

# Pecan walnut (*Carya illinoensis* (Wangenh.) K. Koch) oil quality and phenolic compounds as affected by microwave and conventional roasting

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**Abstract** In this study, the effects of conventional and microwave roasting on phenolic compounds, free acidity, peroxide value, fatty acid composition and tocopherol content of pecan walnut kernel and oil was investigated. The oil content of pecan kernels was 73.78% for microwave oven roasted at 720 W and 73.56% for conventional oven roasted at 110 °C. The highest free fatty acid content (0.50%) and the lowest peroxide value (2.48 meq O<sub>2</sub>/kg) were observed during microwave roasting at 720 W. The fatty acid profiles and tocopherol contents of pecan kernel oils did not show significant differences compared to raw samples. Roasting process in microwave oven at 720 W caused the reduction of some phenolic compounds, while the content of gallic acid exhibited a significant increase.

**Keywords** Pecan walnut · Kernel · Roasting · Microwave · Oil content · Phenolic compounds

## Introduction

Pecan walnut draws attention because of nutritional and functional contents such as phenolic compounds, tocopherols and unsaturated fatty acids. There are reports that pecan phenolic compounds, which have antioxidant activity, reduced the risk of cancer, Alzheimer, Parkinson and the other degenerative diseases (Mertens-Talcott and Percival 2005; Tam et al. 2006; Magnuson et al. 2016). Particular attention has been focused on the antioxidant capacity of nuts due to their content of vitamin E, selenium, and phenols (Anderson et al. 2001). Benefits of nuts in reducing the risk of heart disease has been reasonably attributed to their composition of vitamins, minerals, unsaturated fatty acids, fiber and phytochemicals such as polyphenols, tocopherols, squalene and phytosterols (Azadmard-Damirchi et al. 2011). Monounsaturated fatty acid content as oleic acid, which are determined highly in pecan, are less susceptible to lipid peroxidation (Vaidya and Eun 2013). Phenolic compounds of pecans, except for gallic acid, were the lower in whole roasted kernel. Roasting process caused the negative effects on individual phenolics but not on the total phenolic content and antioxidant activity of kernels (Yang et al. 2015). As the nuts can be consumed as raw or roasted form and the aim of the roasting process is to provide the desirable color, appearance, flavor and textural properties (Saklar et al. 1999). In addition, the reduction of moisture content and antinutritional components, prevention of microbial growth and enzyme activity and increase in the extraction yield are also observed during roasting process. However, heat processing has some disadvantages such as lipid modifications and detrimental effects on the nutritional compounds and natural antioxidants in foods (Arinola and Adesina 2014; Cang et al. 2016; Qu et al. 2016).

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Thermoanalytical techniques are commonly used to evaluate the stability of vegetable oils (Kowalski 1991; Wesolowski and Erecinska 1998) and have the advantage of providing information that conventional methods are incapable (Santos et al. 2004). Microwave energy has been tested in several drying studies (Silva and Marsaioli 2003; Berteli and Marsaioli 2005; Silva et al. 2006). The aim of this study was to investigate the effect of microwave (180, 360, 540 and 720 W) and conventional (70, 90, 110 and 130 °C) oven roasting on phenolic compounds and oil content, free acidity, peroxide value, fatty acid composition and tocopherol contents of pecan kernel and oils.

## Materials and methods

### Materials

About 5 kg pecan walnut (*Carya illinoensis*(Wangenh.) K. Koch.) samples were obtained from Tarsus (Mersin) district in Turkey. After the walnut kernels were separated from hulls, they were dried, and stored at 4 °C up to analysis.

### Methods

#### Roasting process

Pecan kernels were roasted in a conventional oven at 70, 90, 110 and 130 °C for 1 h; and in a microwave oven at 180, 360, 540 and 720 W for 5 min, respectively. The roasted samples were separately ground with a grinder before analysis.

#### Moisture content

Moisture content of pecan kernel was measured at  $100 \pm 5$  °C in an oven (Nüve FN055 Ankara, Turkey) until a constant weight was obtained.

#### Oil content

Oil content of kernel was determined according to AOAC (1990). Total oil content of pecan kernel was extracted by Soxhlet Apparatus for 5 h (with petroleum benzine), and the solvent was removed with a rotary vacuum evaporator at 50 °C. Oil was kept at the  $-18$  °C till analyses.

#### Free acidity and peroxide values

Free fatty acids, expressed as free oleic acid percentage, were determined using AOCS (1989). Peroxide values

(meq O<sub>2</sub>/kg) were determined according to AOCS method (1992).

#### Fatty acid composition

Fatty acid methyl esters esterificated according to ISO-5509 (1978) method were analysed by Gas Chromatography (Shimadzu GC-2010) equipped with flame-ionization detector (FID) and capillary column (Tecnocroma TR-CN100, 60 m × 0.25 mm, film thickness: 0.20 μm). The temperature of injection block and dedector was 260 °C. Mobile phase was nitrogen with 1.51 ml/min flow rate. Total flow rate was 80 ml/min and split rate was also 1/40. Column temperature was programmed 120 °C for 5 min and increased 240 °C at 4 °C/min and held 25 min at 240 °C (AOAC 1990).

#### Tocopherol content

Tocopherol contents of pecan oil were analyzed according to Spika et al. (2015). About 0.1 g of oil was dissolved in 10 ml of *n*-hexane, and filtered through a 0.45 μm nylon filter. HPLC analyses of tocopherols was accomplished using Shimadzu-HPLC equipped with PDA detector and LiChroCART Silica 60 (4.6 × 250 mm, 5 μ; Merck, Darmstadt, Germany) column. Tocopherols were separated by isocratic chromatography using a mobile phase of 0.7% propan-2-ol in *n*-hexane. The flow rate of the mobile phase was 0.9 ml/min, and the injection volume was 20 μl. The peaks were recorded at 295 and 330 nm with PDA detector. Standard solutions of tocopherols ( $\alpha$ ,  $\beta$ ,  $\gamma$  and  $\delta$ -tocopherol) were constructed in the concentrations of 0–100 mg/L. All analyses were made in triplicate.

#### Extraction of phenolic compounds

Phenolic compounds of pecan kernels were extracted according to Slatnar et al. (2015). About 2.5 g of dried and ground samples were added to 22.5 ml of mixture of methanol: water (60:40, v/v). The mixture was kept in ultrasonic water-bath for 1 h, followed by centrifugation at 6000 rpm for 10 min. The supernatant was filtered through a 0.45 μm nylon filter. All analyses were carried out in triplicate.

#### Phenolic compounds

Phenolic compounds of pecan kernels were determined by Shimadzu-HPLC equipped with PDA detector and Inertsil ODS-3 (5 μm; 4.6 × 250 mm) column. The mobile phase consisted of 0.05% acetic acid in water (A) and acetonitrile (B) mixture. The flow rate of the mobile phase and the injection volume were 1 ml/min at 30 °C and 20 μl,

respectively. The peak records were carried out at 280 and 330 nm.

### Statistical analyses

Analysis of variance (ANOVA) was performed by using JMP version 9.0 (SAS Inst. Inc., Cary, N.C.U.S.A). The analytical results were mean  $\pm$  standard deviation (MSTAT C) of independent kernel samples (Püskülcü and İkiz 1989).

## Results and discussion

### Moisture, oil content, acidity and peroxide values

The oil yield, moisture content, free acidity and peroxide values of pecan kernel and oils after microwave and conventional oven roasting are given in Table 1. oil yields of pecan roasted in conventional oven varied between 68.93 and 73.56%, oil yields of pecan roasted in microwave oven remained between 70.22 and 73.78%. The oil content of pecan kernels increased by microwave oven roasting at 720 W (73.78%) and conventional oven roasting at 110 °C (73.56%). Arinola and Adesina (2014) reported that the oil content of African walnut increased from 54.14% (raw nut) to 60.52% after roasting in hots for 1 h. Free acidity of pecan oils varied between 0.22 and 0.50%. The free acidity of pecan oil significantly increased by microwave roasting at 720 W, with the range of 0.50%. Conventional roasting at 70 °C did not effect the free fatty acid content when compared to raw pecan oil. But, the highest peroxide value was found for raw pecan oil (17.33 meq O<sub>2</sub>/kg), followed by pecan roasted in microwave oven at 180 W (10.05 meq O<sub>2</sub>/kg). It was observed that the roasting process, either in conventional oven or in microwave oven, resulted in peroxides degradation. The maximum degradation was determined when samples were roasted at

720 W, and the peroxide values decreased from 17.33 to 2.48 meq O<sub>2</sub>/kg. There were significant differences according to control group in moisture and oil contents, free acidity and peroxide values of pecan kernel and oils roasted in microwave and ordinary oven ( $p < 0.05$ ). According to the study of Vaidya and Eun (2013), peroxide value increased from 0.01 (unroasted walnut) to 2.34 meq/kg oil (roasted at 160 °C for 15 min). Qu et al. (2016) applied different drying methods such as direct oven drying and intermittent oven drying, and reported that the highest acid value was determined in sun dried walnuts. The peroxide values were observed as 1.94 meq/kg in direct oven dried samples and 1.82 meq/kg in oil of kernel dried in intermittent oven. The peroxide values of macadamia kernels increased from 0.54 meq/kg (raw sample) to 0.77 meq/kg roasted in microwave oven, and from 0.54 meq/kg (raw sample) to 1.13 meq/kg roasted in hot air (Silva et al. 2006).

### Fatty acid composition and tocopherol contents

The fatty acid compositions and tocopherol contents of pecan oils are shown in Table 2. The dominant fatty acids of pecan oil were oleic (59.14–61.87%), linoleic (26.31–28.67%), palmitic (6.39–7.19%) and stearic (2.51–3.52%) acids. Both conventional oven and microwave roasting processes affected the fatty acid profiles in pecan oils. There was significant differences between microwave and oven roasted pecan kernel oils in fatty acid compositions ( $p < 0.05$ ). The pecan oil contained only  $\gamma$ -tocopherol, and its content ranged from 3.09 to 4.65 mg/g.  $\alpha$ -,  $\beta$ - and  $\delta$ - tocopherols were not detected in any of the pecan oils. However, microwave roasting at 720 W caused a major  $\gamma$ -tocopherol decrease (from 4.65 to 3.09 mg/g). More  $\gamma$ -tocopherol reduction was observed in conventionally roasted (30 °C) oil which showed a decrease from 28.83 to 26.31%. Both roasting processing did not caused any important changes in tocopherol contents at different

**Table 1** Physico-chemical properties of pecans and pecan oils

Process	Moisture (%)	Oil content (%)	Free acidity (%)	Peroxide value (meq O <sub>2</sub> /kg)
Control	12.13 $\pm$ 0.01*a	68.93 $\pm$ 1.39f	0.22 $\pm$ 0.00d	17.33 $\pm$ 0.03a
70 °C	11.00 $\pm$ 0.02b**	69.11 $\pm$ 1.71e	0.22 $\pm$ 0.06d	7.54 $\pm$ 0.02d
90 °C	10.00 $\pm$ 0.02c	70.11 $\pm$ 1.34d	0.34 $\pm$ 0.03b	7.50 $\pm$ 0.02d
110 °C	10.00 $\pm$ 0.01c	73.56 $\pm$ 0.27a	0.33 $\pm$ 0.00bc	7.07 $\pm$ 0.02d
130 °C	11.00 $\pm$ 0.03b	72.56 $\pm$ 0.32b	0.34 $\pm$ 0.03b	8.96 $\pm$ 0.01c
180 W	11.00 $\pm$ 0.02b	71.56 $\pm$ 0.52c	0.34 $\pm$ 0.01b	10.05 $\pm$ 0.02b
360 W	11.00 $\pm$ 0.01b	73.44 $\pm$ 1.67a	0.28 $\pm$ 0.00c	4.41 $\pm$ 0.01e
540 W	10.00 $\pm$ 0.03c	70.22 $\pm$ 1.53d	0.28 $\pm$ 0.02c	4.75 $\pm$ 0.01e
720 W	11.00 $\pm$ 0.01b	73.78 $\pm$ 0.57a	0.50 $\pm$ 0.01a	2.48 $\pm$ 0.02f

\*Mean  $\pm$  standard deviation; \*\*Values within each column followed by different letters are significantly different ( $p < 0.05$ )

**Table 2** Fatty acid compositions (%) and tocopherol contents (mg/g) of pecan oils

Fatty acids	Oven							Microwave			
	Control	70 °C	90 °C	110 °C	130 °C	180 W	360 W	540 W	720 W		
Palmitic	7.19 ± 0.03 <sup>*a</sup>	6.67 ± 0.02b	6.39 ± 0.03b	7.02 ± 0.18a	7.06 ± 0.05a	6.82 ± 0.03b	6.58 ± 0.03b	6.55 ± 0.03b	7.08 ± 0.01a		
Stearic	2.66 ± 0.00b <sup>**</sup>	2.69 ± 0.00b	3.05 ± 0.00a	2.67 ± 0.02b	2.82 ± 0.01b	2.51 ± 0.01b	2.84 ± 0.04b	2.69 ± 0.00b	3.52 ± 0.00a		
Oleic	60.65 ± 0.01b	59.34 ± 0.00c	61.87 ± 0.03a	59.98 ± 0.22c	59.58 ± 0.16c	59.14 ± 0.01c	59.79 ± 0.01c	60.46 ± 0.01b	59.41 ± 0.03c		
Linoleic	28.83 ± 0.01a	28.67 ± 0.01a	27.92 ± 0.02b	27.75 ± 0.04b	26.31 ± 0.05c	28.17 ± 0.01a	27.02 ± 0.02b	27.71 ± 0.01b	27.49 ± 0.01b		
Arachidic	0.13 ± 0.00c	0.11 ± 0.00e	0.12 ± 0.00d	0.11 ± 0.00	0.14 ± 0.01b	0.11 ± 0.02e	0.12 ± 0.00d	0.12 ± 0.00d	0.19 ± 0.02a		
Linolenic	0.27 ± 0.00a	0.26 ± 0.01b	0.27 ± 0.01a	0.25 ± 0.00c	0.25 ± 0.02c	0.25 ± 0.03c	0.26 ± 0.01b	0.26 ± 0.00b	0.26 ± 0.01b		
γ-Tocopherol	4.65 ± 0.02 <sup>*ab</sup>	4.17 ± 0.02	4.29 ± 0.02b	4.55 ± 0.01b	4.08 ± 0.04b	3.44 ± 0.01c	3.50 ± 0.02c	3.82 ± 0.01c	3.09 ± 0.01c		

\*Mean ± standard deviation; \*\*Values within each column followed by different letters are significantly different ( $p < 0.05$ )

microwave powers and temperatures, however tocopherol contents exhibited significant differences in relation to control group ( $p < 0.05$ ). Vaidya and Eun (2013) informed that significant differences in fatty acid composition of raw and unroasted walnut oils did not observed. γ-Tocopherol was the main isomer of raw and unroasted walnut oils, and its content was decreased from 258.10 to 230.65 μg/g after roasting process (Vaidya and Eun 2013).

**Phenolic compounds**

The results about phenolic compounds are presented in Table 3 and it can be observed that the gallic acid, 3,4-dihydroxybenzoic acid, 1,2-Dihydroxybenzene, (+)-catechin, syringic acid, rutin and trans ferulic acid were the major phenolic compounds found in control and roasted samples. In addition to these phenolic compounds, minor amounts of caffeic acid, p-coumaric acid, apigenin 7 glucoside, resveratrol, quercetin, trans-cinnamic acid, kaempferol, isorhamnetin and naringenin were also detected. The amount of gallic acid content increased during roasting process, while 3,4-dihydroxybenzoic acid, 1,2-Dihydroxybenzene and syringic acid contents were decreased during heat treatment. The results demonstrated that the major decrease in (+)-catechin, 3,4-dihydroxybenzoic acid and 1,2-Dihydroxybenzene were observed when roasted in microwave oven at 720 W. Also, roasting in microwave oven at 540 W provided a major increase in gallic acid. It can be stated that the 3,4-Dihydroxybenzoic Acid, catechin, 1,2-Dihydroxybenzene, syringic, caffeic, trans-ferulic, quercetin, trans-Cinnamic Acid contents of the pecan kernels roasted either in microwave or oven exhibited significantly differences when compared to control group ( $p < 0.05$ ). Significant differences were not observed in case of apigenin 7 glucoside, trans-cinnamic acid, kaempferol contents of roasted at 360, 540 and 720 W. The main phenolic acid of raw pecan kernel was ellagic acid (2.96 mg/g), followed by chlorogenic acid and p-hydroxybenzoic acids (Yang et al. 2015). It can be observed that microwave roasting did not cast much effect on phenolic compounds as observe in case of conventional oven roasting.

**Conclusion**

The oil yields of pecan roasted in microwave oven were higher than those of oil contents of pecan kernel oil roasted in conventional oven. The roasting in both conventional and microwave resulted in peroxides degradation. There were significant differences between roasted and control samples for free acidity. The dominant fatty acids of pecan oil were oleic, linoleic, palmitic and stearic acids. Both

**Table 3** Phenolic compounds of raw and roasted pecan kernels (mg/100 g)

Phenolic compounds	Oven					Microwave				
	Control	70 °C	90 °C	110 °C	130 °C	180 W	360 W	540 W	720 W	
Gallic Acid	13.27 ± 2.48* <sup>g</sup>	32.23 ± 2.24b	11.84 ± 2.51g	27.67 ± 3.58c	25.62 ± 1.77d	20.25 ± 2.71e	16.59 ± 0.92f	34.88 ± 1.33a	24.27 ± 2.18d	
3,4-Dihydroxybenzoic Acid	32.71 ± 0.10a**	28.98 ± 0.53b	21.83 ± 2.07e	24.21 ± 1.38d	24.44 ± 2.19d	27.64 ± 0.24c	20.19 ± 1.11e	23.04 ± 1.70d	14.46 ± 0.64f	
(+)-Catechin	29.81 ± 2.44d	36.06 ± 2.21b	21.63 ± 0.98f	47.59 ± 2.39a	30.88 ± 2.61cd	25.73 ± 1.40e	24.18 ± 4.61e	36.70 ± 2.49b	18.27 ± 0.93g	
1,2-Dihydroxybenzene	28.77 ± 2.45a	27.56 ± 2.28b	26.07 ± 8.04bc	24.63 ± 4.08d	20.54 ± 0.31e	20.80 ± 2.56e	17.93 ± 0.74f	27.12 ± 2.05b	13.38 ± 1.72g	
Syringic Acid	10.76 ± 0.68a	7.59 ± 0.64d	7.83 ± 1.12d	6.58 ± 0.97e	8.61 ± 0.56c	9.26 ± 0.84b	7.45 ± 1.69d	8.33 ± 1.13c	7.44 ± 2.00d	
Caffeic Acid	6.27 ± 1.14c	8.48 ± 0.82a	7.73 ± 1.37b	7.36 ± 0.67b	7.77 ± 0.33b	7.65 ± 0.68b	7.35 ± 0.40b	8.18 ± 0.22a	7.50 ± 0.64b	
Rutin trihydrate	11.96 ± 1.86b	11.66 ± 1.38b	9.70 ± 0.96c	6.71 ± 0.47f	7.15 ± 0.19e	12.67 ± 0.61a	8.45 ± 0.88d	8.29 ± 0.61d	9.79 ± 2.71c	
<i>p</i> -Coumaric Acid	1.26 ± 0.37a	1.23 ± 0.17a	0.86 ± 0.03b	0.87 ± 0.22b	0.76 ± 0.06c	1.09 ± 0.15a	0.73 ± 0.03c	1.15 ± 0.20a	1.22 ± 0.12a	
<i>trans</i> -Ferulic Acid	11.54 ± 2.05a	7.62 ± 0.46c	8.44 ± 1.10d	9.23 ± 1.00c	4.50 ± 1.28f	9.21 ± 0.74c	8.80 ± 1.48d	10.34 ± 0.91b	9.44 ± 0.72c	
Apigenin 7 glucoside	5.24 ± 0.73c	6.19 ± 0.52b	5.96 ± 0.64c	6.39 ± 1.19b	5.64 ± 0.20c	8.50 ± 1.59a	5.86 ± 1.48c	5.69 ± 0.03c	5.65 ± 0.28c	
Resveratrol	2.47 ± 0.37b	3.11 ± 0.51a	2.31 ± 0.23b	1.58 ± 0.08c	1.59 ± 0.31c	1.96 ± 0.19b	1.99 ± 0.37b	1.21cd ± 0.12d	1.76 ± 0.34cd	
Quercetin	8.65 ± 1.82b	10.01 ± 0.45a	8.71 ± 1.18b	6.45 ± 0.74d	3.67 ± 0.72g	6.69 ± 0.66d	5.86 ± 0.92e	4.20 ± 0.44f	7.06 ± 0.84c	
<i>trans</i> -Cinnamic Acid	1.05 ± 0.12a	0.80 ± 0.06b	0.96 ± 0.13b	0.83 ± 0.16b	0.80 ± 0.16b	0.85 ± 0.07b	0.71 ± 0.08c	0.61 ± 0.21c	0.71 ± 0.09c	
Naringenin	1.08 ± 0.14d	1.88 ± 0.29b	1.28 ± 0.06d	1.51 ± 0.50c	1.02 ± 0.25d	2.05 ± 0.14a	2.10 ± 0.51a	1.03 ± 0.16d	1.80 ± 0.51b	
Kaempferol	4.90 ± 0.43b	5.21 ± 0.76a	5.12 ± 0.36a	4.49 ± 0.93b	2.68 ± 0.22d	4.32 ± 0.10b	3.05 ± 0.53c	3.17 ± 1.44c	3.60 ± 0.60c	
Isorhamnetin	2.94 ± 0.08c	3.52 ± 0.67b	3.24 ± 0.69b	2.23 ± 0.52c	2.73 ± 0.54c	4.55 ± 0.68a	2.03 ± 0.50c	1.67 ± 0.40d	2.65 ± 0.48c	

\*Mean ± standard deviation; \*\*Values within each column followed by different letters are significantly different ( $p < 0.05$ )

conventional oven and microwave roasting process affected the fatty acid profiles of oils in comparison to control samples. Both roasting processing did not caused any significant change in tocopherol content, but tocopherol content exhibited significant differences according to control group. It can be stated that some phenolic constituents (3,4-dihydroxybenzoic acid, catechin, 1,2-dihydroxybenzene, syringic, caffeic, trans-ferulic, quercetin, *trans*-cinnamic acid) of the pecan kernels roasted in microwave and conventional oven exhibited significant differences when compared to control group.

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