Cold pressed versus solvent extracted lemon (*Citrus limon* L.) seed oils: yield and properties

Emin Yilmaz¹ · Buket Aydeniz Güneşer¹

Revised: 29 March 2017 / Accepted: 2 April 2017 / Published online: 12 April 2017
© Association of Food Scientists & Technologists (India) 2017

Abstract During the processing of lemon fruit, a large quantity of seeds is produced as a by-product. These seeds contain valuable components; therefore, required to be evaluated. This study aimed to compare the cold pressed with hexane-extracted lemon seed oils and determine their physicochemical and thermal properties. Cold pressing yielded significantly lower oil (36.84%) than hexane extraction (71.29%). In addition, the concentrations of free fatty acids, peroxides, and *p*-anisidine were lower in the cold pressed oil. Cold pressed oil showed higher total phenolics, α -tocopherol and antioxidant capacity. The major fatty acids found in the cold pressed oil were linoleic and palmitic acids, whereas β -sitosterol and campesterol were the dominant sterols. The crystallization and melting temperatures and enthalpies were also elucidated. In conclusion, this study proved that high quality of lemon seed oils can be produced by the cold pressing technique; this oil can be used in industries such as the food, cosmetic or chemical industries.

Keywords Lemon seeds · Cold pressing · Hexane extraction · Oil · Composition · Thermal property

Introduction

Lemon (*Citrus limon* L.) is one of the most important fruits belonging to the *Rutaceae* family, consisting of approximately 140 genera and 1300 species. The lemon tree is a small, evergreen shrub native to Southeast Asia; however, it is now common all over the world. The annual lemon production in 2011 was 13,861,441 tons. In the same year, Turkey produced 790,211 tons and was the seventh biggest producer in the world. The yellow ellipsoidal lemon fruit is commonly used for juice and food products, chemical cleaners and medicinal purposes (Anonymous 2016).

During the processing of lemon fruits for juice or other purposes, valuable by-products such as its peels, seeds, and pulp are produced. These materials account for approximately 50% of the fresh fruit weight, and hence, they must be valorized. The most common methods for valorizing these wastes include animal feeding, the processing of food ingredients, and recovering some compounds (Anwar et al. 2008; El-Adawy et al. 1999; Malacrida et al. 2012; Matthaus and Özcan 2012; Saidani et al. 2004). Currently, much attention has been focused on the processing of agrofood by-products and wastes to extract highly valued chemicals and bioactive substances (Galanakis 2012).

Citrus seeds have been recognized as an important source for vegetable oils and proteins (El-Adawy et al. 1999; Malacrida et al. 2012). Plant seed oils can be used for food, industrial and medicinal purposes. Their application in different industries depends on their fatty acid composition, the presence and amount of minor constituents, the sensory properties of the oil, processing yield, and cost. Citrus oils are considered suitable for food applications, soap and detergent production, cosmetic production, pharmaceutical applications. They can also be used as paint and varnish ingredients, lubricants, organic pesticides,

✉ Emin Yilmaz
eyilmaz@comu.edu.tr

Buket Aydeniz Güneşer
buketaydeniz@comu.edu.tr

¹ Department of Food Engineering, Faculty of Engineering,
Çanakkale Onsekiz Mart University, 17020 Çanakkale,
Turkey

dispersants, and surface coatings (El-Adawy et al. 1999; Hosamani and Sattigeri 2000; Malacrida et al. 2012). Citrus seed oils are extracted by solvents in laboratories. The common oil properties of some citrus seed oils, including the fatty acid composition, have been studied by Abdel-Rahman (1980), El-Adawy et al. (1999), El-Saidani et al. (2004), Habib et al. (1986), Lazos and Servos (1988), Malacrida et al. (2012) and Matthaus and Özcan (2012).

However, a limited number of studies have investigated lemon seed oils (Ajewole and Adeyeye 1993; Habib et al. 1986; Malacrida et al. 2012; Matthaus and Özcan 2012; Saidani et al. 2004; Reda et al. 2005; Trandjiiska and Nguyen 1989). In general, the iodine values in citrus oils ranges from 91.4 to 99.3, acid values from 0.21 to 1.2, saponification values from 186.8 to 191.3, specific gravities from 0.912 to 0.923 and refractive indices from 1.4681 to 1.4662 (Habib et al. 1986). The lipid properties of Tunisian blood orange, sweet orange, lemon, and bergamot seeds were studied by Saidani et al. (2004). Lemon seeds contain approximately 78.90% oil, including 3.30% polar lipids, 2.70% diacylglycerols, and 98.20% triacylglycerols. According to some studies, the fatty acid composition of lemon seed oil was 21.40, 2.30, 36.60, 31.40, and 6.90% for the C16:0, C18:0, C18:1, C18:2 and C18:3 fatty acids, respectively. In another study (Malacrida et al. 2012), lemon seed oils were extracted using a Soxhlet extractor and analyzed. Lemon seeds contain 34.92% oil, constituted by major fatty acids including 21.03% palmitic acid, 3.67% stearic acid, 20.80% oleic acid, 44.31% linoleic acid, and 8.96% linolenic acid with 125.01 mg/kg total tocopherols, 4.36 mg/kg carotenoids and 1196.71 mg gallic acid equivalents (GAE)/kg total phenolics. Although the fatty acid composition data of Malacrida et al. (2012) and Saidani et al. (2004) are similar, the oil content data for lemon seeds are fairly different. In a relatively new study (Matthaus and Özcan 2012), the seeds of citrus varieties from Turkey and Vietnam were extracted by petroleum ether and analyzed comprehensively; the oil content of all the investigated citrus samples varied between 32.1 and 58.8 g/100 g. In particular, *Citrus limon* varieties (Interdonato and Kütdiken) were shown to contain 45.1–45.7 g/100 g oil. The fatty acid composition of Kütdiken lemon in this study was 9.0, 5.0, 38.5, 1.1, 44.5, 0.4 and 0.5% for palmitic, stearic, oleic, vaccenic, linoleic, linolenic and arachidic acids, respectively. Similarly, the tocopherol and sterol contents for the Kütdiken lemon were reported; the total tocopherol content was ≤ 14.2 mg/100 g, with α -tocopherol having the maximum content (13.0 mg/100 g). The total sterol content for this lemon variety was 3530.4 mg/kg oil, of which 75.6, 10.4, 4.4, 2.7, and 2.5 mg/kg were β -sitosterol, campesterol, stigmasterol, cholesterol, and Δ^5 -avenasterol, respectively (Matthaus and Özcan 2012).

In this study, the main compositional and physico-chemical properties of the produced lemon seed oils were reported using a pilot-scale cold press machine and laboratory-scale hexane extraction techniques. Cold pressing of oilseeds and kernels is becoming a popular oil production method due to its very unique, specialty oil yields. Cold pressed oils are not refined and are usually sold after a simple filtration or centrifugation process. They contain all the minor, bioactive components naturally present in the oil, do not contain any chemical contaminants and have distinctive flavors and aromas depending on the source of raw materials. Cold pressed oils can not only be used for edible purposes but can also be used in the cosmetic and pharmaceutical or oleochemical industries. Cold pressing is rather a rapid, environmentally friendly and cheap process, but its oil yield is usually lower than that produced by solvent extraction (Aydeniz et al. 2014; Grajzer et al. 2015; Yılmaz et al. 2015a). The current study differs from the previous lemon seed oil studies in several aspects. First, the seed oil was obtained by using the cold pressing technique and the oil yield and oil properties were compared with seed oil obtained using solvent extraction. In addition, this study presents some physicochemical data, which have not been found in the literature, and thermal data for lemon seed oil. Therefore, this study aimed at evaluating and comparing the cold pressed and solvent extracted lemon seed oils for any possible edible or non-edible applications.

Materials and methods

Materials

In this study, the seeds of *Citrus limon* L. (Kütdiken variety) were used. The lemon seeds were gifted by Limkon Food Industry and Trade Inc. (Adana, Turkey) during the 2013–2014 harvest and processing season. Approximately 30 kg of the seeds were washed, drained, dried and kept in a deep freezer (-20 °C) until cold pressing. All the chemicals used were of analytical and/or *chromatographic* grade and purchased from Merck Co. (Darmstadt, Germany) and Sigma Chem. Co. (St. Louis, USA). Fatty acids, tocopherol and sterol standards used in the chromatographic analyses were purchased from Supelco (Bellefonte, PA, USA), Nu-Check (Elysian, MN, USA) and Sigma Chem. Co.

Lemon seed analyses

The dimensions of 10 randomly sampled lemon seeds were measured using a digital caliper (CD-15CP, Mitutoyo Ltd., Andover, UK). Randomly counted 10 seeds were weighed (Sartorius ED224S, Sartorius, Germany) and multiplied

with 100; this process was repeated four times to determine the 1000-seeds weight. To calculate the skin:flesh ratio, 10 lemon seeds were selected randomly, and the seed skins were removed. The seed flesh and skins were weighed separately. This determination was also repeated four times. The color of the lemon seeds was read using a Minolta colorimeter (CR-400, Osaka, Japan) calibrated with a white tile previously. Seed moisture (%), as a proximate composition, was measured with Ohaus MB45 moisture analyzer (Switzerland) at 110 °C for 30 min; seed water activity was measured using an AquaLab 4TE (Decagon Inc. US) instrument; seed oil content was calculated using the AOAC method 920.39 (AOAC 2006); seed protein was estimated using the Kjeldahl technique of AOCS Aa 5-38 (AOCS 1997) and seed total ash was estimated with the AOCS Ba 5a-49 (AOCS 1997) method.

Cold pressing of lemon seeds

Before cold pressing the lemon seeds, a pre-roasting was applied to the seeds by heating the seeds for 30 min at 150 °C in an oven (Inoksan FPE 110, Bursa, Turkey) with frequent agitation. The seed moisture content was monitored and set to 12% by water conditioning before pressing. A laboratory scale cold press machine (Koçmaksan, ESM 3710, İzmir, Turkey), which is a single head, 2 hp, 1.5 kW power and 12 kg seed/h capacity, was used to cold press the lemon seeds. The machine was operated at the following constant operation parameters: 30 rpm screw rotation speed, 10-mm exit die, and maximum 40 °C oil exit temperature. There were two separate oil pressings (each with 15 kg seeds) for the two replicates of the study. The cold pressed lemon seed oils were centrifuged (Sigma 2-16 K, Postfach, Germany) at $6797\times g$ for 10 min before storing in amber colored glass bottles. Subsequently, the oil was flushed with nitrogen and stored in a refrigerator during the analyses. The press cakes obtained from the cold press machine (meals) were ground (Retch Grindomix GM 300, Germany), placed into zipped refrigerator bags and maintained at -20 °C until analysis.

Solvent extraction of lemon seeds

The lemon seeds were first ground (Retch Grindomix GM 300, Germany) at 2500 rpm for 40 s, and then, the moisture content of the material was reduced to 5% in the oven (Inoksan FPE 110, Bursa, Turkey) at 110 °C. The ground seeds and hexane were mixed at 1:2.5 (w/v) ratios, placed into closed plastic vessels and shaken for 3 h at 45 °C in a water bath (Thermal Electronics, Istanbul, Turkey). Subsequently, the micelle was decanted and the extraction was repeated three times. The collected micelle was vacuum evaporated (Heidolph Rotavapor, Germany) at 60 °C and

the seed oil was collected and centrifuged (Sigma 2-16 K, Postfach, Germany) at $6797\times g$ for 10 min. To remove all the remaining hexanes prior to tight closing and storing the samples in the deep freeze, open vial nitrogen flushing was performed for 30 min at room temperature.

Analysis of lemon seed press cakes

The lemon seed press cakes obtained from seed cold pressing and solvent extraction were analyzed for moisture content, water activity, ash, oil and protein contents, and instrumental color values using the same methods described for the seed analysis.

Physicochemical analysis of lemon seed oils

The specific gravities of the oil samples were measured using oil pycnometer with the AOCS Cc 10c-95 method (AOCS 1997) at 25 °C. The refractive indices of the samples were determined using an Abbe 5 (Bellingham and Stanley, UK) refractometer at 25 °C. The apparent viscosities were measured using a Brookfield Viscometer (model DV II+Pro with Rheocalc software, Brookfield Eng. Lab., Inc., MA, USA) with LV-SC4-18 spindle and 30 rpm at 25 °C. The turbidity (25 °C) values were measured using a Hach 2100 AN Turbidimeter (USA). The instrumental color values (L , a^* and b^*) were determined using a Minolta Colorimeter CR-400 (Minolta Camera Co., Osaka, Japan). The sediment contents of the oil samples were measured gravimetrically according to the method described by Emir et al. (2015). The free fatty acid contents and acid values of the samples were measured using the Ca 5a-40 and Cd 3d-63 methods (AOCS 1997). Similarly, the peroxide value, p -anisidine value, and iodine number were measured using the Cd 8-53, Cd 18-90 and Cd 1-25 methods (AOCS 1997), respectively. The saponification numbers and unsaponifiable matters were evaluated using the Tl 1a-64 (AOCS 1997) and ISO 3596 (ISO 2000) methods, respectively. The total phenolic contents were measured using the Folin–Ciocalteu reagent, and the antioxidant capacities were estimated using the Trolox equivalent antioxidant capacity technique, previously described in our study (Aydeniz and Yılmaz 2012).

Fatty acid, sterol and tocopherol composition of lemon seed oils

The fatty acid methyl esters (FAMES) were prepared using the Ce 2-66 (AOCS 1997) method and quantified using a gas chromatograph (Agilent Technologies 7890B, Palo Alto, CA, USA) with a flame ionization detector (FID) (Agilent Technologies, Palo Alto, CA, USA), equipped with HP 88 capillary column (100 m $\times$ 0.25 mm i.d.,

0.2 μm film thickness, J&W Scientific Co, CA, USA). The analytical GC conditions were as follows: oven temperature 120 °C for 1 min, 175 °C (10 °C/min) for 10 min, 210 °C (5 °C/min) for 5 min and 230 °C (5 °C/min) for 5 min; injection volume 1 μL , injector split ratio 1:50; flow rate 2 mL/min; hydrogen as carrier gas injector and the detector temperatures were 250 °C and 280 °C, respectively. FAMEs were quantified by co-chromatography with FAME mixture standards (37-components, C4-C24, Supelco, Bellefonte, PA, USA).

To determine the sterol composition in the oil samples, the ISO 12228 method (ISO 1999) was used. First, the sterol fractions were separated on a thin layer chromatography (TLC) after the saponification procedure. The separated sterol fraction were then analyzed with a gas chromatograph (Agilent Technologies 7890B, Palo Alto, CA, USA) equipped with a FID (Agilent Technologies, Palo Alto, CA, USA) and DB5 capillary column (30 m $\times$ 0.25 mm i.d., 0.1 μm film thickness, J&W Scientific Co, CA, USA). The analysis conditions were as follows: oven temperature 60 °C for 2 min, 60–220 °C (40 °C/min) for 1 min, 220–310 °C (5 °C/min) for 30 min; injection volume 1 μL ; injector split ratio 1:100; flow rate 0.8 mL/min; hydrogen carrier gas (30 mL/min) and injector and the detector temperatures of 290 and 300 °C, respectively. The relative retention times of commercially available standards (cholesterol, brassicasterol, stigmasterol, and β -sitosterol) under the same operating conditions were compared with those of the samples for identification and quantification.

α -Tocopherol contents of the samples were measured using reverse-phase HPLC (Shimadzu Corporation, Kyoto, Japan) equipped with an LC-20AT HPLC pump, DGU-20A5R degasser, CTQ-10ASVP column oven, and RF-20A diode array detector. The method of Grilo Câmara et al. (2014) was followed with minor modifications. In brief, 20 μL of the samples (0.15 g oil in 3 mL dichloromethane) was injected by an autosampler (SIL-20AHT) into the Inertsil ODS-3 column (250 mm $\times$ 4.6 mm i.d., 5 μm film thickness, GL Sciences Inc., Japan). The isocratic elution flow rate was 1.6 mL/min. Methanol:water (98:2 v/v) was used as the mobile phase. The wavelengths of the detector were 290 nm for excitation and 330 nm for emission. Tocopherol quantification was performed using an α -tocopherol standard (Merck, Darmstadt, Germany).

Thermal analysis of the lemon seed oils

A Perkin-Elmer 4000 Series Differential Scanning Calorimeter-DSC (Groningen, The Netherlands) was used to assess the thermal parameters of the lemon seed oils. The instrument was calibrated with indium and zinc standards previously and purged with nitrogen at a flow rate of

50 mL/min for 30 min to prepare the instrument for the analysis. Approximately 5–7 mg of oils were weighed and sealed hermetically in aluminum pans. The temperature program applied for the analysis was as follows: heating from room temperature to 110 °C by 10 °C/min rate, cooling to –70 °C by 10 °C/min rate and holding at this temperature for 3 min for full crystal formation and finally heating the sample again to 50 °C by 5 °C/min rate. The thermal parameters of melting temperature (T_m), melting enthalpy (ΔH_m), crystallization temperature (T_c), and crystallization enthalpy (ΔH_c) were calculated using the Pyris 1 Manager software of the instrument (Yılmaz et al. 2015b).

Furthermore, the oxidative induction time (OIT) was estimated with the DSC. The samples that were sealed in aluminum pans were first heated from 30 to 130 °C at a 20 °C/min heating rate under constant nitrogen flushing (50 mL/min) and were then subjected to an isothermal temperature programming at 130 °C with 50 mL/min purified oxygen (99.8%) application on the samples. The thermograms of the analysis were used to calculate the OIT value using the Pyris 1 Manager Software (Yılmaz et al. 2015b).

Statistical analysis

Cold pressing and solvent extraction of the lemon seeds were replicated twice. For each of these replicate samples, all the listed analyses were performed in at least duplicate or triplicate. The comparison of the oil samples for the measured properties between the groups was done using one-way ANOVA and Tukey's tests with the Minitab ver. 16.1.1 (Minitab 2010) and SPSS package (SPSS 1994) programs. All the values presented in the tables are an average of four determinations $\pm$ standard error. There was a minimum 95% level of confidence for all the statistical analyses.

Results and discussion

Characteristics of lemon seeds

The major seed properties are shown in Table 1. The seed dimensions are important criterion of the seed geometry. The seed sizes vary according to different varieties. Similarly, 1000-seed weight and seed skin: flesh ratio provides the data on the fractions of the seed components.

The color values indicated that the lemon seeds were moderately light and yellow colored. Initially, the washed, drained and dried seeds contained approximately 42% moisture with 0.96 water activity value. The seeds were maintained in a deep freeze until utilization. The moisture

Table 1 Material properties of the lemon seeds

	Mean value
Seed size (mm)	
Length	11.29 ± 0.38
Width	5.36 ± 0.28
Height	3.96 ± 0.18
1000-seed weight (g)	152.04 ± 6.58
Skin:flesh ratio	0.65 ± 0.06
Color L	54.86 ± 1.17
a*	3.06 ± 0.57
b*	19.17 ± 0.70
Moisture (%)	41.94 ± 0.40
Water activity (25 °C)	0.96 ± 0.01
Oil (%) ^a	34.55 ± 0.53
Protein (%) ^a	19.41 ± 0.18
Ash (%)	1.41 ± 0.05

^a Values on dry weight basis

level before cold pressing was reduced to approximately 12% using a conventional oven. The oil and protein contents of the seeds, were 34.55 and 19.41%, respectively. The total ash content of the seeds was approximately 1.41%. Large differences have been reported in the oil content of lemon seeds in the literature. Saidani et al. (2004) reported 78.90% oil from Tunisian lemon seeds, while Malacrida et al. (2012) reported 34.92% oil from lemon seeds. In another study (Matthaus and Özcan 2012), oil content in the Küt diken variety lemon seeds from Turkey was found to be 45.1%. Although the same Küt diken variety was used in the earlier (Matthaus and Özcan 2012) and our studies, the harvest year and the methods of oil extraction were different. The total ash content of sweet lemon seeds was reported (Anwar et al. 2008) as 5.50%, which was significantly higher than our finding (1.41%). The total ash contents of other citrus seeds were as follows: orange seed contained 2.95% ash, grapefruit seeds contained 2.60% ash (Habib et al. 1986) and orange seed contained approximately 3.17% ash (El-Adawy et al. 1999). In general, ash content measured in this study for lemon seeds was lower than the values reported in the literature for citrus seeds.

Oil yields and composition of lemon seed meals

One of the primary objectives of this study was to compare the oil yield of lemon seeds from laboratory scale cold pressing and solvent extraction. The total oil content of the seeds determined by the Soxhlet extraction technique was approximately 34.55% (Table 1). On overall seed weight, cold pressing provided approximately 12.73% oil, whereas

Table 2 Proximate composition of the lemon seed press meals

Property	Lemon seed meal	
	Cold pressed	Solvent extracted
Moisture (%)	10.53 ± 0.42 ^A	11.91 ± 0.65 ^A
Water activity (25 °C)	0.51 ± 0.02 ^B	0.69 ± 0.03 ^A
Ash (%)	2.79 ± 0.04 ^A	2.57 ± 0.04 ^B
Oil (%) ^a	17.18 ± 0.14 ^A	5.25 ± 0.62 ^B
Protein (%) ^a	24.59 ± 0.58 ^A	23.42 ± 0.92 ^A
Color L	57.10 ± 0.30 ^B	75.26 ± 0.08 ^A
a*	5.99 ± 0.22 ^A	1.72 ± 0.22 ^B
b*	21.84 ± 0.26 ^A	18.64 ± 1.01 ^B

^a Values on dry weight basis

^{A,B} The same horizontal rows followed by different superscript letters were significantly different ($p < 0.05$)

hexane extraction provided approximately 24.63% oil. Table 2 shows the oils remaining in the lemon seed cakes (meals). The percentage of residual oil was significantly higher in the cold press meal (17.18%) than in the solvent extraction meal (5.15%). Therefore, the oil yield values of cold pressing and solvent extraction processes were calculated to be 36.84 and 71.29%, respectively. This result is consistent with the literature, reporting that the total oil yield is significantly lower with the cold pressing method. The solvent extraction of chia seeds yielded approximately 30% more oil than cold pressing. Cold pressing, hexane extraction and ethanol extraction of niger seeds yielded 28.3, 38.3 and 29.2 g/100 g oil, respectively (Aydeniz et al. 2014; Bhatnagar and Krishna 2014; Ixtaina et al. 2011; Yılmaz et al. 2015a).

Table 2 presents the composition of lemon seed meals. Although the moisture contents of the lemon seed meals from cold pressing and solvent extraction were almost the same, there were some differences in the measured water activity values. Similarly, the protein and ash contents were not significantly different between the meals. The protein and ash contents in the meals (Table 2) were better than those in the seeds (Table 1). The percentage of residual oil content in the cold press meal was quite higher than that in the solvent extracted meal. This difference explains the differences in the oil yield between the two oil processing methods. The color parameter examination showed that the solvent extracted meal was lighter and less red and yellow than the cold pressed meal (Table 2). However, it is still unknown if the meals could be further used to extract bioactive compounds such as flavonoids, fiber, and protein. Further studies are needed for lemon seed meal valorization. In general, cold pressing is a low-yielding, but high-quality oil production process (Aydeniz et al. 2014; Yılmaz et al. 2015a). Cold pressed oils contain all the originally present minor bioactive compounds and the oil is free from

any solvents or chemicals that are used in classical solvent extraction and oil refining processes (Anderson 1996; Yilmaz et al. 2015a).

Physicochemical properties of lemon seed oils

There was no statistically significant difference between the cold pressed and solvent extracted lemon seed oils for specific gravity, refractive index, and viscosity values (Table 3). Anwar et al. (2008) reported that the specific gravity of sweet lemon, orange, grapefruit, and mandarin orange seed oils was 0.941, 0.920, 0.932 and 0.927 mg/mL and the refractive index values were 1.4670, 1.4645, 1.4639, and 1.4658 (at 40 °C), respectively. For different citrus seed oils, the specific gravity values range from 0.884 to 0.962 g/cm³, refractive index values range from 1.4672 to 1.4684 (at 25 °C), and viscosities range from 0.05 to 0.08 Pa s (El-Adawy et al. 1999). Our findings are in accordance with those reported in the literature. The turbidity values largely differ between the two oils; cold pressed oils are very turbid compared to the solvent extracted oil (Table 3). Although no refining after solvent extraction of the lemon seed oil was performed, its turbidity value was much lower. To the best of our knowledge, no data for lemon seed oil turbidity was found in the literature. Cold pressed Indian niger seeds were golden orange clear liquids, whereas hexane and ethanol extracted ones were golden yellow liquid with a slight haze and

brownish thick liquids, respectively (Bhatnagar and Krishna 2014). The color parameters of both the samples were significantly different. The L-value of the solvent extracted oil was higher than that of the cold pressed oil, indicating a more luminous appearance (Table 3). Both the oils had little reddish tones (a* value) but the level of yellowness was significantly higher in the solvent extracted oil (17.48 b* value) than in the cold pressed oil (4.38 b* value). Higher pigments in the solvent extracted oil provided the luminous yellow colors. Lovibond tintometer color values of 3.00 red unit and 30.00 yellow unit for sweet lemon seed oils were reported (Anwar et al. 2008). Although the color measurement technique used by Anwar et al. was different from ours, both the results consistently showed that lemon seed oils are mostly yellow and little reddish. Although the solvent extracted oil sample had a little more sediment than the cold pressed oil sample, this difference was not statistically significant. In addition, cold pressed poppy seed oil samples had lower levels of sediment content (Emir et al. 2015).

The important chemical properties of the oils determined are enlisted in Table 3. The free fatty acidity (FFA) and acid value (AV) of the solvent extracted lemon seed oils were significantly higher than those of the cold pressed lemon seed oils. Both the values were quite low (0.64 vs. 1.05% FFA and 1.27 vs. 2.11 AV) and acceptable for the cold pressed and solvent extracted lemon seed oils according to the Turkish codex for named vegetable oils, which state that AV up to

Table 3 Some physicochemical properties of the lemon seed oils

Property	Lemon seed oil	
	Cold pressed	Solvent extracted
Specific gravity (g/ml) (25 °C)	0.94 ± 0.01 ^A	0.94 ± 0.01 ^A
Refractive index (25 °C)	1.47 ± 0.01 ^A	1.47 ± 0.01 ^A
Viscosity (25 °C, cP)	56.55 ± 1.16 ^A	54.57 ± 0.16 ^A
Turbidity (25 °C, NTU)	19.50 ± 4.99 ^A	1.00 ± 0.01 ^B
Color L	28.09 ± 0.99 ^B	33.71 ± 0.55 ^A
a*	0.40 ± 0.21 ^B	1.88 ± 0.25 ^A
b*	4.38 ± 0.46 ^B	17.48 ± 2.12 ^A
Sediment content (%)	5.93 ± 0.27 ^A	7.50 ± 0.56 ^A
Free fatty acid (% linoleic acid)	0.64 ± 0.01 ^B	1.05 ± 0.01 ^A
Acid value (mg KOH/g oil)	1.27 ± 0.01 ^B	2.11 ± 0.09 ^A
Peroxide value (meq O ₂ /kg oil)	9.49 ± 1.35 ^B	39.11 ± 6.65 ^A
p-anisidine value	1.12 ± 0.50 ^B	3.39 ± 0.85 ^A
Iodine number (g I/100 g oil)	117.49 ± 2.92 ^B	129.11 ± 0.52 ^A
Saponification number (mg KOH/g oil)	199.96 ± 1.78 ^A	200.33 ± 1.31 ^A
Unsaponifiable matter (%)	1.08 ± 0.23 ^A	1.17 ± 0.01 ^A
Total phenolics (μg GA/100 g)	4916.0 ± 326.0 ^A	3863.0 ± 59.70 ^B
TEAC (μmol Trolox/100 g oil)	11,669.0 ± 36.20 ^A	7311.0 ± 662.0 ^B

^{A,B} The same horizontal rows followed by different superscript letters were significantly different ($p < 0.05$)

4.0 mg KOH/g oil is acceptable for cold pressed and virgin oils (Codex 1999). An AV of 2.18 mg KOH/g oil for sweet lemon seed oil was reported by Anwar et al. (2008), whereas AVs ranging from 0.673 to 1.120 mg KOH/g oil were reported for four other citrus seed oils (El-Adawy et al. 1999). In another study (Habib et al. 1986), AVs of 0.21, 0.65, 1.20 and 0.90 mg KOH/g oil were reported for orange, mandarin, lime and grapefruit seeds oils, respectively. The FFA of cold pressed canola oils was 0.13 and 0.32%, hot pressed oil was 0.41%, solvent extracted oil was 0.50%, and refined–bleached–deodorized oil was 0.04% (Ghazani et al. 2014). Similarly, AVs of solvent extracted and cold-pressed chia seed oils were 2.05 and 0.91 mg KOH/g oil, respectively (Ixtaina et al. 2011). In general, our results are in agreement with those reported in the literature. The peroxide values and *p*-anisidine values were measured in the oil samples as the most widely accepted oxidation parameters (Table 3). Both the values were significantly lower in the cold pressed samples than in the solvent extracted samples. This is an important finding, indicating the advantages of cold pressing technique. The codex (Codex 1999) permits maximum 15 meq O₂/kg oil peroxide value for virgin oils. Therefore, the peroxide value of the solvent extracted lemon seed oil sample exceeds the legal limit and must be refined. This again highlights the advantage of cold press oil processing, if the lower oil yield is considered. Anwar et al. (2008) reported 1.97 meq O₂/kg oil peroxide value and 2.85 *p*-anisidine value for sweet lemon seed oil. Peroxide values ranging from 5.90 to 6.37 meq O₂/kg oil were reported for four different citrus seed oils (El-Adawy et al. 1999). Similarly, peroxide and *p*-anisidine values of orange seed oil were 2.33 meq O₂/kg oil and 1.86, respectively (Saloua et al. 2009). The peroxide and *p*-anisidine values of cold pressed commercial rose hip oils ranged between 1.2 and 2.1 meq O₂/kg oil and between 2.5 and 7.7, respectively (Grajzer et al. 2015). In another study (Bhatnagar and Krishna 2014), the peroxide values of Indian niger seed oils were reported as 2.2 for cold pressed, 3.2 for hexane-extracted and 2.0 meq O₂/kg oil for ethanol extracted oil samples. Thus, the results are in general in agreement with those reported in the literature.

Although the iodine numbers of the two oil samples were statistically different, the saponification numbers and unsaponifiable matter contents were quite similar (Table 3). The iodine value for sweet lemon seed oil was 110 g I/100 g oil (Anwar et al. 2008). Similarly, the iodine values for some citrus seed oils range from 91.54 to 102.57 g I/100 g oil (El-Adawy et al. 1999). The iodine numbers determined in this study for lemon seed oils are higher (117.49 and 129.11 g I/100 g oil) than those reported in the literature for citrus seed oils. The saponification values of the cold pressed and solvent extracted lemon seed oils were 199.96 and 200.33 mg of KOH/g of oil. The saponification values for sweet lemon, grapefruit, orange and mandarin

orange seed oils were 180.90, 198.85, 189.50 and 186.00 mg of KOH/g of oil (Anwar et al. 2008). In another study (El-Adawy et al. 1999), the saponification values for four citrus seed oils ranged from 187.2 to 190.2 mg of KOH/g of oil. Overall, the saponification values determined in this study appear to be higher than those reported in the literature. This difference can be attributed to the variety of seeds, climate, and other factors. Hexane extracted sweet lemon seed oil contain approximately 0.31% unsaponifiable matter (Anwar et al. 2008); this value is much lower than the unsaponifiable matter determined in cold pressed (1.08%) and hexane extracted (1.17%) lemon seed oils in this study. Furthermore, Saloua et al. (2009) reported that the unsaponifiable matter for hexane extracted orange seed oil was 1.57%.

The total phenolic content of cold pressed lemon seed oil (4916.0 µg GA/100 g) was significantly higher than that of the solvent extracted lemon seed oil (3863.0 µg GA/100 g). The same significant difference was also evident in the antioxidant capacity values for cold pressed and solvent extracted lemon seed oils (11,669.0 vs. 7311.0 µmol Trolox/100 g oil). These results indicate that phenolic and other antioxidant compounds leach more into the lemon seed oil by the cold pressing technique. There is no refining step after cold pressing; therefore, the phenolic and other antioxidant compounds remain in the oil, providing more health benefits and extending the shelf life of the oils. The total phenolic content of orange, lemon and tangerine seed oils extracted by petroleum ether was 1152.88, 1196.71 and 1007.77 mg GAE/kg oil, respectively. Furthermore, the order of effectiveness of the oils in inhibiting DPPH free radicals was determined for orange, lemon, and tangerine seed oils (Malacrida et al. 2012). The phenolic content and antiradical activity of cold pressed rose hip oils (Grajzer et al. 2015) were 783.55 µg/kg and up to 3.00 mM/kg, respectively. In another study (Ghazani et al. 2014), the total phenolic content of solvent extracted, hot pressed, cold pressed and refined–bleached–deodorized canola oil samples was 112.9, 48.6, 2.9–3.0 and 1.4 mg/kg oil. Although the measurement techniques and units of expression were different to directly compare the oils, data show that lemon seed oils are rich in phenolic compounds and have very high antioxidant capacities.

Fatty acid, sterol and tocopherol composition of lemon seed oils

Table 4 shows the fatty acid, sterol and α-tocopherol composition of the lemon seed oils. On quantifying six fatty acids in both the samples, no significant difference was found between the cold pressed and solvent extracted lemon seed oils. Linoleic, oleic and palmitic acids corresponded to approximately 34, 30 and 20% of the total fatty

Table 4 Fatty acid, sterol and tocopherol compositions of the lemon seed oils

	Lemon seed oil	
	Cold pressed	Solvent extracted
Fatty acids (%)		
Palmitic (C16:0)	20.88 ± 0.03	20.90 ± 0.03
Palmitoleic (C16:1)	0.27 ± 0.01	0.26 ± 0.01
Stearic (C18:0)	4.28 ± 0.01	4.32 ± 0.01
Oleic (C18:1 n-9)	30.86 ± 0.20	30.27 ± 0.01
Linoleic (C18:2 n-6)	33.77 ± 0.12	33.99 ± 0.03
Linolenic (C18:3 n-3)	8.35 ± 0.13	8.65 ± 0.11
Sterols (%)		
Cholesterol	1.23 ± 0.03	1.12 ± 0.05
Brassicasterol	0.05 ± 0.01	0.05 ± 0.02
24-Methylen cholesterol	0.10 ± 0.06 ^A	0.03 ± 0.01 ^B
Campesterol	10.71 ± 0.16 ^B	12.57 ± 0.05 ^A
Campestanol	0.18 ± 0.03	0.19 ± 0.01
Stigmasterol	4.52 ± 0.04 ^B	5.04 ± 0.01 ^A
Delta-7 campesterol	0.32 ± 0.01	0.34 ± 0.01
Delta-5,23 stigmastadienol	0.06 ± 0.01	0.04 ± 0.01
Chlerosterol	0.98 ± 0.16	0.94 ± 0.02
Beta-sitosterol	76.55 ± 0.52	75.10 ± 0.04
Sitostanol	0.79 ± 0.08 ^A	0.60 ± 0.06 ^B
Delta-5 avenasterol	3.24 ± 0.09	3.19 ± 0.04
Delta-5,24 stigmastadienol	0.21 ± 0.03 ^A	0.09 ± 0.02 ^B
Delta-7 stigmastenol	0.69 ± 0.04 ^A	0.48 ± 0.01 ^B
Delta-7 avenasterol	0.34 ± 0.06 ^A	0.20 ± 0.11 ^B
Tocopherol (mg/kg oil)		
α-Tocopherol	155.00 ± 28.80 ^A	110.20 ± 12.60 ^B

^{A,B} The same horizontal rows followed by different superscript letters were significantly different ($p < 0.05$)

acid content, respectively. In addition, approximately 8.5% linolenic, 4.0% stearic and 0.2% palmitoleic acids were quantified. El-Adawy et al. (1999) reported that the fatty acid composition of citron seed oil included 0.39, 0.43, 29.52, 4.32, 22.25, 33.21, 9.56 and 0.32% of lauric, myristic, palmitic, stearic, oleic, linoleic, linolenic and arachidic acids, respectively. In another study (Saidani et al. 2004), 17.17% palmitic, 26.20% linoleic and 24.70% linolenic acids were reported for lemon seed oils. In a more recent study (Malacrida et al. 2012), the fatty acid composition of lemon seed oil included 21.03% palmitic, 0.65% palmitoleic, 3.67% stearic, 20.80% oleic, 44.31% linoleic, 8.96% linolenic, 0.31% arachidic, 0.10% behenic and 0.17% lignoceric acids. This result is similar to our findings. Another study by Matthaus and Özcan (2012) also analyzed the same lemon variety Kütdiken, reporting a fatty acid composition that included 9.0% palmitic, 5.0% stearic, 38.5% oleic, 1.1% vaccenic, 44.5% linoleic, 0.4% linolenic and 0.5% arachidic acids. When this study was

compared with our study, it was observed that the palmitic and linolenic acids were higher and oleic and linoleic acids were lower in our study (Table 4). This variation could be due to the difference in the cultivation region, harvest season and climate conditions of the lemon orchards.

Fifteen sterols were quantified in the lemon seed oil samples (Table 4). The most abundant one was β-sitosterol (approximately 76%), followed by campesterol (10%–12%), stigmasterol (4.5–5.0%) and Δ-5-avenasterol (3.20%). Except for cholesterol (1.12%–1.23%), all other sterols were <1.0% in both the cold pressed and solvent extracted lemon seed oil samples. According to Matthaus and Özcan (2012), the sterol composition of the Kütdiken lemon seed oil was 75.6, 10.4, 4.4, 2.7 and 2.5% for β-sitosterol, campesterol, stigmasterol, cholesterol and Δ-5-avenasterol, respectively. The findings of both the studies concur.

In this study, the α-tocopherol content and not the tocopherol composition was determined (Table 4). Cold pressed lemon seed oils had significantly higher (155.00 mg/kg) α-tocopherol content than the solvent extracted (110.20 mg/kg) counterparts. The effects of different extraction conditions on canola oil tocopherols were investigated by Ghazani et al. (2014). The total tocopherol content of solvent extracted canola oil was 492.5 mg/kg, whereas that of cold pressed canola oils was 354.1 and 365.9 mg/kg. This finding contradicts our results. Similar contradictory results were obtained for chia seed oils (Ixtaina et al. 2011); significantly higher total tocopherols (around 300–400 mg/kg) were measured in the solvent extracted samples than in the pressed samples (250–330 mg/kg). In general, the α-tocopherol content of lemon seed oil was lower than that reported in literature for peanut oil (399 mg/kg), flaxseed oil (589 mg/kg), sunflower oil (635 mg/kg) and soybean oil (1798 mg/kg) (Tuberoso et al. 2007). Anwar et al. (2008) found that 26.40, 58.03 and 17.27 mg/kg α-, γ- and δ-tocopherols were found in sweet lemon seed oils. Our finding is significantly higher than those reported by them in terms of α-tocopherol content. In another study (Malacrida et al. 2012), lemon seed oil contained 102.49 mg/kg α-tocopherol, 2.20 mg/kg β-tocopherol, 1.33 mg/kg γ-tocopherol and 18.98 mg/kg δ-tocopherol. This result is more similar to our result. The tocopherol content of Kütdiken lemon seed oil (Matthaus and Özcan 2012) was 13.0, 0.1, 0.4, 0.3, 0.1 and 0.3 mg/100 g for α-tocopherol, α-tocotrienol, β-tocopherol, γ-tocopherol, plastochroman-8 and γ-tocotrienol, respectively. Overall, the α-tocopherol contents reported in our study and their study concur. Since tocopherols are strong antioxidants and possess vitamin E activity, they are extremely important for oil stability and increase the nutritional value of the oils (Matthaus and Özcan 2012; Grajzer et al. 2015).

Thermal properties of lemon seed oils

The crystallization and melting temperatures and enthalpies of the lemon seed oils are shown in Table 5. There was no significant difference for the thermal parameters of both the oils. Both the samples start crystallization (onset_c) at around -4 to -5 °C and fully crystallize (T_c) at around -6 to -8 °C. During the thermal cycling experiment, the samples were cooled to -70 °C before heating them to 50 °C by increasing the temperature at a rate of 5 °C/min. Hence, it was possible to note the temperatures at which the fully crystallized samples started to melt. The melting started (onset_m) at around -25 °C and three other different fractions having melting temperatures of around -20 , -5 to -6 and -4 °C were observed in the cold pressed lemon seed oil. In the solvent extracted sample, only two additional fractions were observed. These thermal data indicate that lemon seed oils contain different triglyceride fractions, which is evident from the different melting profiles. There were no significant differences for the crystallization (ΔH_c) and melting (ΔH_m) enthalpies between the two samples. Although there are no thermal data on the lemon seed oil in the literature, one study (Saloua et al. 2009) reported the following thermal properties of orange seed oils: crystallization onset and peak temperatures of -35.38 and -32 °C with a -15.58 J/g enthalpy value and melting onset and peak temperatures of -22.10 and -13.81 °C with a 52.37 J/g melting enthalpy. Although the lemon seed oils in this study and the orange seed oils (Saloua et al. 2009) had different crystallization and melting temperatures, the enthalpy values were similar. Hence, this study provides important data for the lemon seed oil literature. Lemon seed oils are liquid at refrigerator temperatures, and

hence, they can be useful for varied applications. In general, the fatty acid composition and the presence of other minor components, such as waxes, sterols, and phenols, greatly affect the thermal behaviors of edible liquid oils (Co and Marangoni 2012).

The oxidation induction times (OIT) of the lemon seed oil samples were also measured at a constant temperature of 130 °C (Table 5). The OIT value indicates the time elapsed when the curve starts to change due to the formation of lipid hydroperoxides through the constant flow of pure oxygen in the sample pan. Hence, it is an indicator of oil stability against oxidation. Higher OIT values indicate that the oil is better against oxidation or has longer shelf life. There was no significant difference between the cold pressed and solvent extracted lemon seed oils. Moreover, the difference between the two oils for the measured total phenolic content and antioxidant capacity value (Table 3) was not enough to create any difference in terms of OIT values. In a study (Grajzer et al. 2015) determining the OIT values of rose hip oils, it was reported that the both fatty acid composition and the presence of antioxidant compounds most strongly affect the OIT values. Unfortunately, we could not locate DSC-measured OIT values for citrus seed oils for a comparison. Overall, this study adds important data to the literature.

Conclusion

The main objective of this study was to compare the oil yield and oil properties of lab scale cold pressed and solvent extracted lemon seed oils. The oil yield of cold pressing (36.84%) was significantly lower than that of the solvent extraction (71.29%). Cold pressed lemon seed oils were more turbid and less yellow than the solvent extracted counterparts. As important chemical parameters, it was evident that the FFA, ACs, peroxide values and *p*-anisidine values were significantly lower in the cold pressed sample, indicating superior oil quality. Furthermore, the total phenolic content and antioxidant capacity of the cold pressed lemon seed oil were significantly higher. This is very important in terms of oil stability and nutrition value. Linoleic, oleic, and palmitic acids were quantified as the major fatty acids in both oils, and there was no significant difference between the samples. Although 15 sterols were quantified, β -sitosterol, campesterol, stigmasterol and Δ -5-avenasterol were the most abundant ones in both oil samples. The α -tocopherol content was significantly higher in cold pressed sample. Crystallization and melting temperatures and enthalpies were quantified as useful parameters for determining the different application areas of oils. In conclusion, the utilization of waste lemon seeds for cold press oil production may enhance the profitability of lemon

Table 5 Thermal properties of the lemon seed oils

	Lemon seed oil	
	Cold pressed	Solvent extracted
Crystallization		
Onset _c (°C)	-4.62 ± 0.53^A	-5.04 ± 0.01^A
T_c (°C)	-6.74 ± 0.66^A	-8.10 ± 0.08^A
ΔH_c (J/g)	-14.81 ± 1.06^A	-16.59 ± 1.04^A
Melting		
Onset _m (°C)	-25.11 ± 0.81^A	-25.37 ± 2.14^A
T_{m1} (°C)	-20.72 ± 0.89^A	-20.70 ± 1.58^A
T_{m2} (°C)	-6.19 ± 0.04^A	-4.82 ± 0.60^A
T_{m3} (°C)	-3.97 ± 0.01	nd
T_{m4} (°C)	nd	nd
ΔH_m (J/g)	49.30 ± 0.31^A	48.7 ± 52.90^A
OIT (min)	29.86 ± 1.20^A	28.30 ± 1.38^A

^{A,B} The same horizontal rows followed by different superscript letters were significantly different ($p < 0.05$)

juice industry and could provide valuable stock oil for food, cosmetic, and chemical industries.

Acknowledgements This study was funded by the TUBITAK (The Scientific and Technological Research Council of Turkey) and COST (European Cooperation in Science and Technology) Action TD 1203 (Food Waste Valorisation for Sustainable Chemicals, Materials and Fuels) Project No: 114O876. The authors wish to thank for financial support. We would also thank Limkon Food Industry and Trade Inc. (Adana, Turkey) for supplying the lemon seeds used in the study.

References

- Abdel-Rahman AY (1980) A study on some Egyptian citrus seed oils. *Grasas Aceites* 31:331–333
- Ajewole K, Adeyeye A (1993) Characterisation of Nigerian citrus seed oils. *Food Chem* 47:77–78
- Anderson D (1996) A primer on oils processing technology. In: Hui YH (ed) *Bailey's industrial oil and fat products*. Wiley, New York, pp 1–60
- Anonymous (2016) Lemon. <https://en.wikipedia.org/wiki/Lemon>. Accessed 04 Jan 2016
- Anwar F, Naseer R, Bhanger MI, Ashraf S, Talpur FN, Aladedunye FA (2008) Physico-chemical characteristics of citrus seeds and seed oils from Pakistan. *J Am Oil Chem Soc* 85:321–330
- AOAC (Association of Official Analytical Chemist) (2006) Official methods of analysis of the AOAC, 18th edn. Association of Official Analytical Chemists, Washington
- AOCS (American Oil Chemists' Society) (1997) Official methods and recommended practices, vol I and II, 4th edn. American Oil Chemists' Society, Champaign
- Aydeniz B, Yilmaz E (2012) Enrichment of frying oils with plant phenolic extracts to extend the usage life. *Eur J Lipid Sci Technol* 114:933–941
- Aydeniz B, Güneşer O, Yilmaz E (2014) Physico-chemical, sensory and aromatic properties of cold press produced safflower oil. *J Am Oil Chem Soc* 91:99–110
- Bhatnagar AS, Krishna AGG (2014) Lipid classes and subclasses of cold-pressed and solvent-extracted oils from commercial Indian niger (*Guizotia abyssinica* (L.f.) Cass.) seed. *J Am Oil Chem Soc* 91:1205–1216
- Co ED, Marangoni AG (2012) Organogels: an alternative edible oil-structuring method. *J Am Oil Chem Soc* 89:749–780
- Codex (1999) Codex standard for named vegetable oils, CXSTAN 210. Turkish Food Codex, Ankara, Turkey
- El-Adawy TA, Rahma EH, El-Bedawy AA, Gafar AM (1999) Properties of some citrus seeds. Part 3. Evaluation as a new source of protein and oil. *Nahrung* 43:385–391
- Emir DD, Aydeniz B, Yilmaz E (2015) Effects of roasting and enzyme pretreatments on yield and quality of cold-pressed poppy seed oils. *Turk J Agric For* 39:260–271
- Galanakis CM (2012) Recovery of high added-value components from food wastes: conventional, emerging technologies and commercialized applications. *Trends Food Sci Technol* 26:68–87
- Ghazani SM, Garcia-Llatas G, Marangoni AG (2014) Micronutrient content of cold pressed, hot pressed, solvent extracted and RBD canola oil: implications for nutrition and quality. *Eur J Lipid Sci Technol* 116:380–387
- Grajzer M, Prescha A, Korzonek K, Wojakowska A, Mariusz Dziadas M, Kulma A, Grajeta H (2015) Characteristics of rose hip (*Rosa canina* L.) cold-pressed oil and its oxidative stability studied by the differential scanning calorimetry method. *Food Chem* 188:459–466
- Grilo Câmara E, Costa PN, Gurgel CSS, Beserra AFM, Almeida FNS, Dimenstein R (2014) Alpha-tocopherol and gamma-tocopherol concentration in vegetable oils. *Food Sci Technol (Campinas)* 34:379–385
- Habib MA, Hammam MA, Sakr AA, Ashoush YA (1986) Chemical evaluation of Egyptian citrus seeds as potential sources of vegetable oils. *J Am Oil Chem Soc* 63:1192–1196
- Hosamani KM, Sattigeri RM (2000) Industrial utilization of *Rivea ornata* seed oil: a moderate source of vernolic acid. *Ind Crops Prod* 12:93–96
- ISO (International Standards Office) (1999) 12228:1999, Animal and vegetable fats and oils-determination of individual and total sterols contents—gas chromatographic method. International Standards Official Methods, Geneva, Switzerland
- ISO (International Standards Office) (2000) Animal and vegetable fats and oils—Determination of unsaponifiable matter—method using diethyl ether extraction. International Standards Official Methods, Geneva, Switzerland
- Ixtaina VY, Martinez ML, Spotorno V, Mateo CM, Maestri DM, Diehl BWK, Nolasco SM, Tomas MC (2011) Characterization of chia seed oils obtained by pressing and solvent extraction. *J Food Compos Anal* 24:166–174
- Lazos ES, Servos DC (1988) Nutritional and chemical characteristics of orange seed oil. *Grasas Aceites* 39:232–234
- Malacrida CR, Kimura M, Jorge N (2012) Phytochemicals and antioxidant activity of citrus seed oils. *Food Sci Technol Res* 18:399–404
- Matthaus B, Özcan MM (2012) Chemical evaluation of citrus seeds, an agro-industrial waste, as a new potential source of vegetable oils. *Grasas Aceites* 63:313–320
- Minitab (2010) Minitab statistical software (version 16.1.1). Minitab Inc., State College
- Reda SY, Sauer EL, Batista AEC, Barana AC, Schnitzel E, Carneiro PIB (2005) Characterization of rangpur lime (*Citrus limonia* Osbeck) and “Sicilian” lemon (*Citrus limon*) seed oils, an agro-industrial waste. *Cienc Tecnol Aliment* 25:672–676
- Saidani M, Dhifil W, Marzouk B (2004) Lipid evaluation of some Tunisian citrus seed. *J Food Lipids* 11:242–250
- Saloua F, Eddine NI, Hedi Z (2009) Chemical composition and profile characteristics of Osage orange *Maclura pomifera* (Rafin.) Schneider seed and seed oil. *Ind Crops Prod* 29:1–8
- SPSS (1994) SPSS professional statistics (version 10.1). SPSS Inc., Chicago
- Trandjiiska R, Nguyen H (1989) Triglyceride composition of seed oils from Vietnamese citrus fruits. *Riv Ital Sostanze Gr* 66:99–102
- Tuberoso CIG, Kowalczyk A, Sarritzu E, Cabras P (2007) Determination of antioxidant compounds and antioxidant activity in commercial oilseeds for food use. *Food Chem* 103:1494–1501
- Yilmaz E, Arsunar ES, Aydeniz B, Güneşer O (2015a) Cold pressed capia pepperseed (*Capsicum annum* L.) oils: composition, aroma and sensory properties. *Eur J Lipid Sci Technol* 117:1016–1026
- Yilmaz E, Ögütücü M, Arifoglu N (2015b) Assessment of thermal and textural characteristics and consumer preferences of lemon and strawberry flavored fish oil organogels. *J Oleo Sci* 64:1049–1056