oils obtained from fig seeds roasted at different temperatures and times (%)

Roasting temperature (°C)	*Roasting time (min)*	*Peroxide value (meq O2 kg^{-1} oil)*	*K232*	*K270*	*α-tocopherol (mg kg^{-1})*	*γ-tocopherol (mg kg^{-1})*	*Antioxidant activity (%)*
Unroasted		6.23±2.33A	2.81±0.09A	0.83±0.06A	114.07±0.89A	3952.39±7.64A	92.55±1.85A
100	10	10.71±1.27BC	2.86±0.17A	0.87±0.13A	108.47±2.91B	3889.77±10.23A	90.57±1.77A
	20	10.75±1.23BC	2.98±0.16A	0.73±0.09A	108.99±3.82B	3960.04±91.43A	90.82±1.66A
150	10	9.27±0.47B	3.12±0.13A	0.88±0.05A	107.14±1.96B	3954.73±48.29A	90.13±1.27A
	20	10.75±0.54BC	3.26±0.13A	0.81±0.27A	108.09±1.61B	3914.25±61.17A	90.57±1.10A
200	10	12.18±0.51C	3.12±0.05A	0.95±0.03A	108.86±1.51B	4016.40±133.08A	89.53±5.72A
	20	16.26±2.38D	4.86±0.85B	1.95±0.40B	101.63±2.29C	3629.66±123.86B	77.32±3.40B

Data in the same column followed by different letters are significantly different at $p<0.05$

Tocopherols are efficient antioxidants responsible for the stability of edible oils. α-, β-, γ-, δ- tocopherols are known to be different forms of vitamin E, and α-tocopherol is the preferred one due to its higher vitamin E activity (Delgado et al. 2020). The fig seed oil samples contained 101.63-114.07 mg kg-1 of α-tocopherol, slightly higher than the findings of İçyer et al. (2017) and Baygeldi (2018) who reported 46 mg kg-1 and 4.6 mg 100 g-1 of α- tocopherol for fig seed oils, respectively. The major tocopherol was γ-tocopherol ranging in 3914-4016 mg kg-1 in accordance with former works (Şentürk and Karaca 2021; Tarlacı 2021). The α-tocopherol contents of fig seed oils decreased by 10.90 % and γ-tocopherol quantities decreased by 8.17% through the roasting process indicating a possible

destruction in tocopherol homologues which may be attributed to thermal induced oxidation (Ji et al. 2019). The highest loss was determined at the most intense roasting process with the highest temperature (200 °C) and longer process time (20 min). Time was more effective on the reduction of both α- and γ-tocopherol contents when the temperature was increased.

The antioxidant activity of the unroasted fig seeds was 92.55% and decreased by the raise in roasting temperatures. The lowest antioxidant activity was determined at sample roasted at 200 °C for 20 minutes. Güven et al. (2019) reported lower antioxidant capacity (52.54%) for fig seed oil. The decrease in antioxidant activity is likely due to the loss of tocopherols by the extended roasting process. Antioxidant activity was found to be high and positively correlated with α- (r=0.87) and γ- (r=0.92) tocopherol contents.

The changes in fatty acid composition by roasting at different temperatures and times, were presented in Table 2. The major fatty acid was linolenic acid ranging among 40.68-40.95% in accordance with previously published studies (İçyer et al. 2017; Duman & Yazıcı 2018; Duman et al. 2018). The variation in roasting conditions did not cause any significant change on the linolenic acid ratio of the oil samples ($p>0.05$). Linoleic acid was the second dominating fatty acid covering 31.76-31.97% of the fatty acids and was found to be the lowest in unheated oil sample. Oleic acid was the main monounsaturated fatty acid with a range of 17.06-17.30%, and, similar with the findings of Nakilcioğlu-Taş (2018), found to be the highest at the most intense roasting conditions. Modest changes were determined for palmitoleic, heptadecanoic and arachidic acids; wheras myristic, palmitic, heptadecenoic, stearic and gadoleic acids remained unchanged. Previous studies have revealed either no marked differences between unroasted and roasted seeds (Ji et al. 2019; Zhang et al. 2020), or slight modifications in the fatty acid profiles of various oils through a roasting process (Hama 2017; Suri et al. 2019b).

Table 2- Fatty acid composition of oils obtained from fig seeds roasted at different temperatures and times (%)

Fatty acids	*Unroasted*	*100 °C*		*150 °C*		*200 °C*	
		10 min	*20 min*	*10 min*	*20 min*	*10 min*	*20 min*
C 14:0	0.02±0.01A	0.01±0.00A	0.01±0.00A	0.01±0.00A	0.01±0.00A	0.01±0.00A	0.01±0.00A
C 16:0	7.02±0.08A	7.06±0.04A	7.08±0.06A	7.00±0.03A	7.04±0.09A	7.00±0.07A	7.11±0.03A
C 16:1	0.12±0.02A	0.14±0.03AB	0.18±0.01B	0.15±0.00AB	0.13±0.04AB	0.15±0.04AB	0.18±0.02AB
C 17:0	0.03±0.01A	0.04±0.01AB	0.04±0.00AB	0.04±0.00AB	0.04±0.00A	0.03±0.00B	0.04±0.00AB
C 17:1	0.02±0.01A	0.02±0.00A	0.03±0.01A	0.02±0.01A	0.03±0.01A	0.02±0.00A	0.02±0.01A
C 18:0	2.53±0.04A	2.50±0.05A	2.45±0.03A	2.52±0.06A	2.50±0.06A	2.49±0.05A	2.50±0.03A
C 18:1	17.23±0.27AB	17.13±0.10AB	17.06±0.10AB	17.21±0.05AB	17.10±0.04AB	17.17±0.08AB	17.30±0.06B
C 18:2	31.76±0.09A	31.94±0.10B	31.97±0.09B	31.90±0.08AB	31.92±0.03AB	31.90±0.12AB	31.89±0.06AB
C 18:3	40.74±0.29A	40.86±0.09A	40.95±0.08A	40.83±0.11A	40.95±0.19A	40.93±0.12A	40.68±0.09A
C 20:0	0.10±0.02AB	0.11±0.01B	0.08±0.01A	0.11±0.01B	0.10±0.02AB	0.11±0.01B	0.10±0.01AB
C 20:1	0.18±0.03A	0.19±0.01A	0.16±0.01A	0.13±0.01A	0.18±0.02A	0.19±0.02A	0.18±0.02A

Data in the same line followed by different letters are significantly different at $p<0.05$

Triglycerides are the major components of edible oils and represent 95-99% of the structure. The changes in the triacylglycerol composition of fig seed oils during roasting were given in Table 3. The major triglycerides of fig seed oils were determined to be LnLO (oleolinoleolinolenin), LnLnLn (trilinolenin), LnLLn (linoleodilinolenin) and LnLL (dilinoleolinolenin). Additionally, LnLnO (oleodilinolenin), LnLP (palmitolinoleolinolenin), LLO (oleodilinolein), SLLn (stearolinoleolinolenin), LnLnP (palmitodilinolenin), LLP (palmitodilinolein), LOP (palmitooleolinolein), LOO (dioleolinolein), LLL (trilinolein), LnOO (dioleolinolenin), LnLnS (stearodilinolenin), SLO (stearooleolinolein), SOLn (stearooleolinolenin) and OOO (triolein) were detected in smaller ratios. The major triglyceride, LnLO ranged in 12.27-12.46% and slightly increased by the roasting process. The other three main triglycerides, namely LnLnL, LnLnLn and LnLL, varied in 12.18-12.40%, 9.32-9.52%, 9.34-9.50% respectively and decreased by roasting at 200 °C for 20 minutes. LnLnP, LnLnS, LLO, LOP, SOLn, OOO, SLO were determined to remain unchanged, wheras slight changes were detected for the remaining triacylglycerols.

Table 3- Triacylglycerol profile of oils obtained from fig seeds roasted at different temperatures and times (%)

Triacylglycerols	*Unroasted*	*100 °C*		*150 °C*		*200 °C*	
		10 min	*20 min*	*10 min*	*20 min*	*10 min*	*20 min*
LnLnLn	9.52±0.07A	9.45±0.03A	9.50±0.04A	9.48±0.02A	9.43±0.07AB	9.47±0.14A	9.32±0.09B
LnLnL	12.40±0.13A	12.38±0.04A	12.36±0.05A	12.38±0.06A	12.29±0.07AB	12.38±0.11A	12.18±0.12B
LnLL	9.47±0.07AB	9.50±0.07A	9.45±0.11AB	9.37±0.04AB	9.34±0.12B	9.48±0.13AB	9.39±0.06AB
LnLnO	8.73±0.13AB	8.73±0.09AB	8.81±0.05AB	8.76±0.07AB	8.86±0.12A	8.75±0.13AB	8.68±0.08B
LnLnP	4.92±0.05A	4.93±0.01A	4.92±0.06A	4.87±0.04A	4.90±0.05A	4.86±0.08A	4.90±0.07A
LLL	3.86±0.10A	3.83±0.05AB	3.74±0.06ABC	3.78±0.09ABC	3.59±0.12D	3.68±0.07CD	3.72±0.08BCD
LnLO	12.27±0.13A	12.32±0.05AB	12.39±0.12AB	12.38±0.11AB	12.46±0.09B	12.39±0.05AB	12.37±0.08AB
LnLP	7.13±0.10A	7.18±0.04A	7.15±0.12A	7.16±0.09A	7.12±0.16A	6.94±0.11B	7.03±0.14AB
LnLnS	2.24±0.04A	2.16±0.05A	2.01±0.19A	2.09±0.22A	2.05±0.15A	2.03±0.19A	2.24±0.19A
LLO	6.79±0.06A	6.76±0.08A	6.86±0.15A	6.78±0.05A	6.87±0.08A	6.89±0.05A	6.88±0.07A
LnOO	3.02±0.20A	2.85±0.09AB	2.84±0.03AB	2.80±0.04B	2.76±0.16A	2.81±0.12A	2.86±0.09AB
LLP	4.05±0.07A	4.30±0.09B	4.33±0.09B	4.27±0.10B	4.29±0.09B	4.36±0.10B	4.33±0.07B
SLLn	5.41±0.14A	5.44±0.05A	5.64±0.17B	5.64±0.05B	5.70±0.09B	5.77±0.03B	5.73±0.09B
LOO	3.15±0.12AB	3.12±0.09AB	3.05±0.11A	3.26±0.21AB	3.33±0.15B	3.28±0.21AB	3.31±0.18AB
LOP	4.08±0.04A	4.05±0.05A	4.01±0.03A	4.06±0.07A	4.08±0.10A	4.04±0.07A	4.10±0.06A
SOLn	0.74±0.05A	0.77±0.04A	0.73±0.06A	0.72±0.08A	0.71±0.06A	0.69±0.04A	0.74±0.02A
OOO	0.66±0.01A	0.63±0.03A	0.66±0.01A	0.64±0.04A	0.65±0.01A	0.66±0.05A	0.65±0.03A
SLO	1.58±0.02A	1.60±0.03A	1.55±0.01A	1.55±0.10A	1.59±0.04A	1.52±0.03A	1.58±0.0A

Data in the same line followed by different letters are significantly different at $p<0.05$

The sterol profiles of fig seed oils obtained by oven-roasting are given in Table 4. The main sterols of fig seed oils were β-sitosterol, Δ-5-avenasterol and Δ-7-avenasterol. In addition, campesterol, sitostanol, clerosterol, stigmasterol, 24-methylene-cholesterol, Δ-7-stigmastenol, campestanol, Δ-5-24-stigmastadienol and Δ-7-campesterol were detected in lower amounts. The total sterol contents varied in 4582.46 and 5499.98 mg kg-1 and the extension in roasting time increased the total sterol contents of fig seed oils at 100 and 200 °C of processing ($p<0.05$). The results may be attributed to the damage of the cell membrane that increases the release of sterols and enrich their contents in the extracted oil, as was also previously shown by Azadmard-Damirchi et al. (2010). Güven et al. (2019) determined 6516.20 mg kg-1 of sterols in fig seed oil samples. Roasting process has been reported to cause either increments (Rękas et al. 2015) or decreases (Amaral et al. 2006) in the total sterol contents of resulting oils. β-sitosterol accounted for 64.66-65.85% of the sterols and had a similar tendency with the total sterol content. The prolongation of process time, increased β-sitosterol content at 100 and 200 °C of roasting ($p<0.05$). Δ-5-avenasterol was the second dominating sterol and covered 19.52-20.60% of the sterols. Different process temperatures and times were found to be uninfluential on Δ-5-avenasterol content. Δ-7-avenasterol was the third noticeable sterol ranging in 4.16-4.81% and was not affected by the roasting process. Campesterol and stigmasterol were the other two sterols varying in 2.92-3.15% and 2.20-2.45%, respectively. Sitostanol was not significantly affected by the roasting process, whereas modest changes were determined for the remaining 24-methylene cholesterol, campestanol, Δ-7-campesterol, Δ-5,24 stigmastadienol clerosterol and Δ-7-stigmastenol.

Table 4- Sterol content of oils obtained from fig seeds roasted at different temperatures and times (mg kg^{-1})

Sterols	*Unroasted*	*100 °C*		*150 °C*		*200 °C*	
		10 min	*20 min*	*10 min*	*20 min*	*10 min*	*20 min*
24-methylene cholesterol	15.54±3.04AB	12.33±4.58A	18.69±3.01B	15.26±2.68AB	16.81±1.61AB	16.35±2.43AB	17.11±4.00AB
Campesterol	150.69±8.62ABC	134.85±2.99A	172.01±25.06C	154.42±7.75ABC	146.31±5.24AB	142.75±9.51AB	165.90±20.53BC
Campestanol	1.13±0.63A	1.06±0.64A	2.14±0.53AB	1.48±0.36A	1.31±0.58A	1.57±0.32A	2.69±1.26B
Stigmasterol	115.92±13.30AB	105.63±2.10A	119.93±10.59AB	116.24±6.12AB	118.11±5.93AB	109.59±6.80AB	121.38±10.59B
Δ-7-campesterol	5.41±0.60AB	6.10±1.33AB	6.87±1.03AB	5.21±1.30A	7.10±1.26B	6.05±0.81AB	6.14±0.45AB

Table 4- Continued

Sterols	*Unroasted*	*100 °C*		*150 °C*		*200 °C*	
		10 min	*20 min*	*10 min*	*20 min*	*10 min*	*20 min*
Clerosterol	29.26±3.66AB	25.53±0.65A	33.15±3.36B	25.33±4.33A	28.71±2.38AB	26.48±3.51A	30.52±1.79AB
β-sitosterol	3314.47±585.85AB	2985.07±96.37A	3623.24±319.16B	3448.72±227.99AB	3173.30±293.05AB	2990.86±205.45A	3616.35±513.77B
Sitostanol	15.13±4.33A	15.51±9.79A	15.99±7.07A	11.85±1.70A	16.30±3.61A	18.97±6.58A	14.10±7.25A
Δ-5-avenasterol	990.86±129.97A	950.82±11.73A	1048.55±100.65A	1055.15±45.62A	934.97±105.67A	914.35±70.30A	1072.70±144.76A
Δ-5,24 stigmastadienol	95.04±4.22A	85.88±2.16A	95.21±9.23A	90.00±4.41A	88.83±8.54A	83.47±7.63A	123.59±39.40B
Δ-7-stigmastenol	55.24±8.26A	50.79±3.36A	56.51±5.58AB	50.27±3.62A	53.26±5.76A	48.49±3.94A	68.42±17.86B
Δ-7-avenasterol	227.86±31.07A	242.25±18.69A	263.63±14.98A	242.05±32.21A	244.02±29.05A	223.52±19.13A	261.09±22.82A
Total sterols	5018.69±751.63AB	4615.83±85.75A	5455.92±434.04B	5215.98±316.79AB	4829.04±427.41AB	4582.46±207.49A	5499.98±778.20B

Data in the same line followed by different letters are significantly different at $p<0.05$

The PCAs is a multivariate analysis method that reduces the variables to a smaller number of factors with maximum variation. In this study PCA was carried out to provide an overview of the roasting pretreatment, oil quality and compositional parameters. To perform the analysis, analytical data were arranged in a matrix. The variables which had the Kaiser-Meyer-Olkin measure of sampling adequacy index lower than 0.5 were removed and the remaining adequate variables were the peroxide value, K232, K270, α- and γ-tocopherols, antioxidant activity, C14:0, C16:0, C16:1, C17:0, C17:1, C18:0, C18:1, C18:2, C18:3, C20:0, LnLnLn, LnLnL, LnLnO, LLL, LnLO, LnLP, LnLnS, LLO, LnOO, LLP, SLLn, LOO, SOLn, 24-methylene cholesterol, campesterol, stigmasterol, campestanol, Δ-7-campesterol, β-sitosterol, clerosterol, sitostanol, Δ-5-avenasterol, Δ-5,24 stigmastadienol, Δ-7-avenasterol, Δ-7-stigmastenol and total sterols. The factor score plot is given in Figure 1. The first two factors explained 62.83% of the total variance (Factor 1: 37.70%, Factor 2: 25.13%). F1 showed high and positive correlations with peroxide value, K232, C16:1, campestanol, Δ-5,24-stigmastadienol, Δ-7-stigmastenol and Δ-7-avenasterol. The factor score plot showed that unroasted and intense-roasted (200 °C, 20 min) oil samples were clearly discriminated from the others. Mildly roasted samples for a short period (100 °C and 150 °C for 10 minutes) were clustered together, whereas a similar group was detected for samples roasted for longer time (100 °C and 150 °C for 20 minutes).

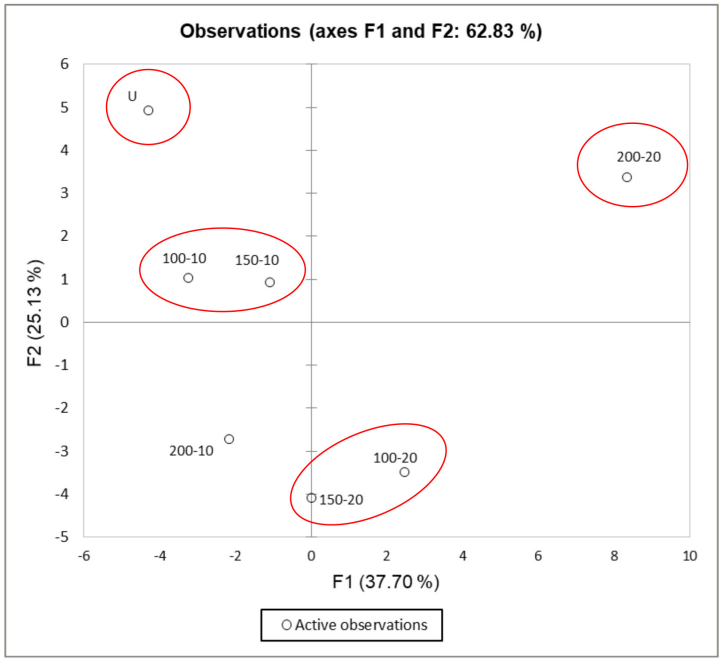

Figure 1- PCA score plot presenting the relations between unroasted and roasted (100, 150 and 200 °C for 5, 10and 15 minutes) fig seed oils

4. Conclusions

The current study reports the changes in quality parameters, antioxidant activity, chemical composition and bioactive contents of the fig seed oil by roasting process. Significant losses were detected in quality parameters, antioxidant activities and tocopherol contents of oils by roasting process. Although modest changes were observed for fatty acid, triacylglycerol and sterol profiles of oils; no clear pattern was determined by increasing time and temperature. Principle component analysis enabled an obvious separation between unroasted, mildly heated and intense heated samples. A great number of studies have been published about the effect of roasting on the composition of different vegetable oils. Yet, this is the first report investigating the effect of roasting process and parameters on fig seed oil. Further studies should be conducted to investigate the unenlighted compositional parameters of fig seed oil.

Data availability: Data are available on request due to privacy or other restrictions.

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