Short Note

Effect of conventional and microwave heating on the oxidative stability of oil from a wild Turkish hazelnut cultivar (*Corylus colurna*)

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The effects of microwave (5, 10, 15, and 20 min at 600 W) and conventional heating (60°C) on the quality and stability of oil extracted from a wild Turkish hazelnut (*Corylus colurna*) cultivar was performed and compared. Oxidative stability of oil samples was measured by measuring peroxide value (PV) and UV absorption characteristics (K_{232} and K_{270}). Degradation of total phenolics, tocopherols, and fatty acids was also examined during heating experiments. The increase in PV and conjugated dienes (K_{232}) of conventional-heated oils was higher than that of microwave-heated oils. α-Tocopherol is consumed fast during both conventional and microwave heating treatments. Microwave and conventional heating resulted in the degradation of phenolics content in the heated oils.

Keywords: Tocols, Phenolic compounds, UV absorption, *Corylus colurna*, Oxidation, Thermal treatment.

1. INTRODUCTION

Hazelnut is an important crop with an estimated production exceeding 1.000.000 tonnes of shelled hazelnuts, according to FAOSTAT 2017 data. Among hazelnut producers, Turkey is one of the important countries with 675.000 tonnes of shelled hazelnuts [1-5] Giresun or Levant terms are used to classify hazelnuts according to quality. Hazelnuts contained in the Giresun group are provided as premium quality, while the Levant group is qualified as a secondary quality. Giresun type hazelnuts contain more oil than Levant type, and most of Giresun hazelnuts preferred for its shape and unique aroma [6, 7].
Turkish hazelnut (*Corylus colurna*) is a wild cultivar not under cultivation. This hazelnut cultivar is quite different from the other cultivated hazelnuts. Cultural hazelnuts usually grow to 5-6 m, this cultivar is defined as the tree and could grow up to 25 m [8]. This cultivar grows as a wild hazelnut spice in the forests of Romania, Transcaucasia, the Balkans, northern Turkey, and Iran [9].
Hazelnuts are commonly used in a variety of foods, such as dairy and candy [10]. This wild hazelnut could be used in the confectionery industry, especially chocolates locally due to their small size. Hazelnut contains major components such as carbohydrates, oils, proteins, vitamins, and minerals. Some of the components are important for human nutrition [11].
The oil content of the Turkish hazelnut (*Corylus colurna*) is over 60% [12]. The oil content of this cultivar is comparable to European hazelnut cultivars [13]. This wild hazelnut cultivar contains up to 86% oleic acid, and 16% linoleic acid [12]. Besides, this oil contains different bioactive compounds, including tocopherols, sterols, and phenolics. The total sterol content of oil samples from five Turkish hazelnut genotypes (*Corylus colurna* L) varied between 4.52 and 6.50 mg/g oil.

β-Sitosterol was predominant sterol, with a mean percentage of 65.1% [14].
Hazelnuts are generally used as a whole; however, hazelnut contains a high amount of oil that contains some nutritional compounds such as tocols, phenolics, sterols that contribute to human health. Besides health, oxidation stability of oils from this hazelnut cultivar is essential to determine its shelf life.
Oxidation tests under different conditions are employed to determine the oxidative stability of vegetable oils. In the classical thermal oxidation conditions, vegetable oils exposure to heating. Conventional heating is a standard method used for the determination of the degradation of oils; however, highly time-consuming, many solvents used are the disadvantages of this classical method [15-17]. Microwave heating alone is not an oxidation test; it is applied in food processing for cooking techniques such as roasting, boiling, and frying [18]. Numerous researchers tested the classical Schaal oven test at 60°C applied to hazelnut oils to determine the oils' oxidative stability [19-21]. Few studies deal with the oxidation of hazelnut oil as affected by microwave heating. Kiralan and Kiralan [22] determined oxidative changes in hazelnut oil heated with microwave energy, using ultraviolet absorbance and values of volatile oxidation compounds.
Turkish hazelnut (*Corylus colurna*) is rich in oils so they could be evaluated for edible oils. The oil also contains bioactive compounds that contribute to the oxidative stability of the oil. To the best of our knowledge, there are no studies on the effects of different thermal oxidation conditions (oven test and microwave heating) on oils from this cultivar. The goal of the current investigation was to study the effects of conventional heating and microwave treatment on the oxidative stability of oils from a wild Turkish hazelnut (*Corylus colurna*) cultivar. The degradation of phenolics and tocopherols, contributing to the oil's oxidative stability was also determined.

2. MATERIALS AND METHODS

2.1. SAMPLE PREPARATION AND HEATING PROCEDURE

Raw shelled hazelnuts were procured from the Bolu province of Turkey and shelled before the oil extraction. Hazelnut oils were extracted using *n*-hexane for 4 h using Soxhlet extraction. A rotary evaporator removed the solvent, and the extract was dried under nitrogen. The extracted oil was kept in a brown bottle, flushed with nitrogen, and stored in a deep-freezer (-18°C) until heating experiments.
For conventional heating, extracted hazelnut oil (20 g) was stored in darkness at 60°C in 50 mL open amber glass bottles. Bottles were taken from the oven (Memmert UN 160) for analysis at four-day intervals. The oxidation of hazelnut oils was followed by peroxide value (PV), UV absorption (K_{232} and K_{270}) characteristics. The total phenolics, tocopherol composition, and fatty acid composition were also determined in the samples received from eight-day intervals.
For microwave heating, 20 g of oil samples were filled in a 50 mL open amber glass bottles and placed on the turntable of a microwave oven (Samsung Model Model MW71E, Malaysia). The oil was heated for 5, 10, 15, and 20 min at 600 W. After the microwave heating; the temperature was measured with an electronic thermometer. The samples were allowed to cool down at room temperature. PV, UV absorption (K_{232} and K_{270}) characteristics, total phenolics, tocopherol, and fatty acid composition were determined to monitor hazelnut oil stability.

2.2. ANALYSES

2.2.1. Peroxide value and UV absorption (K_{232} and K_{270})

PV and UV absorption (K_{232} and K_{270}) were measured according to the American Oil Chemists' Society [23] Official Methods Cd 8-53 and Ch 5-91. PV was expressed as milliequivalents of active oxygen per kilogram oil (meq O_2/kg). K_{232} (CD) and K_{270} (CT) extinction coefficients were recorded from absorption at 232 and 270 nm.

2.2.2. Phenolic compounds

Phenolic compounds of oil samples were extracted according to Panagiotopoulou and Tsimidou [24] with some modifications. The phenolic fraction of hazelnut oils were extracted using solid-phase extraction (SPE) cartridges on a diol-bonded phase cartridge (Supelco, Bellefonte, USA), which were conditioned by flushing with 5 mL methanol and 5 mL hexane before the addition of sample. Hazelnut oil (1 g) was dissolved in 5 mL of hexane, and this solution passed through this cartridge, then cartridge washed with 5 mL portions of hexane. The column was eluted with 10 mL of methanol, and this solution was rewashed with 5 mL methanol. Afterward, the solvent was evaporated at 45°C using a rotary evaporator under vacuum until dryness. The residue was washed with 1 mL methanol. An aliquot (20 µL) of the final solution was injected into the HPLC system.
The HPLC working conditions were partially modified, according to Caponio *et al*. [25]. HPLC analysis was carried out using a Shimadzu HPLC system equipped with a column oven, an automatic injector, and a diode array detector (DAD). The analysis was performed on an Agilent Eclipse XDB-C18 column (250 × 4.6 mm, 5 µm) at 30°C in an isocratic mode with a constant flow rate 0.8 mL/min. An injection volume of the sample was 20 µL, and the mobile phase consisted of methanol: 3% acetic acid in water (50:50 v/v).

2.2.3. Tocopherol composition

For the analysis of tocopherols, AOCS Official Method (Ce 8-89) was performed [23]. The α-, γ-, and δ-tocopherols were quantified using external calibration for each tocopherol. The amounts of tocopherols in the samples were given in mg/kg using the calibration curve of each tocopherol isomer. The HPLC system consisted of LC-10ADvp pump, SIL–10ADvp Autosampler, an SCL-10Avp System controller, CTO-10Avp column heater, and fluorescence detector with wavelengths set at 330 nm for emission and 295 nm for excitation. The column was a normal phase Luna Silica (5 µm; 250 × 4.6 mm). Of each sample, 10 µL was injected and separated by the mobile phase, which consisted of heptane: tetrahydrofuran (95:5, v/v) with a 1.2 mL/min flow rate.

2.2.4. Fatty acid analysis

The fatty acid analysis of the oils was performed using gas chromatography (GC). Fatty acid methyl esters (FAME) were prepared according to IUPAC [26] official methods. The analysis was performed using a Shimadzu GC-2010 chromatograph equipped with an RTX-2330 fused-silica capillary column (60 m, 0.25 mm i.d., 0.20 µm film thickness, Restek, USA) and fitted with flame ionisation detector (FID). The temperature of the split injector was 250°C, with a splitting ratio of 1:100. The temperature of FID was 260°C, and the initial column temperature was 140°C (held for 5 min), raised 4°C/min to 240°C (held for 20 min). The carrier gas was helium at a flow rate of 1 mL/min. FAME was identified by comparison of their retention times with those of the reference standards.

2.3 STATISTICAL ANALYSES

The results were expressed as the mean values ± standard deviation (SD) for each measurement carried out in duplicate. The effects of heat treatments on the total phenolics, tocopherols, fatty acid profile, and on the oxidation parameters were analysed by one-way analysis of variance (ANOVA). Post hoc analysis was performed using the DUNCAN test. When p <0.05, differences were considered significant. Data were analysed using IBM SPSS 20 software.

3. RESULTS AND DISCUSSION

3.1. OVEN TEST

The changes in the PV during oven storage conditions are shown in Figure 1. PV in oil samples showed a gradual increase with storage time. At the end of 24 days of storage, PV increased to 32.78 meq O_2/kg from an initial value of 1.54 meq O_2/kg. This increase observation in PV was also noted in the oil from Tombul hazelnut cultivar [27].

The formation of K_{232} (CD) and K_{270} (CT) in hazelnut oils during the 24-days oxidation test at 60°C is summarised in Figure 2. Like PV, CD contents of hazelnut oils increased gradually as the storage time was increased. The CD reached up to 6.76 for hazelnut oil after 24-days storage. These values are comparable with those of oils from Delisava hazelnut (reach up to 7) reported by Özkan *et al*. [21]. Similarly, the CD values of *n*-hexane-extracted hazelnut oil reached up to 6.951 [20]. Specific extinction at 270 increased to 0.26 during 16 days in an oven; after this time, a decline was observed in K_{270}. This fluctuation behavior in K_{270} could be similar in Delisava and Kara Fındık hazelnut oils [21]. Phenolic compounds are important natural antioxidants that affect oil stability [28]. Table I shows the total phenolics (TP) in hazelnut oils during storage at 60°C. The initial mean value of total phenolics was 6.29 µg/g oil. Compared to fresh oil, TP content decreased with an increase of storage time. However, phenolics of oils stored at 60°C were more or less same as they showed no significant difference (p >0.05). The results concurred with those reported by Ayadi *et al*. [29], who mentioned that the content of polar phenolic compounds in the olive oils decreased slightly during heating at 60°C. α-Tocopherol (86.4 mg/kg) was the major tocopherol found in all hazelnut oils (Table I). The α-tocopherol amount in hazelnuts was like those reported previously [30]. During oven storage, the content of this isomer decreased, and at the end of storage, its concentration declined to 300.8 mg/kg. Besides, there was a slight decline trend for other isomers during storage. In agreement with our results, α-tocopherol decreased dramatically with storage time at 60°C [6, 27]. This means that α-tocopherol is the

Table I - Changes in total phenolics and tocopherol composition of hazelnut oil during oven test (60°C)

Storage (day)	Total phenolics (µg/g)	Tocopherol (mg/kg)			
		α	β	γ	δ
0	6.29±1.15a*	486.45±2.76a	27.86±1.28a	8.42±0.62a	3.61±0.13a
8	4.35±0.29b	412.45±5.40b	25.01±0.35b	7.70±0.06b	3.38±0.03b
16	4.30±0.20b	341.15±9.72c	24.21±0.45b	7.41±0.07b	3.22±0.08c
24	4.35±0.45b	300.85±3.33d	24.87±0.08b	7.49±0.07b	3.42±0.10b

*Data are expressed as means ± SD.

Means followed by different letters in the same column are significantly different (p < 0.05).

Table II - Changes in the fatty acid composition of hazelnut oil during oven test (60°C)

Fatty acid	C14:0	C16:0	C16:1	C17:0	C17:1	C18:0	C18:1	C18:2	C18:3	C20:0	C20:1	C22:0
0	0.02±0.00	6.41±0.01c	0.47±0.01	0.04±0.00	0.07±0.00	2.16±0.01c	80.59±0.02e	9.87±0.01a	0.11±0.00 a	0.12±0.01	0.14±0.00	0.02±0.00
4	0.02±0.00	6.42±0.01bc	0.47±0.00	0.04±0.00	0.07±0.00	2.20±0.00b	80.89±0.04d	9.51±0.03b	0.11±0.00a	0.11 ±0.00	0.14±0.00	0.02±0.00
8	0.02±0.00	6.43±0.01abc	0.47±0.00	0.04±0.00	0.07±0.00	2.20±0.00b	80.93±0.05d	9.47±0.04b	0.11±0.00a	0.12±0.01	0.14±0.00	0.02±0.00
12	0.02±0.00	6.43±0.01abc	0.47±0.00	0.04±0.00	0.07±0.00	2.21±0.01ab	81.02±0.04c	9.37±0.02c	0.11±0.01ab	0.12±0.01	0.14±0.00	0.02±0.00
16	0.02±0.00	6.44±0.01ab	0.47±0.00	0.04±0.00	0.07±0.00	2.20±0.00b	81.06±0.00bc	9.31±0.00c	0.10±0.00b	0.11 ±0.00	0.14±0.00	0.02±0.00
20	0.02±0.00	6.45±0.01a	0.47±0.00	0.04±0.00	0.07±0.00	2.21±0.00a	81.13±0.01ab	9.24±0.01d	0.10±0.00b	0.11 ±0.00	0.14±0.00	0.02±0.00
24	0.02±0.00	6.45±0.01a	0.47±0.00	0.04±0.00	0.07±0.00	2.21±0.00a	81.17±0.04a	9.18±0.04d	0.10±0.00b	0.12±0.01	0.14±0.00	0.02±0.00

*Data are expressed as means ± SD.
Means followed by different letters in the same column are significantly different ($p < 0.05$).

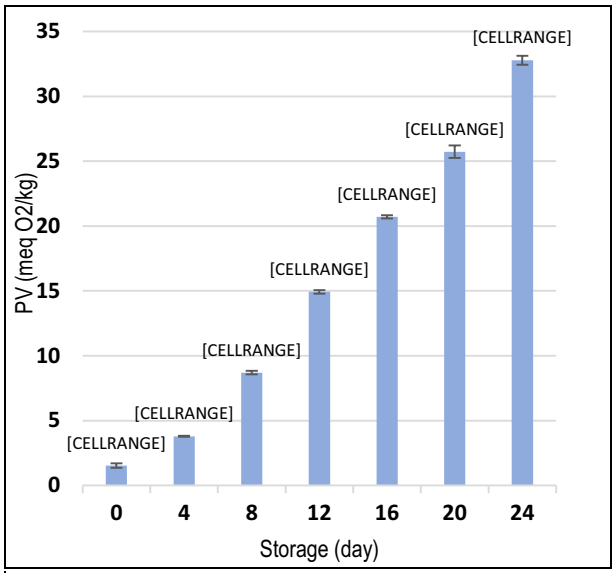

Figure 1 - Changes in PV of hazelnut oil during oven test (60°C). Data are expressed as means ± SD.
Means ± SD followed by the different letter, on bars are significantly different ($p < 0.05$).

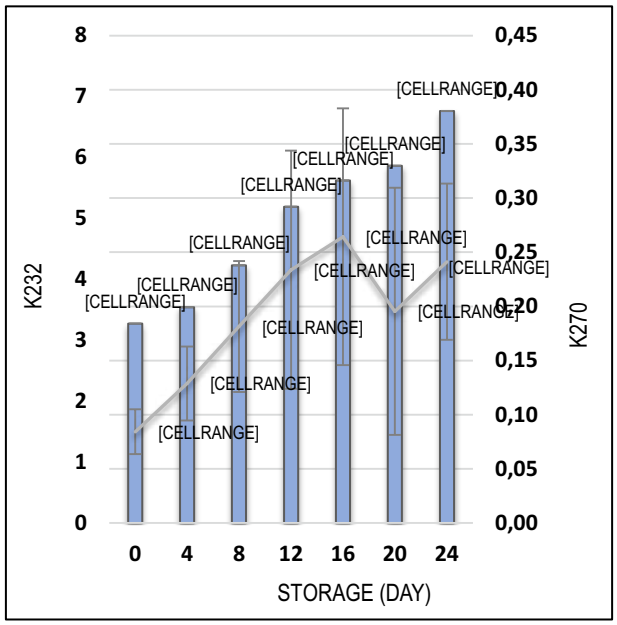

Figure 2 - Conjugated diene (K_{232}) and triene (K_{270}) values of hazelnut oils during oven test (60°C)
Data are expressed as means ± SD. Significant differences in K_{232} ($p < 0.05$) among samples are shown by different lowercase letters (a-e) and significant differences in K_{270} ($p < 0.05$) between samples are indicated by different capital letters (A-C).

first isomer to be oxidized, providing stability to the oil. This finding is like that previously reported, which mentioned that α-tocopherol tended to be more degraded in perilla and corn oils than γ- or δ-tocopherol at 60°C [31]. A similar trend was also observed in the report by Player *et al*. [32]. In this report, α-tocopherol degraded faster in soybean oil during 10 days of storage (50°C) compared those of γ -

and δ-tocopherol. Kamal-Eldin and Appelqvist [33] demonstrated that degradation of α-tocopherol could be related to the donation of hydrogen to peroxy radical (ROO·). The results also emphasised that the decline observed for phenolics was lower than those observed for α-tocopherol as previously observed for olive oil [34].

The major fatty acids in hazelnut oil samples, as determined by gas chromatography (Table II), were oleic acid, linoleic acid, and palmitic acid, which accounted for 80.59%, 9.87% and 6.41% of the total acids, respectively. The fatty acid composition found in this work is comparable to data previously reported in literature for this wild hazelnut cultivar [12]. During storage, linoleic acid content decreased. However, oleic acid content increased with storage time. Similar changes for these fatty acids were reported in Ghirardello et al. [35]. The decrease in linoleic acid during the storage of hazelnuts could be attributed to the peroxidation and subsequent loss during storage that was examined by Ghirardello et al. [35].

3.2 MICROWAVE HEATING

As expected, microwave treatments applied to oils in the current study induced a temperature increase in the samples proportional to the treatment time. Temperatures of the samples at the end of microwave treatments are given in Table III. Similar results were encountered for hazelnut oil exposed to microwave heating [22].

The results of PV for hazelnut oils subjected to oxidation are indicated in Figure 3. At 600 W power setting, the PV of hazelnut oils reached up to the highest value (2.43 meq O_2/kg) after 10 min of heating, afterward, PV decreased with increasing exposure time. These results agree with those reported for soybean oil that indicated PV increased up higher values later than decreased as exposure time increased at medium power microwave heating. These results could be related to the rapid hydroperoxide decomposition to secondary oxidation products at elevated temperature [36]. Comparing the literature on hazelnut oil, some of the results agree with the results of Arifoğlu and Öğütcü [37]. They observed

TABLE III - Mean temperature for oil samples at different treatment times

Exposure time (min)	Temperature (°C)
5	135
10	184
15	205.5
20	221

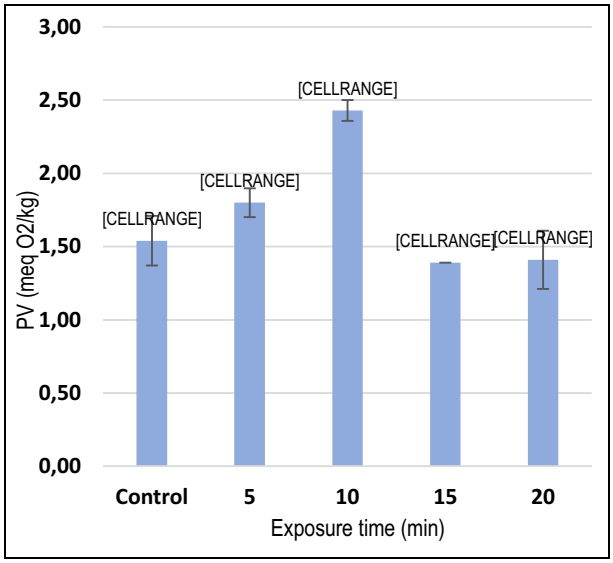

Figure 3 - Changes in PV of microwave-heated oils at different treatment times. Data are expressed as means ± SD. Means ± SD followed by the different letter, on bars are significantly different ($p < 0.05$).

fluctuations in PV with an increment of exposure time at 1200 W power.

Figure 4 shows the K_{232} and K_{270} of untreated and microwave-heated oils. K_{232} value of oils increased slightly until 15 min of heating. At the end of microwave heating (20 min), this value increased up to 4.70. Like for the K_{232} value, there was an increment for K_{270} value with an increase of exposure time. Hazelnut oil reached the highest K_{270} value, 0.60 at the end of 20 min of exposure to microwave heating. Arifoğlu and Öğütcü [37] reported a similar increase for the K_{232} and K_{270} values of hazelnut oils after 30 min of microwave heating. The increase in K_{232} may be due to the increased formation of CD. Besides, increment for K_{270} value could

TABLE IV - Changes in total phenolics and tocopherol composition of hazelnut oil during microwave heating

Exposure time (min)	Total phenolics (μg/g)	Tocopherol (mg/kg)			
		α	β	γ	δ
0	6.29±1.15a*	486.45±2.76a	27.86±1.28a	8.42±0.62a	3.61±0.13a
5	2.82±0.25d	418.15±2.75b	24.07±0.09b	7.16±0.10b	3.13±0.16b
10	4.25±0.15c	373.9±15.81cd	22.51±0.60c	6.74±0.19c	3.14±0.24b
15	4.99±0.52bc	383.43±8.52c	22.81±0.23c	6.74±0.12c	3.22±0.10b
20	5.69±0.77ab	360.53±4.28d	22.13±0.28c	6.68±0.12c	3.17±0.11b

*Data are expressed as means ± SD.

Means followed by different letters in the same column are significantly different ($p < 0.05$).

TABLE V - Changes in the fatty acid composition of hazelnut oil during microwave heating

Exposure time (min)	Fatty acid (%)									
	C14:0	C16:0	C16:1	C17:0	C17:1	C18:0	C18:1	C18:2	C18:3	C20:0	C20:1	C22:0
0	0.02±0.00	6.41±0.01	0.47±0.01	0.04±0.00	0.07±0.00	2.16±0.01b*	80.59±0.02c	9.87±0.01a	0.11±0.00	0.12±0.01	0.14±0.00	0.02±0.00
5	0.02±0.00	6.42±0.01	0.47±0.00	0.04±0.00	0.07±0.00	2.21±0.01a	80.91±0.01b	9.49±0.01b	0.11±0.00	0.11 ±0.00	0.14±0.00	0.02±0.00
10	0.02±0.00	6.39±0.04	0.47±0.00	0.04±0.00	0.07±0.00	2.20±0.01a	80.92±0.12ab	9.51±0.06b	0.11±0.00	0.11 ±0.00	0.14±0.00	0.02±0.00
15	0.02±0.00	6.40±0.00	0.47±0.00	0.04±0.00	0.07±0.00	2.21±0.01a	80.96±0.00ab	9.46 ±0.01b	0.11±0.00	0.12±0.01	0.14±0.00	0.02±0.00
20	0.02±0.00	6.40±0.05	0.47±0.00	0.04±0.00	0.07±0.00	2.20±0.01a	81.09±0.08a	9.34±0.01c	0.10±0.00	0.11 ±0.00	0.14±0.00	0.02±0.00

*Data are expressed as means ± SD.
Means followed by different letters in the same column are significantly different ($p < 0.05$).

Figure 4 - Changes in conjugated diene (K_{232}) and triene (K_{270}) values of microwave heated oils at different treatment times
Data are expressed as means ± SD. Significant differences in K_{232} ($p < 0.05$) among samples are shown by different lowercase letters (a c) and significant differences in K_{270} ($p < 0.05$) between samples are indicated by different capital letters (A-D).

be related to the formation of secondary oxidation products such as CT and unsaturated aldehydes or ketones due to their molecular friction in the microwave heating process [38].
Phenolic compounds decreased in hazelnut oils by increasing the microwave treatment time, as presented in Table IV. At the duration of 5 min, the TP of the hazelnut oils were decreased a minimum value of 2.82 µg/g. After that time, slight increases in phenolics were observed, not exceed the initial value of 6.29 µg/g. The fluctuation in the phenolic components, possibly due to the heating temperature, may influence individual phenolic compounds, and the other components have an antagonistic effect on the phenolics [39]. As for α-tocopherol, sharp degradation was observed during microwave heating. This result is consistent with Malheiro *et al*. [40], who reported that α-tocopherol decreased with microwave heating of virgin olive oil. Yoshida *et al*. [41] stated that tocopherols have different antioxidative activity, being α-tocopherol the most potent, followed by γ- then β- and δ-tocopherol, respectively. This report contributed to a lower loss of other tocopherol isomers compared to those of α-tocopherol. With higher exposition times in microwave heating, tocopherols decreased but did not disappear. A similar trend was observed in the study of Borges *et al*. [42], who reported that tocopherol isomers do not consume completely soybean oils microwave heated up to 15 min.
Fatty acid composition of oil samples after both micro-

wave heating is given in Table V. Linoleic acid showed a decreasing trend, while oleic acid increasing behaviour with an increment of exposure time. The results agree with those reported by Cossignani *et al*. [43]. They observed a decrease in linoleic and linolenic acid percentages and an increase in oleic acid when olive oils are microwave heated. In this study, the increase in oleic acid could be explained as an apparent increase in the percentage content.

CONCLUSION

Turkish hazelnut (*Corylus colurna*) has been grown as a wild hazelnut cultivar in Turkey. This cultivar is generally used in confectionery products. However, these hazelnuts are rich in oil and could be evaluated as a source of edible oil. Oxidation is a major problem in oils from this wild cultivar due to the high content of lipids. In this respect, oil samples were oxidized at two thermal conditions (oven and microwave heating). The results showed that conventional heating contributes to higher peroxide values and conjugated dienes than those of microwave-heated samples. The degradation of phenolics and tocopherol, which contribute to the oil's oxidative stability, were also evaluated, and the results were almost similar among all tested oils. Among tocopherols isomers, β-, γ- and δ-tocopherols were more stable than α-tocopherol during the oxidation of both treated hazelnut oils.

Compliance with ethical standards

Conflict of interest
None.

Compliance with ethics requirements
This article does not contain any studies with human or animal subjects.

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