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ORIGINAL ARTICLE

Comparison of cold-pressing and soxhlet extraction systems for bioactive compounds, antioxidant properties, polyphenols, fatty acids and tocopherols in eight nut oils

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Abstract Antioxidant activities of different nut oils ranged from 11.43 (peanut) to 65.58% (pistachio) in cold pressed oils whereas in case of soxhlet extracted oils they were in the range of 11.32 (hazelnut) to 51.28% (pistachio). β-Carotene contents of oils obtained by cold pressing and soxhlet extraction changed between 7.53 (almond) and 13.58 µg/100 g (pistachio). The highest total phenol contents (2.36 mg gallic acid equivalent/100 g) were observed in pistachio oils obtained by cold press. The oleic acid contents of cold pressed and soxhlet extracted oils were between 19.88 (walnut) and 69.43% (pecan) to 19.07 (walnut) and 68.53% (pecan), respectively. The linoleic acid contents of nut oils from cold press system vary between 12.78 (hazelnut) and 63.56% (walnut), whereas in case of soxhlet extraction, it changed between 11.78 (hazelnut) and 62.41% (walnut). The α-tocopherol contents of cold pressed nut oils changed between 0.07 (walnut) and 257.42 mg/kg (hazelnut) α-tocopherol contents of nut oils extracted by soxhlet extraction changed between 0.03 (pistachio) and 209.73 mg/kg (hazelnut). The catechin contents of cold pressed nut oils were between 0.56 (cashew) and 3.76 µg/100 g (pistachio), whereas that of soxhlet extracted oil varied between 0.64 (cashew) and 3.82 µg/100 g (cashew).

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Keywords Nut oils · Cold press · Soxhlet extraction · Bioactive properties · Fatty acids · Tocopherols · Phenolic compounds

Introduction

Nuts are recommended constituents of the daily diet due to their high contents of proteins, oils, unsaturated fatty acids, vitamins and essential minerals (Kornsteiner et al. 2006). Tocopherols which are themajor lipophilic antioxidants, occur in plants in variable amounts and their biological and antioxidative activities vary among individual compounds (Saldeen and Saldeen 2005; Gliszczynska-Swiglo et al. 2007). Recently, oil seeds and/or nuts have been investigated, especially for minor phytochemicals (tocopherols, squalene and phenolic compounds) (Tuberoso et al. 2007). Nuts such as almond, hazelnut and pecan are not only important oil crops but also essential dietary components, acting as energy and functional compound sources (Fernandes et al. 2017). Phenolic compounds are very important for the oxidative stability of the polyunsaturated fatty acids of vegetable oils (Siger et al. 2008; Shinagawa et al. 2015). Three different methods are commonly used for the production of oils: pressing, soxhlet extraction and a combination of pre-pressing and solvent extraction (Özcan et al. 2013). The cold pressing procedure is being desired as it neither usesheat nor chemical treatments to obtain natural and safe edible oil products. The cold-pressed oils are important and cherished due to human health improvement and prevention of certain diseases (Goldberg 2003). Cold press which does not need much energy has some disadvantages such as low productivity and difficulties in obtaining a product of constant quality (Rotkiewicz et al. 1999). Plant oils contain certain amounts of fatty

acids, phenolic compounds, tocopherols and hydrocarbons (Lecker and Rodriguez-Estrada 2000). Caffeic acid contents of cold-pressed oils changed between 18 and 99 ppm caffeic acid equivalents (Bail et al. 2008). Cold pressed vegetable seed oils contain higher amounts of essential fatty acid and many others bioactive compounds (tocopherol, sterol, squalen) (Bail et al. 2008). Solvent/soxhlet extraction is the most widely used procedure on an industrial scale, although extraction with cold-pressing has been proposed. The information on comparative studies of nut oils obtained by both cold-pressing and solvent extraction systems is scarce. Hence, the aim of present study was to investigate bioactive properties, fatty acids, tocopherols and phenolic compounds of eight nut (almond, apricot, cashew, hazelnut, peanut, pistachio, pecan and walnut) oils obtained by cold pressing and soxhlet extraction systems.

Materials and methods

Materials

Nut samples (almond, apricot, cashew, hazelnut, peanut, pecan, pistachio and walnut) were obtained from local market in Turkey in 2017. After the kernels were separated from hulls and impurities, they were dried in shade, and stored at 4 °C till analysis. Each sample was analyzed as the whole kernel, without the shell.

Oil extraction using cold pressing

Once broken or damaged nuts and other impurities such as stem, skin etc. were removed, whole seeds were extracted with cold press (2–6 l/h capacity) without heat treatment. The impurities can negatively affect the quality of oil and once obtained by pressing, the oil was purified from solid impurities by sedimentation for 1 week followed by filtration. Purified oil was kept in hermetically closed colored bottle under nitrogen at + 4 °C.

Soxhlet assisted oil extraction

The nut kernels were extracted using a Soxhlet Apparatus with petroleum ether for 5 h. The solvent was removed with a rotary vacuum evaporator at 50 °C. The separated oil was kept in colored glass bottles at − 18 °C till analysis (AOAC 1990).

Carotenoid content

Extraction of carotenoids was performed according to Silva da Rocha et al. (2015). 25 ml Acetone was added to 2 g oil sample in beherglas. The mixture was shaken by vortex for 10 min and passed through filter paper (Whatman No. 1), followed by separation in funnel. The filtrate was fractionated with 20 ml of petroleum ether and washed with 100 ml of distilled water in order to remove acetone and process repeated twice. Whatman No. 1 covered with anhydrous sodium sulfate (5 g) for removing residual water was used to filter petroleum ether layer. The volume of the extracts was completed to 25 ml using petroleum ether. After these procedures, the absorbance was measured at 450 nm.

Determination of flavonoid

Once methanol extracts were diluted with distilled water, 5% $NaNO_2$ was added into each test tube. Then 10% $AlCl_3$ solution and 1.0 M NaOH were added. After 5 ml water was added, test tube was mixed well. Absorbance was measured at 510 nm versus blank. Results were expressed as mg catechol equivalents (CE) per g of dry weight (Dewanto et al. (2002).

Determination of anthocyanins

For anthocyanin analysis, 0.5 g oil was homogenized in a solution containing propanol, chlorhydric acid and water (18: 1: 81) and homogenate was boiled in a water bath for 3 min. Then homogenate was stored in dark for 24 h. Afterwards 3 ml supernatant was centrifuged at 6500 rpm for 40 min, absorbance were measured at 535 and 650 nm (Ticconi et al. 2001). The absorbance value was calculated and corrected by the following formula:

$$A = A_{535} - A_{650}.$$

Fatty acid composition

Nut oil samples were esterificated according to ISO-5509 (1978) method. A drop of the oil was dissoleved in 1 ml of *n*-heptane, 50 µg of sodium methylate was added, and the closed tube was agitated vigorously for 1 min at room temperature. After addition of 100 µl of water, the tube was centrifuged at 4500*g* for 10 min and the lower aqueous phase was removed. Then 50 µl of HCl (1 mol with methyl orange) was added, the solution was shortly mixed, and the lower aqueous phase was rejected. Fatty acid methyl esters of oils were analyzed using 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 detector was 260 °C. Mobile phase was nitrogen with 1.51 ml/min flow rate. Total flow rate and split rate were 80 ml/min and 1/40, respectively.

Column temperature was programmed 120 °C for 5 min and increased 240 °C at 4 °C/min and held 25 min at 240 °C. The peak areas were computed by the integration software, and percentages of fatty acid methyl esters (FAME) were obtained as weight percent by direct internal normalization.

Tocopherol contents

20 µl of a solution of 250 mg of oil in 25 ml of *n*-heptane was directly injected to a Diol phase HPLC column 25 cm × 4.6 mm ID (Merck, Darmstadt, Germany) used with a flow rate of 1.3 ml/min. Tocopherol content was determined according to Balz et al. (1992). Tocopherols were determined by Shimadzu-HPLC equipped with PDA detector and LiChroCART Silica 60 (4.6 × 250 mm, 5 µ; Merck, Darmstadt, Germany) column. The standard solutions of α, β, γ and δ-tocopherols were prepared in the concentrations of 0–100 mg/l. The mobile phase used was *n*-heptane/*tert*-butyl methyl ether (99 + 1, v/v). All analyses were made in triplicate.

Extraction of nut oils for phenolics

The nut oils were extracted according to Slatnar et al. (2015). In order to analyze total phenolics, approximately 5 g of oil sample was added to 15 ml of methanol. The mixture was kept in ultrasonic water-bath for 1 h, followed by centrifugation at 6000 rpm for 10 min (Hermle, Germany), and then the supernatant was filtered through a 0.45 µm membrane filter. 10 ml of *n*-hexane was added and mixed using a vortex apparatus. The methanol and hexane layer were separated in separating funnel. This step was carried out twice with 10 ml of *n*-hexane. After the methanol phase was collected in each step, and then evaporated at 50 °C, it was dissolved in 1.5 ml of methanol.

Total phenolic content

Total phenol contents of samples were determined by using the Folin–Ciocalteu (FC) reagent according to Yoo et al. (2004). Folin–Ciocalteu (1 ml) was added to sample and solution was mixed for five minutes. Afterwards, 10 ml of 7.5% Na_2CO_3 was added into solution tubes, mixed and the final volume was completed to 25 ml with distilled water. Absorbance for total phenolic content was measured at 750 nm in a spectrophotometer (Shimadzu UV–Vis spectrophotometer, UV mini 1240, Japan) against gallic acid (0–200 mg/ml) as the standard for calibration curve. The results were expressed as mg of gallic acid equivalents (mg GAE/100 g).

Antioxidant activity

The antioxidant activity of oil samples were determined by using DPPH (1,1-diphenyl-2-picrylhydrazyl) according to Lee et al. (1998). After the extract was mixed with 2 ml methanolic solution of DPPH, the mixture was shaken vigorously and allowed to stand at room temperature for 30 min. The absorbance was measured at 517 nm in a spectrophotometer (Shimadzu UV–Vis spectrophotometer, UV mini 1240). The antioxidant activity was expressed as % in dry matter (dw). Inhibition (%) was determined according to formula given below.

$$\text{Inhibition}\,(\%) = \left[\frac{\Delta A\ Control\,517 - \Delta A\ Extract\,517}{\Delta A\ Control\,517}\right] \times 100$$

Phenolic compounds

Phenolic compounds of oil extracts were carried out using Shimadzu-HPLC equipped with PDA detector and Inertsil ODS-3 (5 µm; 4.6 × 250 mm) column. Elution was carried by using a gradient procedure with a mobile phase containing solvent A and solvent B. The 0.05% acetic acid in water (A) and acetonitrile (B) mixture of mobile phase were used. The flow rate of mobile phase was set at 1.0 ml/min for a total run time of 72 min at 30 °C., and a 20 µL sample volume was injected. The peaks were recorded at 280 and 330 nm with PDA detector. The gradient program was as follows: 0–0.10 min 8% B; 0.10–2 min 10% B; 2–27 min 30% B; 27–37 min 56% B; 37–37.10 min 8% B; 37.10–45 min 8% B. The total running time per sample was 60 min. Phenolic compounds were determined according to the retention time and absorption spectra of peaks of standard compounds. The total area under peak was used to quantify the each phenolics.

Gradient time programme

Time (min)	%AcCN
0	5
8	5
15	13
30	13
40	18
50	30
55	80
65	80
68	5
72	5

Statistical analyses

The analysis of variance (ANOVA) was performed by using JMP version 9.0 (SAS Inst. Inc., Cary, N.C.U.S.A). All analyses were carried out three times and the results are mean ± SD (MSTAT C) of independent nut oils (Püskülcü and İkiz 1989).

Results and discussion

Effects of cold pressing and soxhlet extraction on bioactive compounds of nut oils

The β-carotene, total phenol, antioxidant activity, flavonoid, carotenoid and anthocyanin contents of eight nut (almond, apricot, cashew, hazelnut, peanut, pistachio, pecan and walnut) oils obtained by cold press and soxhlet extraction are given in Table 1. The total phenolic and antioxidant activity values of all nut oils extracted by cold pressing were higher while compared to those of oil samples obtained by soxhlet extraction. However, other parameters (β-carotene, flavonoid, carotenoid and anthocyanin contents) values of oils obtained by soxhlet extraction were higher. β-carotene contents of cold pressed nut oils ranged between 7.53 (almond) and 13.58 μg/100 g (pistachio). In oils extracted by soxhlet extraction, β-carotene contents ranged from 8.13 (almond) to 15.64 μg/100 g (pistachio). Peanut oil extracted by soxhlet extraction showed the lowest level of total antioxidant activity with only 9.63%. The highest total phenol content (2.36 mg GAE/100 g) was determined in pistachio oils obtained by cold press. The antioxidant activities of cold pressed nut oils ranged between 11.43 (peanut) and 65.58% (pistachio), whereas that of nut oils obtained by soxhlet extraction ranged from 11.32 (hazelnut) to 51.28% (pistachio). Anthocyanin and carotenoid contents of the nut oils obtained by both techniques were less than 1.0 (except for pistachio oils' carotenoid contents). In another study (Wu et al. (2004), the mean values of total phenolics changed between 0.68 (pines) and 20.16 mg GAE/g (pecans) and they also found a high total antioxidant capacity in pecans (179 μmol TAC/g), pistachios (80 μmol TAC/g) and walnuts (135 μmol TAC/g). Kornsteiner et al. (2006) reported that the mean value of total phenolics changed between 32 mg GAE/100 g and 1625 mg GAE/100 g in different nut oils. In addition, the cold-pressed olive and rapeseed oils contained 4 ppm total phenolic. Siger et al. (2008) studied total phenolic contents of several plant oils, and they determined 1.48, 1.31, 1.20, 1.26, 0.51 and 2.46 mg CAE/100 g total phenol in soybean, rapeseed, sunflower, corn and pumpkin oils, respectively. The hemp oil contained 0.44 mg GAE/g total phenolic (Yu et al. (2005). In other study, the total phenolic contents changed between 0.98 and 3.35 mg GAE/g of cold pressed oils of onion, parsley, cardamom, mullein, roasted pumpkin and milk thistle (Parry et al. 2006). The phenols extracted from sunflower seeds while obtaining cold-pressed oil, protect the oil from autoxidation more effectively than butylated hydroxyanisole due to their antioxidant potential (De Leonardis et al. 2003). In previous study on olive oils, methanol-soluble phases from maize and sunflower oils had an antioxidant activity much lower than methanol-soluble phase of olive oils (Papadopoulos et al. (2003). Some of results showed similarity with literature values. Differences can be probably due to process conditions, solvent types and chemical structure of oil and the matrix from where it is obtained. Generally, the better contents of phenolics and higher antioxidant activity of cold pressed nut oils make them superior to those obtained by solvent/soxhlet system.

Effects of cold pressing and soxhlet extraction on individual phenolics of nut oils

The data about individual phenolic compounds of almond, apricot, cashew, hazelnut, peanut, pistachio, pecan and walnut oils obtained by cold press and soxhlet extraction systems is presented in Table 2. The catechin contents of cold pressed nut oils were in the range of 0.56 (cashew) and 3.76 μg/100 g (pistachio), whereas those of nut oil samples extracted by soxhlet extraction varied between 0.64 (cashew) and 3.82 μg/100 g (cashew). In addition, naringenin contents of cold pressed nut oils vary between 0.54 (cashew) and 3.86 μg/100 g (walnut), naringenin contents of soxhlet extraction nut oils changed between 0.63 (cashew) and 3.94 μg/100 g (walnut). The chlorogenic acid contents of cold pressed oils were determined between 0.32 (cashew) and 1.45 μg/100 g (pistachio). The chlorogenic acid contents of oils from solvent extraction ranged between 0.39 (cashew) and 1.49 μg/100 g (almond). The highest luteolin contents of cold pressed and soxhlet extraction nut oils were found in pistachio oil samples as 1.17 and 1.24 μg/100 g, respectively. The highest gallic acid was found in almond oil (1.13 μg/100 g) extracted by soxhlet extraction. In addition, the highest caffeic acid was found in pistachio oils as 1.13 and 1.28 μg/100 g depending on extraction system, respectively (Table 4). Siger et al. (2008) studied the phenolic compounds of some plant oil extracts and determined 0.8 (soybean oil) to 6.0 μg/100 g (hemp oil) *p*-hydroxybenzoic, 1.0 (flax) to 6.9 (sunflower) μg/100 g vanillic, 0.3 (rapeseed) to 4.9 (sunflower) μg/100 g caffeic, 1.5 (soybean) to 13.1 (rapeseed) μg/100 gp-coumaric, 0.4 (rice bran) to 5.8 (corn) μg/100 g ferulic, 0.2 (grape seed) to 236.0 (rape seed) μg/100 g

Table 1 Bioproperties of nut oils obtained by cold press and soxhlet extraction

Bioproperties	Cold pressed oils							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
β-Carotene (μg/100 g)	7.53 ± 0.67*g	8.61 ± 0.48f	12.47 ± 1.17b	11.89 ± 1.29c	10.47 ± 1.36d	9.61 ± 0.87e	13.58 ± 1.43a	10.62 ± 1.27d
Total phenol (mg GAE/100 g)	1.56 ± 0.15b**	1.29 ± 0.13c	0.98 ± 0.21d	0.78 ± 0.09e	0.84 ± 0.14d	1.32 ± 0.11b	2.36 ± 0.22a	1.13 ± 0.17b
Antioxidant activity (%)	27.81 ± 1.32c	13.56 ± 1.27f	11.43 ± 0.98g	17.58 ± 0.45d	14.61 ± 1.43e	32.45 ± 1.56b	67.58 ± 2.37a	17.43 ± 2.53d
Flavonoid (mg/100 g)	1.13 ± 0.07d	1.21 ± 0.34c	0.09 ± 0.03e	5.27 ± 0.47a	3.89 ± 0.58b	1.36 ± 0.21c	1.17 ± 0.19d	0.97 ± 0.07d
Carotenoid (mg/100 g)	0.68 ± 0.09c	0.56 ± 0.03d	0.42 ± 0.07e	0.53 ± 0.11d	0.76 ± 0.14b	0.57 ± 0.19d	1.18 ± 0.21a	0.26 ± 0.03f
Anthocyanin (mg/100 g)	0.09 ± 0.01de	0.07 ± 0.01e	0.13 ± 0.03c	0.21 ± 0.07b	0.19 ± 0.03c	0.11 ± 0.03d	0.36 ± 0.09a	0.05 ± 0.01e
Bioproperties	Soxhlet extraction oils							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
β-Carotene (μg/100 g)	8.13 ± 1.15f	9.27 ± 0.56e	13.84 ± 0.61b	13.72 ± 2.34b	11.61 ± 1.32d	9.94 ± 0.65e	15.64 ± 1.57a	12.49 ± 1.38c
Total phenol (mg GAE/100 g)	1.13 ± 0.07b	0.95 ± 0.03c	0.57 ± 0.09e	0.45 ± 0.11e	0.77 ± 0.13d	0.88 ± 0.15c	1.34 ± 0.21a	1.03 ± 0.18b
Antioxidant activity (%)	18.48 ± 1.45c	11.32 ± 1.29f	9.63 ± 0.67g	13.44 ± 0.71d	12.39 ± 1.28	19.86 ± 1.13b	51.28 ± 2.43a	12.27 ± 1.61e
Flavonoid (mg/100 g)	1.49 ± 0.11d	1.87 ± 0.23d	0.61 ± 0.07e	6.37 ± 0.23a	4.89 ± 0.46b	2.43 ± 0.25c	2.81 ± 0.17c	1.66 ± 0.13d
Carotenoid (mg/100 g)	0.76 ± 0.09c	0.69 ± 0.03d	0.73 ± 0.07c	0.88 ± 0.09b	0.97 ± 0.11b	0.81 ± 0.07b	1.97 ± 0.13a	0.65 ± 0.07d
Anthocyanin (mg/100 g)	0.21 ± 0.03d	0.33 ± 0.05b	0.19 ± 0.01e	0.38 ± 0.07b	0.27 ± 0.03c	0.16 ± 0.01e	0.69 ± 0.09a	0.18 ± 0.03e

*Mean ± SD; **values within each row followed by different letters are significantly different ($p < 0.05$)

Table 2 Phenolic compounds of nut oils obtained by cold press and soxhlet extraction (μg/100 g)

Phenolics	Cold pressed oils							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
Gallic	0.89 ± 0.07*a	0.21 ± 0.03e	0.18 ± 0.01e	0.47 ± 0.09d	0.36 ± 0.05d	0.51 ± 0.11c	0.68 ± 0.13b	0.13 ± 0.01f
Protocatechuic	0.24 ± 0.03f**	0.47 ± 0.12c	0.32 ± 0.07e	0.53 ± 0.03b	0.42 ± 0.01d	0.32 ± 0.01e	0.67 ± 0.09a	0.28 ± 0.03f
Catechin	1.17 ± 0.15d	0.89 ± 0.13f	1.34 ± 0.11d	2.38 ± 0.23b	1.67 ± 0.21c	1.26 ± 0.19e	3.76 ± 0.27a	0.56 ± 0.03f
Caffeic	0.43 ± 0.01c	0.67 ± 0.05b	0.21 ± 0.0d	0.06 ± 0.01e	0.18 ± 0.03d	0.15 ± 0.0d1	1.13 ± 0.09a	0.09 ± 0.01e
Ferulic	0.27 ± 0.03c	0.38 ± 0.07b	0.09 ± 0.01e	0.43 ± 0.03a	0.23 ± 0.01d	0.31 ± 0.07c	0.13 ± 0.01e	0.16 ± 0.03d
Sinapic	0.73 ± 0.07b	0.42 ± 0.09d	0.11 ± 0.01g	0.38 ± 0.05e	0.41 ± 0.03d	0.68 ± 0.03c	0.83 ± 0.11a	0.23 ± 0.01f
Naringenin	1.38 ± 0.13c	1.13 ± 0.09c	0.98 ± 0.07d	2.17 ± 0.15b	3.86 ± 0.21a	0.77 ± 0.09e	2.75 ± 0.13b	0.54 ± 0.03ef
Chlorogenic	1.41 ± 0.11a	0.65 ± 0.04d	0.74 ± 0.03c	0.53 ± 0.01e	0.61 ± 0.07d	1.07 ± 0.03b	1.45 ± 0.09a	0.32 ± 0.01f
p-coumaric	0.19 ± 0.03c	0.17 ± 0.01d	0.15 ± 0.03e	0.11 ± 0.01g	0.22 ± 0.01b	0.13 ± 0.01f	0.34 ± 0.07a	0.19 ± 0.03c
Rutin	0.15 ± 0.01b	0.09 ± 0.01d	0.05 ± 0.01f	0.07 ± 0.01e	0.09 ± 0.01d	0.11 ± 0.01d	0.17 ± 0.03a	0.14 ± 0.01c
Resveratrol	0.37 ± 0.03a	0.05 ± 0.01f	0.09 ± 0.01d	0.03 ± 0.01g	0.07 ± 0.01e	0.29 ± 0.03c	0.31 ± 0.07b	0.09 ± 0.01d
Vanillic	0.17 ± 0.03d	0.03 ± 0.01g	0.05 ± 0.01f	0.11 ± 0.01e	0.18 ± 0.03c	0.23 ± 0.01b	0.34 ± 0.01a	0.16 ± 0.01de
Kampferol	0.32 ± 0.01b	0.21 ± 0.03d	0.13 ± 0.01f	0.15 ± 0.01e	0.21 ± 0.03d	0.28 ± 0.03c	0.45 ± 0.07a	0.11 ± 0.01g
Quercetin	0.07 ± 0.01f	0.03 ± 0.01g	0.12 ± 0.01d	0.17 ± 0.01c	0.27 ± 0.03b	0.09 ± 0.01e	0.29 ± 0.03a	0.03 ± 0.01g
Luteolin	0.78 ± 0.03d	0.97 ± 0.07b	0.68 ± 0.05e	0.81 ± 0.07c	0.55 ± 0.03g	0.65 ± 0.03f	1.17 ± 0.03a	0.21 ± 0.01h
Pinocembrin	0.03 ± 0.01h	0.05 ± 0.01g	0.07 ± 0.02f	0.12 ± 0.03d	0.16 ± 0.03c	0.09 ± 0.01e	0.21 ± 0.1a	0.18 ± 0.03b
Phenolics	Soxhlet extraction oils							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
Gallic	1.13 ± 0.17a	0.38 ± 0.03f	0.27 ± 0.01g	0.55 ± 0.09d	0.43 ± 0.01e	0.59 ± 0.11c	0.73 ± 0.09b	0.19 ± 0.01h
Protocatechuic	0.35 ± 0.04f	0.59 ± 0.07c	0.47 ± 0.03d	0.62 ± 0.09b	0.58 ± 0.07c	0.41 ± 0.03e	0.74 ± 0.07a	0.30 ± 0.03g
Catechin	1.26 ± 0.117	1.09 ± 0.21	1.47 ± 0.23	2.51 ± 0.29	1.78 ± 0.09	1.33 ± 0.05	3.82 ± 0.26	0.64 ± 0.07
Caffeic	0.52 ± 0.03c	0.78 ± 0.09b	0.34 ± 0.03d	0.14 ± 0.01h	0.27 ± 0.01e	0.23 ± 0.03f	1.28 ± 0.14a	0.17 ± 0.03g
Ferulic	0.32 ± 0.03de	0.45 ± 0.09b	0.18 ± 0.01g	0.51 ± 0.03a	0.34 ± 0.01d	0.39 ± 0.03c	0.22 ± 0.01f	0.21 ± 0.01f
Sinapic	0.84 ± 0.07b	0.51 ± 0.03d	0.19 ± 0.01g	0.43 ± 0.03e	0.41 ± 0.03e	0.77 ± 0.09c	0.89 ± 0.05a	0.34 ± 0.01f
Naringenin	1.44 ± 0.21d	1.22 ± 0.07e	1.09 ± 0.05f	2.35 ± 0.18c	3.94 ± 0.23a	0.78 ± 0.09g	2.87 ± 0.27b	0.63 ± 0.09h
Chlorogenic	1.49 ± 0.13a	0.71 ± 0.09e	0.83 ± 0.03d	0.59 ± 0.05g	0.67 ± 0.01f	1.23 ± 0.09c	1.58 ± 0.13b	0.39 ± 0.03h
p-coumaric	0.24 ± 0.03c	0.28 ± 0.05b	0.21 ± 0.03d	0.19 ± 0.03e	0.27 ± 0.09bc	0.17 ± 0.01f	0.40 ± 0.03a	0.28 ± 0.01b
Rutin	023 ± 0.01a	0.16 ± 0.03d	0.11 ± 0.01e	0.19 ± 0.03c	0.21 ± 0.01b	0.15 ± 0.01de	0.23 ± 0.03a	0.21 ± 0.03b
Resveratrol	0.42 ± 0.09a	0.11 ± 0.01g	0.17 ± 0.03e	0.19 ± 0.01d	0.17 ± 0.03e	0.36 ± 0.07c	0.39 ± 0.03b	0.15 ± 0.01f
Vanillic	0.24 ± 0.03d	0.28 ± 0.01c	0.13 ± 0.01f	0.23 ± 0.03e	0.29 ± 0.01c	0.34 ± 0.07b	0.42 ± 0.03a	0.28 ± 0.01c
Kampferol	0.41 ± 0.03b	0.37 ± 0.09c	0.21 ± 0.03f	0.19 ± 0.01g	0.28 ± 0.05e	0.34 ± 0.0d	0.51 ± 0.03a	0.19 ± 0.03g
Quercetin	0.11 ± 0.01h	0.13 ± 0.01g	0.19 ± 0.01d	0.23 ± 0.01c	0.38 ± 0.07b	0.17 ± 0.03e	0.43 ± 0.09a	0.15 ± 0.01f
Luteolin	0.89 ± 0.7d	1.15 ± 0.13b	0.81 ± 0.03e	0.98 ± 0.09c	0.76 ± 0.03f	0.73 ± 0.05g	1.24 ± 0.11a	0.36 ± 0.03h

Table 2 continued

Phenolics	Soxhlet extraction oils							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
Pinocembrin	0.09 ± 0.01g	0.13 ± 0.03f	0.17 ± 0.01e	0.24 ± 0.03c	0.28 ± 0.06b	0.19 ± 0.03d	0.36 ± 0.07a	0.27 ± 0.01bc

*Mean ± SD; **values within each row followed by different letters are significantly different ($p < 0.05$)

sinapic acids in oil samples. In a previous study, the presence of *p*-coumaric, ferulic and sinapic acids in corn oil was determined (Niwa et al. 2001). The ferulic acid contents of flaxseed oil changed between 130 and 220 mg/100 g (Wanasundara and Shahidi 1994). Choo et al. (2007) reported that cold-pressed flaxseed oils contained 76.8–307.3 mg/100 g total phenolic acid. In other study, Tuberoso et al. (2007) determined 2.8 vanillin, 0.9 *trans*-cinnamic acid and 0.5 mg/kg ferulic acid in maize seed oil, and 6.8 mg/kg syringic acid in rapeseed oil. Depending on nut species, phenolic compounds of nut oils showed several differences. Generally, the phenolic compounds of nut oil samples obtained by cold pressing were found to be somewhat lower than those of phenolic compounds of nut oils extracted by solvent extraction system with some exceptions. Catechin and naringenin were the major phenolic constituents of nut oils extracted either by cold press and soxhlet extraction systems.

Effects of cold pressing and soxhlet extraction on fatty acids of nut oils

Fatty acid profiles of almond, apricot, cashew, hazelnut, peanut, pistachio, pecan and walnut nut oils extracted by cold press and soxhlet extraction using petroleum ether are shown in Table 3. The palmitic acid contents of cold pressed nut oils ranged between 4.87 (almond) and 9.45% (peanut), whereas those of soxhlet extracted nut oils ranged between 4.13 (almond) and 8.97% (peanut). Similarly, oleic acid contents of cold pressed oils were found between 19.88 (walnut) and 69.43% (pecan) while those of oils obtained by soxhlet extraction were minimum 19.07 (walnut) and maximum 68.53% (pecan). The linoleic acid contents of cold pressed oils varied between 12.78 (hazelnut) and 63.56% (walnut) whereas those of soxhlet extracted oils changed between 11.78 (hazelnut) and 62.41% (walnut). The highest stearic acid (7.69%) was found in cashew oil obtained by cold press. Other fatty acids analyzed in nut oils obtained by two extraction systems were found to be in minor amounts (Table 3). The walnut oil was rich in linoleic acids and other oils higher amount of oleic acid as seen in Table 3. Walnut oil contained the highest level of linoleic acid (63.56%) and the lowest linoleic acid contents were observed in hazelnut oil obtained by soxhlet extraction. Oils extracted from *Juglans regia* showed fatty acid profile, especially linoleic (61%), oleic (15%) and α-linolenic (12%) acids. The major fatty acids found in *Carya illinoensis* oil were oleic, linoleic, and palmitic acids with 45, 43, and 7%, respectively (Colic et al. 2015). They observed fatty acid profiles of almond kernel oils grown in Serbia as 0.01–0.10% myristic, 4.68–6.48% palmitic, 0.24–0.56% palmitoleic, 1.45–2.56% stearic and 15.57–27.72% linoleic acids. Kırbaşlar et al.

Table 3 Fatty acid composition of nut oils obtained by cold press and soxhlet extraction (%)

Fatty acids	Cold pressed oils							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
Myristic	0.04 ± 0.01*e	0.05 ± 0.02d	0.04 ± 0.01e	0.03 ± 0.01f	0.05 ± 0.01c	0.05 ± 0.03c	0.13 ± 0.03a	0.06 ± 0.01b
Palmitic	4.87 ± 0.17e**	6.87 ± 0.21c	9.45 ± 0.46a	5.36 ± 0.62d	6.84 ± 0.84c	5.13 ± 0.49d	8.57 ± 0.87b	8.16 ± 0.93b
Palmitoleic	0.38 ± 0.03b	0.21 ± 0.05c	0.18 ± 0.01d	0.23 ± 0.01c	0.13 ± 0.01e	0.21 ± 0.03c	0.61 ± 0.07a	0.17 ± 0.01d
Stearic	1.17 ± 0.17c	2.97 ± 0.09b	2.23 ± 0.07b	1.49 ± 0.03c	2.78 ± 0.21b	1.34 ± 0.15c	1.32 ± 0.23c	7.69 ± 0.09a
Oleic	61.56 ± 1.21c	62.74 ± 1.28b	41.68 ± 1.13e	69.43 ± 0.45a	19.88 ± 0.57	62.57 ± 0.74b	54.89 ± 0.68d	58.94 ± 0.36d
Linoleic	21.56 ± 0.34c	12.78 ± 0.27e	28.43 ± 0.13b	19.32 ± 0.41d	63.56 ± 0.68a	18.44 ± 0.81d	28.61 ± 0.48b	19.65 ± 1.07d
Linolenic	0.05 ± 0.01e	0.09 ± 0.03e	1.27 ± 0.14a	0.68 ± 0.09b	0.11 ± 0.01d	0.05 ± 0.01e	0.21 ± 0.03c	0.13 ± 0.01d
Arachidic	0.11 ± 0.03c	0.04 ± 0.01d	0.91 ± 0.07a	0.04 ± 0.01d	0.05 ± 0.03d	0.05 ± 0.01d	0.07 ± 0.01d	0.62 ± 0.09b
Behenic	0.09 ± 0.02e	0.04 ± 0.01e	0.56 ± 0.09a	0.23 ± 0.01c	0.31 ± 0.03b	0.07 ± 0.01e	0.14 ± 0.03d	0.13 ± 0.01d
Arachidonic	0.17 ± 0.03b	0.09 ± 0.01d	0.57 ± 0.09a	0.07 ± 0.03d	0.13 ± 0.01c	0.11 ± 0.03c	0.05 ± 0.01d	0.10 ± 0.03b
Fatty acids	Soxhlet extraction							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
Myristic	0.03 ± 0.01b	0.04 ± 0.01b	0.02 ± 0.01b	0.02 ± 0.01b	0.03 ± 0.01b	0.03 ± 0.01b	0.09 ± 0.03a	0.03 ± 0.01b
Palmitic	4.13 ± 0.23d	6.21 ± 0.58c	8.97 ± 0.79a	4.83 ± 0.43d	6.17 ± 0.32c	4.91 ± 0.48d	7.56 ± 0.27b	7.69 ± 0.13b
Palmitoleic	0.27 ± 0.03b	0.18 ± 0.01c	0.13 ± 0.03d	0.17 ± 0.03c	0.09 ± 0.01	0.15 ± 0.03d	0.52 ± 0.05a	0.11 ± 0.01e
Stearic	1.03 ± 0.07d	2.11 ± 0.25b	1.98 ± 0.17c	0.96 ± 0.11e	2.09 ± 0.14b	0.91 ± 0.09e	1.03 ± 0.09d	7.23 ± 0.21a
Oleic	60.13 ± 0.72c	61.27 ± 1.28b	41.14 ± 0.35f	68.53 ± 1.42a	19.07 ± 0.48g	61.49 ± 1.27b	53.76 ± 0.98e	57.43 ± 0.86d
Linoleic	20.17 ± 0.77c	11.78 ± 0.59g	27.65 ± 1.36b	18.46 ± 0.61e	62.41 ± 1.27a	17.81 ± 0.34f	27.86 ± 0.67b	19.12 ± 0.48d
Linolenic	0.03 ± 0.01f	0.04 ± 0.01f	1.67 ± 0.17a	1.45 ± 0.21b	0.57 ± 0.09c	0.49 ± 0.03d	0.36 ± 0.03e	0.43 ± 0.07d
Arachidic	0.07 ± 0.01b	0.03 ± 0.01b	0.35 ± 0.07a	0.03 ± 0.01b	0.03 ± 0.01b	0.04 ± 0.01b	0.05 ± 0.01b	0.39 ± 0.03a
Behenic	0.06 ± 0.01d	0.03 ± 0.01e	0.17 ± 0.03b	0.14 ± 0.01b	0.25 ± 0.07a	0.03 ± 0.01e	0.11 ± 0.01c	0.09 ± 0.01c
Arachidonic	0.11 ± 0.01b	0.05 ± 0.01c	0.45 ± 0.03a	0.05 ± 0.01c	0.13 ± 0.03b	0.09 ± 0.01bc	0.03 ± 0.01c	0.07 ± 0.03c

*Mean ± SD; **values within each row followed by different letters are significantly different ($p < 0.05$)

(2012) reported 7.36, 9.48, 5.39, and 7.18% palmitic, 63.21, 55.41, 67.18, 71.98 and 13.55% oleic and 13.64, 26.51, 20.53, 20.37 and 63.42% linoleic acids in hazelnut, peanut, pistachio, almond, and walnut kernel oils, respectively. Fernandes et al. (2017) reported that almond, hazelnut and pecan kernel oils contained 6.62, 5.55 and 5.57% palmitic, 1.33, 1.85 and 2.58% stearic, 59.70, 78.34 and 70.96% oleic, and 29.54, 11.53 and 17.97% linoleic acids, respectively. Palmitic, stearic, oleic and linoleic acids were the key fatty acids of cold pressed and soxhlet extracted nut oils in the current study. In general, the fatty acid contents of nut oils obtained by soxhlet extraction were somewhat lower than those of cold pressed oil. This reduction can be attributed to the possible impurities in oil extracted with solvent.

Effects of cold pressing and soxhlet extraction on tocopherol contents of nut oils

Tocopherol contents of nut (almond, apricot, cashew, hazelnut, peanut, pistachio, pecan and walnut) oils obtained by cold pressing and soxhlet extraction are shown in Table 4. The α-tocopherol contents were between 0.07 (walnut) and 257.42 mg/kg (hazelnut) in cold pressed oils and they ranged between 0.03 (pistachio) and 209.73 mg/kg (hazelnut) in case of soxhlet extracted oils. In addition, β-tocopherol contents of cold pressed nut oils were between 0.27 (pecan) and 65.93 mg/kg (peanut) whereas those of soxhlet extracted oils ranged from 0.11 (pecan) to 60.57 mg/kg (peanut). Similarly, γ-tocopherol contents of cold pressed oils were between 3.21 (cashew) and 278.61 mg/kg (pecan) whereas those in soxhlet extracted oils ranged from 2.87 (cashew) to 236.48 mg/kg (pecan). The highest δ-tocopherol was found in peanut oil obtained by cold pressing (8.33 mg/kg). Generally, the tocopherol contents of nut oil samples extracted by soxhlet extraction were found to be somewhat lower than those for the cold pressed oils. The mean α-tocopherol contents of peanut, almond and hazelnut kernel oils were found to be 6.1, 24.2, and 31.4 mg/100 g, respectively in another study (Kornsteiner et al. 2006). In previous study, while α-, β-, γ- and δ-tocopherol contents of walnut (*Juglans regia*) obtained by soxhlet and cold pressing are determined as 4.07 and 4.87, 1.20 and 1.20, 138.13 and 144.97, and 11.10 and 14.37 mg/kg, α-, β-, γ- and δ-tocopherol contents of pican (*Carya illinoensis*) were established as 3.70 and 5.17, 0.97 and 1.30, 153.23 and 165.73, and 7.80 and 7.90 mg/kg, respectively (Colic et al. 2015). On the other hand, Yang (2009), reviewing the benefits of chestnuts related to health, found 122 mg/kg of total tocopherols for macadamia (*Macadamia integrifolia*) and 291 mg/kg for pistachio (*Pistacia vera* L.) nuts. They also reported that cashew, hazelnut, peanut, pecan, pistachio and walnut kernel oils

Table 4 Tocopherol profiles of nut oils obtained by cold press and soxhlet extraction (mg/kg)

Tocopherols	Cold pressed oils						
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
α-Tocopherol	198.37 ± 3.56*b	257.42 ± 5.62a	98.65 ± 1.67d	11.85 ± 1.13e	0.08 ± 0.01	167.85 ± 4.78c	0.07 ± 0.01f	0.11 ± 0.03f
β-Tocopherol	6.83 ± 0.78e**	10.67 ± 0.64d	65.93 ± 1.67a	0.27 ± 0.03h	13.56 ± 1.17c	4.71 ± 0.89f	17.34 ± 1.32b	2.69 ± 0.78g
γ-Tocopherol	11.57 ± 1.54e	134.76 ± 3.87b	47.52 ± 1.17c	278.6 ± 2.38a	11.24 ± 0.51e	7.59 ± 0.63f	21.78 ± 1.45d	3.21 ± 0.26g
δ-Tocopherol	0.31 ± 0.05e	6.71 ± 0.68b	8.33 ± 1.17a	3.78 ± 0.54c	2.81 ± 0.32d	0.17 ± 0.03e	0.06 ± 0.01e	0.43 ± 0.07e
Tocopherols	Soxhlet extraction oils							
	Almond	Hazelnut	Peanut	Pecan	Walnut	Apricot	Pistachio	Cashew
α-Tocopherol	158.85 ± 3.74b	209.73 ± 4.89a	82.49 ± 2.45d	9.53 ± 0.71e	0.05 ± 0.01f	149.84 ± 3.76c	0.03 ± 0.01f	0.07 ± 0.01f
β-Tocopherol	5.62 ± 0.55e	8.84 ± 0.97d	60.57 ± 1.48a	0.11 ± 0.03h	11.71 ± 1.27c	4.21 ± 0.36f	15.71 ± 1.23b	2.17 ± 0.23g
γ-Tocopherol	9.56 ± 0.19f	113.79 ± 1.57b	42.61 ± 1.32c	236.48 ± 3.61a	10.38 ± 1.13e	6.32 ± 0.74g	19.42 ± 1.13d	2.87 ± 0.19h
δ-Tocopherol	0.19 ± 0.03f	5.94 ± 0.47b	7.22 ± 0.56a	3.17 ± 0.21c	2.34 ± 0.27d	0.13 ± 0.01f	0.04 ± 0.01f	0.34 ± 0.03e

*Mean ± SD; **values within each row followed by different letters are significantly different ($p < 0.05$)

contained 0.3, 0.1, 1.8, 0.2, 0.5, and 3.8 mg/100 g δ-tocopherol, respectively. Peanut and almond kernel oils contained 9.4 and 186.4 μg/g α-tocopherol, respectively as reported by Maguire et al. (2004). Peanut, hazelnut, almond, walnut and pistachio oils contained 170, 160, 150, 150 and 150 mg/kg α-tocopherol, respectively (Kırbaşlar et al. 2012). Commercial almond, hazelnut and pecan oils were observed to contain 236.06, 8.52, 10.12 and 0.91 mg/kg α-, β-, γ- and δ–tocopherol for almond, 205.35, 7.12, 8.26 and 0.78 mg/kg α-, β-, γ- and δ–tocopherol for hazelnut, and 5.07, 0.0, 197.09 and 1.57 mg/kg α-, β-, γ- and δ-tocopherol for pecan oil, respectively (Fernandes et al. 2017). Gliszczynska-Swiglo et al. (2007) reported that peanut oil contained 102 mg/kg α-tocopherol, 112 mg/kg (β + γ)-tocopherol and 12.0 mg/kg δ–tocopherols. Both α-tocopherol and other tocopherols are were the main antioxidant component of nuts, and recently considered to be of biological importance in humans (Jiang et al. 2001). Also, γ-tocopherol is the most prevalent form of vitamin E in nut oils (Jiang et al. 2001; Yang 2009). The results obtained in current study are partly in agreement with the previously published data as discussed above although some differences were also observed. Tocopherol contents of nut oils extracted by both systems varied depending on nut types. These differences may be attributed to some climatic factors, varietal and species differences and analytical conditions. Tocopherol contents can change not only from one oil source to another but also among cultivars. The fatty acids and tocopherol contents of cold pressed oil were higher than those of extracted by soxhlet. The increase of the fatty acids and tocopherol content of the cold pressed may possibly be due to the fact that soxhlet oil contains more impurities. On the other hands the nut oils extracted by soxhlet method contained more phenolics when compared to cold-pressed oils.

Conclusion

Oils from eight different nuts were observed to contain substantial amounts of bioactive compounds, fatty acids (especially oleic and linoleic acids), tocopherols and phenolic compounds irrespective of the extraction system. However, variations in bioactive compounds, antioxidant properties, phenolic compounds, fatty acid composition and tocopherol contents were also observed depending on extraction systems. The nut oils were observed to be excellent source of tocopherols, fatty acids, the antioxidant potential and polyphenols. The dominating polyphenols in nut oils were catechin and naringin. Nut oils are monounsaturated fats whose fatty acid composition is dominated by oleic acid (except walnut oil). The oleic acid contents of cold pressed oils were found higher than those of soxhlet extracted oil samples. Nuts and their oils when included in human diet can provide nutritionally important biomolecules as detected in this study. The extraction system can contribute to the improvement in contents and preservation of important nutrients which may be sensitive to heat. In addition, it is important to study oil extraction procedures that minimize use of toxic organic solvents. Cold pressing can be an economical method as it not only excludes the use of heat but also organic solvent as in soxhlet method. The composition of nut oils and nutrients can also change with variety and species, place of origin, harvest time and agro technical measures, in addition to oil extraction procedures.

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