

Changes in the quality parameters of *Cephalaria syriaca* L. seed oil after the refining process

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SUMMARY: The present study has determined that the crude-oil refining process from the *Cephalaria syriaca* (CS) seed, which could be a new vegetable oilseed source, changed its physical and chemical quality properties (except specific gravity and refractive index). It was also determined that the dominant saturated and unsaturated fatty acids in the crude and refined oils were myristic (21.06–11.80%), palmitic (10.8–8.91%), stearic (2.26–2.70%), oleic (29.17–34.24%) and linoleic (35.56–40.57%). The vitamin E values of the crude and refined CS seed oils were 51.95–50.90 mg/kg, respectively. The oxidative stability values for crude and refined CS seed oils were 2.32–2.69 h, respectively. β -sitosterol and campesterol were the predominant sterols. As a result of the refining process, although magnesium, potassium, iron and copper decreased, the ratios of sodium, aluminum, calcium, chromium, strontium, rubidium, and barium increased. The results provide preliminary data for the future consumption of CS oil in particular for refined CS seed oil.

KEYWORDS: *Cephalaria syriaca* seed; Crude and refined vegetable oil; Physico-chemical; Quality properties; Food.

RESUMEN: *Cambios en los parámetros de calidad del aceite de semillas de Cephalaria syriaca L. tras un proceso de refinación.* El presente estudio ha determinado que el proceso de refinación de aceite crudo de la semilla de *Cephalaria syriaca* (CS), que podría ser una nueva fuente de oleaginosas vegetales, modificó sus propiedades de calidad física y química (excepto la gravedad específica y el índice de refracción). También se determinó que los ácidos grasos saturados e insaturados dominantes en los aceites crudos y refinados eran mirístico (21,06–11,80%), palmítico (10,8–8,91%), esteárico (2,26–2,70%), oleico (29,17–34,24%), y linoleico (35,56–40,57%). Los valores de vitamina E de los aceites de semillas CS crudos y refinados fueron 51,95–50,90 mg/kg, respectivamente. Los valores de estabilidad oxidativa de los aceites de semilla CS crudos y refinados fueron de 2,32–2,69 h, respectivamente. El β -sitosterol y el campesterol fueron los esteroides predominantes. Como resultado del proceso de refinación, aunque disminuyeron el magnesio, potasio, hierro y cobre, aumentaron las proporciones de sodio, aluminio, calcio, cromo, estroncio, rubidio y bario. Los resultados proporcionan datos preliminares para el consumo futuro de aceite CS, en particular como aceite de semilla CS refinado.

PALABRAS CLAVE: *Aceite vegetal crudo y refinado; Alimento; Parámetros de calidad; Propiedades físico-químicas; Semilla de Cephalaria syriaca.*

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1. INTRODUCTION

Climate change, rapid population growth, and other similar factors have increased people's demand for food, and this situation has prompted people to seek new food sources. Consumable vegetable oilseeds, which are important to human nutrition, are among these sources. *Cephalaria syriaca* L. (CS) seeds, which are widely distributed throughout the world and can be an alternative vegetable oilseed source, are part of the Dipsacaceae family. It has been reported that 94 *Cephalaria* species are found in Europe, Eastern Mediterranean, East Asia, and North and Central African countries. This plant grows spontaneously in inefficient, clay and loamy soils and is a single-year weed that is resistant to cold (Göktürk and Sümbül, 2014). It is also found as a weed in wheat cultivation areas. It has been reported that the pelemir seed is generally used as pelemir seed flour after its oil is removed (Karahan and Kılınççeker, 2019). It has been reported that CS seeds, which are similar to wheat seeds, have high protein (14.21%), fat (22.28%) and fiber contents (9–30%). CS seed oil is greenish-yellow with a distinctive fragrance, and it has been reported to be rich in saturated fatty acids, such as myristic, palmitic, and stearic and in oleic and linoleic unsaturated fatty acids (Yıldırım *et al.*, 2019). Kavak and Baştürk (2020) have identified 30 different volatile compounds dominated by aldehydes and alcohols. In addition, there are studies with positive and negative results on various antibacterial, antifungal, antioxidant, and cytotoxic properties (Kırmızıgül *et al.*, 2012; Pasi *et al.*, 2009). These studies focused on the seed flour, which is generally obtained from defatted seeds. It has been stated that the reason for removing the oil was its bitter taste.

In evaluating CS seed oil in terms of vegetable oil technology and nutrition, we found that a detailed study of the composition of the crude and refined CS oil has not been conducted, and that those that were conducted generally focused on the properties and fatty acid composition of the crude oil. In the present study, the physicochemical composition of crude oil from CS seed, which is important in terms of vegetable oil technology, was determined and, different from previous studies on this subject, this CS oil was refined under *in vitro* conditions. Thus, the importance of the crude and refined CS oils in terms of the quality

parameters of vitamin E, oxidative stability, fatty acids, sterol and mineral substance compositions, vegetable oil technology and nutritional properties were determined.

2. MATERIALS AND METHODS

2.1. Material

The seeds of the CS plant used in this study were obtained from the Tekirdağ Province, Turkey, in June 2021 and crude CS oil was extracted using a solvent in the first stage and then refined by neutralizing, bleaching, winterizing, and deodorizing, and the composition of fatty acids, vitamin E values, and sterol and mineral compositions were determined in addition to the physicochemical quality parameters using the methods noted below.

2.2. Methods

2.2.1. Refining process

The chemical refining process was carried out as neutralization, bleaching, winterization and deodorization, respectively. In order to remove gummy matter and soap compounds, CS crude oil was first treated with water, then phosphoric acid/sodium hydroxide at 75 °C was applied and the oil separated to remove impurities (soap stock) to obtain neutralized CS oil. In the bleaching stage, neutralized CS oil was processed with activated carbon and diatomaceous earth under 710 mm Hg vacuum at 90 °C for 40 minutes for the separation of color substances. During the winterization stage, CS oil, which was cooled to 5-6 °C and treated with perlite, was filtered and purified from waxy substances, and volatile components such as aldehydes and ketones were removed by applying deodorization at 220 °C under 3-4 mBar. Finally, refined CS oil was obtained by cooling it to below 40 °C using a plate heat exchanger (De Greyt, 2013).

2.2.2. Physico and chemical methods

Specific gravity, viscosity, flash point, peroxide value, free fatty acidity, iodine value, color, saponification value, unsaponifiable matter, oxidative stability and amount of sterols were analyzed according to AOAC (2000), Lazaridou *et al.* (2004) and Knothe (2006).

2.2.3. Vitamin E analysis

The analytical method developed by Arnaud *et al.* (1991) was used, and the reading process was done in HPLC. The properties of the phase and column used are given below.

Mobile phase: 970 ml hexane + 30 ml 1-4 dioxane. Column: MAXSIL 5 SILICA 250*4.00 mm 5 micron P/NO OOG-0053-DO Phenomenex or Li-crosorb S160-5 micron 25 cm x 4.6 mm. Wavelength in fluorescence detector ex:293, em:326. Flow Rate: 1ml/min. Loop: 20 µl.

2.2.4. Analysis of fatty acid composition

The fatty acid compositions of the oil samples were determined according to AOCS Method No: Ce 1-62 (1998). FAMES were analyzed on a GC-2025 series gas chromatograph (Shimadzu, Japan), equipped with a hydrogen flame ionization detector (FID) and a fused silica capillary column (60 m × 0.25 mm id), coated with 0.20 µm RTX-2330. Analysis conditions were as follows: colon: 180 °C, injection: 200 °C, detector: 200 °C, gas flow rates with carrier gas (N₂): 30 ml/min, combustible gas (H₂): 28 ml/min. Dry air: 220 ml/min. Injection amount: 1 µl. The injector and FID were set at 260 °C. Commercial mixtures of fatty acid methyl esters standard (FAME) mix (Merck-USA) were analyzed under the same operating conditions to determine relative area percentage.

2.2.5. Sterol analysis

The sterol compositions of the oil samples were determined according to Lechner *et al.* (1999). Commercial mixtures of sterol standard mix (Merck-USA) were used.

GC (Gas chromatography) operating conditions were as follows:

Instrument: GC 2025 (Shimadzu-Japan)

Column: Fused silica capillary column (RTX-2330) (60 m × 0.25 mm i.d.; film thickness 0.20 micrometer). Detector: Flame ionizing detector. Carrier gas: Nitrogen. Split ratio: 50:0. Flow rate: 0.80 ml/min. Injection block temperature: 280 °C. Detector temperature: 290 °C. Column temperature: 260 °C. Injection volume: 1 µl.

2.2.6. Oxidative stability analysis

In this study, the induction time was determined with a Metrohm 743 Rancimat device (Methrom)

and rancimat device by using 3 grams of CS seed oil obtained from each period for oxidative stability. Oxidative stability was measured at an airflow rate of 10 l/hour set at 110 degrees. Conductivity of 0.055 µs ultra-pure water was used in the study (Coppin and Pike, 2001).

2.2.7. Determination of mineral contents

The mineral matter compositions of the oil samples were determined by pre-burning, according to Skujins (1998). Inductively Coupled Plasma Atomic Emission was used for mineral matter analyses. Results were calculated as ppm.

Working conditions of ICP-AES Instrument: Alet: ICP-AES (Varian-Vista), Plasma gas (Ar): 20-22.5 l/min in 1.5 l/min, Auxiliary gas (Ar): 0.50-2.25 l/min in 0.75 l/min Signal washout: Four orders (10.000 x)

2.2.8. Statistical Analysis

The data obtained in the study were analyzed using the SPSS (Statistical Package for Social Sciences) Windows 22.0 program. In the evaluation of the data, as descriptive statistical methods, mean standard deviation and quantitative continuous data were compared between two independent groups, One-Way Anova test and Duncan's multiple comparison test were used (Nelson, 1987).

3. RESULTS AND DISCUSSION

As seen in Table 1, the fat content of CS seeds was 20.91%. Some researchers have reported that the oil content of CS seeds is within the range of 11–34% (Hallen *et al.*, 2004). Others have reported that these values may vary according to various conditions, such as soil, climate, and geographical location (Sezgin *et al.*, 2017; Kavak and Baştürk, 2020).

In vegetable oil technology, the % oil content of some seeds is specified as follows; 22–36% for sunflower seeds, 25–37% for safflower and 22-49% for rapeseed (Gökalp *et al.*, 2001). The fat content in CS seeds, compared to that in other vegetable oilseeds, was the same or higher than that in cottonseed and corn (18–20%) seeds.

As seen in Table 1, the physical properties of CS oil, such as density, refractive index, and viscosity values were 0.9258 g/cm³, 1.4708, and 84.4 mPain crude CS seed oil, respectively. These values were

TABLE 1. Physico-chemical quality parameters of crude and refined oils from CS.

Parameter	Crude Oil	Refined Oil
Acidity (%)	0.40±0.01 ^{*a}	0.19±0.02 ^b
Peroxide (%)	4.93±0.10 ^a	3.77±0.15 ^b
Specific Gravity (g/cm ³)	0.925±0.003 ^a	0.926±0.005 ^a
Viscosity	84.40±0.25 ^a	76.90±0.50 ^b
Refractive Index	1.4708±0.002 ^a	1.4707±0.006 ^a
Iodine Number	89.45±0.90 ^b	103,308±1.20 ^a
Saponification Number (mg KOH/g)	196.98±1.40 ^a	151.60±1.32 ^b
Unsaponifiable Substances (g/100g)	1.05±0.06 ^a	0.77±0.08 ^b
Vitamin E (mg/100 g)	51.90±1.15 ^a	50.95±0.91 ^b
Flash Point (°C)	238±2.02 ^b	282±2.66 ^a
Oxidative Stabilité (h)	2.32±0.19 ^b	2.69±0.12 ^a
Total Sterol amount (g/kg)	4.91±0.23 ^a	4.03±0.14 ^b

determined to be 0.9263 g/cm³, 1.4707, and 76.9 mPa, respectively, in refined CS seed oil. According to these results, the change in the specific gravity and refractive index of CS oil was not statistically significant at $p < 0.05$ as a result of the refining process, while a significant change was found in viscosity values. Yazıcıoğlu *et al.* (1978) reported that the specific gravity and refractive index values of crude CS oil is 0.9229 g/cm³ and 1.4706, respectively. The specific gravity value is one of the properties that provides general information about the sources of oils. Accordingly, it was observed that the specific gravity and refractive index values of the crude and refined oils from CS seeds were comparable to those of other vegetable oils (0.910–0.930 g/cm³ and 1.4670–1.4750, respectively).

Viscosity is resistance to flow and is also an important parameter in vegetable oil technology. Often, the viscosity of oils containing high molecular weight fatty acids is higher than that of low molecular weight oils. There was no reported value related to the viscosity of CS seed and refined oils, which was observed to be comparable to that of canola oil (78.8 cP) compared to other vegetable oils (Nouredidini *et al.*, 1992). In general, it was determined that the physical properties of CS seed oil after the refining process were statistically different from before the refining process, except for its specific gravity and refractive index ($p < 0.05$) (Table 1).

As seen in Table 1, the chemical properties of CS seed oil, such as free fatty acidity, peroxide, iodine number, saponification number, and unsaponifiable substance number values, were 0.40%, 4.93 meqO₂/kg, 89.45, 196.98 mg KOH/g, and 1.05 g/100 g in crude CS oil, respectively, while these values were 0.19%, 3.72 meqO₂/kg, 103.308, 151.6 mg KOH/g, and 0.77, respectively, in refined CS oil. With the refining process, it was determined that the free fatty acidity and peroxide values of crude CS oil decreased and the iodine value increased. Kavak and Baştürk (2020) have determined that the free fatty acidity and peroxide values of CS seed crude oil obtained from different locations is 0.27–0.83% and 2.46–5.39 meqO₂/kg, respectively. It was observed that the crude and refined free fatty acidity and peroxide values of CS seed oil were lower than those of sunflower, corn, and canola seed crude oils, which are commonly used vegetable oils.

Yazıcıoğlu *et al.* (1978) have reported that in CS crude oil, the iodine value is 88.4, saponification number is 192 mg KOH/g, and unsaponifiable substance amount is 1.24 g/100 g. The iodine value in sunflower, corn, canola, hazelnut, and cotton varies between 99 and 141.29. The iodine value in refined CS seed oil is comparable to that in other vegetable oils. On the other hand, the saponification number values in vegetable oils such as sunflower, corn, canola, hazelnut and cotton were between 187 and 195 mg/g. It was determined that the saponification values in CS seed crude oil were comparable with those in other vegetable oils, and that in the refined CS seed oil they were lower than in other vegetable oils. In terms of unsaponifiable substance, crude and refined oils from CS seeds could be a new source for food and industrial use.

As seen in Table 1, the vitamin E values of crude and refined oils from CS seeds were 51.95 and 50.90 mg/kg, respectively. It was also determined that vitamin E decreased as a result of the refining process. Kavak and Baştürk (2020) have stated that the vitamin E values in the crude oil from CS seeds, which they collected from different locations, were between 54.0 and 467.0 mg/kg. The vitamin E amounts in other refined vegetable oils have been reported in corn (3.11–4.46 mg/kg), soybean (1.19–1.42 mg/kg), sunflower (9.52–11.4 mg/kg), and canola (3.82–4.95 mg/kg) (Castelo-Branco *et al.*, 2016). It was determined that the vitamin E contents in the crude and

TABLE 2. Color values for CS crude and refined oils.

Oil	Red	Yellow	Blue	Dark
Crude Oil (1 st)	2.8±0.1 ^{*a}	70.0±0.2 ^a	0	0
Refined Oil (5,25 th)	1.3±0.1 ^b	12.3±0.1 ^b	0	0

CS: *Cephalaria syriaca* L. seed; * Mean ± standard deviation; One-way ANOVA analysis was determined (n=3) by Duncan's multiple range tests.

Degree of significance: ^{a-b}p-value < 0.05;

In each column, means with different letters are significantly different.

refined oils from CS seeds were higher than in other vegetable oils, and in this respect, it was considered a good source for oxidation resistance, oil stability, and health.

The color values for CS seed oil are provided in Table 2. The redness, yellowness, and blueness values of the crude oil obtained from these seeds varied at 2.8 R, 70.0 Y, 0 B (1st), respectively; while the redness, yellowness and blue values of refined rosemary seed oil were 1.3 R, 12.3 Y, 0 B (5,25th), respectively. As a result of the refining process, the level of redness and yellowness statistically decreased (p < 0.05) after bleaching. Kavak and Baştürk (2020) have found that the L*, a*, and b* values of crude oil from CS seeds is 18.63/24.87, -1.01/2.37, and 4.73/13.32, respectively. There have been no studies on the color values of refined oil from CS seeds. Compared to other vegetable oilseeds, it was determined that the color values for crude and refined CS seed oil were as good as those of edible vegetable oils, such as sunflower, canola, and hazelnut (Duman and Özcan, 2020).

As seen in Table 3, 14 types of fatty acid components were determined in the crude and refined CS oil—myristic (21.06–11.80%), palmitic (10.8–8.91%), stearic (2.26–2.70%), oleic (29.17–34.24%), linoleic (35.56–40.57%), linolenic (0.17–0.24%), arachidic (0.32–0.51%), and erucic (0.03–0.05%). In other studies on CS crude oil, the fatty acids were myristic (14.60–17.30%), palmitic (9.41–23.81%), oleic (28.10–33.20%), and linoleic (10.28–36.9%). Sarikahya *et al.* (2013), Sarikahya *et al.* (2015), Bretagnolle *et al.* (2016) and Kavak and Baştürk (2020) reported that CS crude oil had similar properties to those of babassu oil (11–27%) and coconut oil (16–21%) in terms of myristic acid composition and to corn oil in terms of the composition of other fatty acids. In studies on the physico-chemical changes

TABLE 3. Fatty acid compositions of CS crude and refined oils.

Fatty acids	Crude Oil	Refined Oil
Myristic	21.06±1.25 ^{*a}	11.80±1.05 ^b
Palmitic	10.80±1.02 ^a	8.91±0.40 ^b
Palmitoleic	0.28±0.05 ^a	0.23±0.03 ^b
Margaric	0.07±0.01 ^a	0.06±0.02 ^b
Heptadecanoic	0.08±0.02 ^a	0.03±0.01 ^b
Stearic	2.26±0.45 ^b	2.70±0.80 ^a
Oleic	29.17±2.05 ^b	34.24±2.25 ^a
Linoleic	35.56±2.50 ^b	40.57±2.75 ^a
Linolenic	0.17±0.03 ^b	0.24±0.04 ^a
Arachidic	0.32±0.02 ^b	0.51±0.05 ^a
Eicosanoic	0.16±0.04 ^b	0.27±0.07 ^a
Behenic	0.02±0.01 ^b	0.37±0.10 ^a
Erucic	0.03±0.01 ^b	0.05±0.02 ^a
Lignoceric	0.03±0.02 ^a	0.02±0.01 ^b

CS: *Cephalaria syriaca* L. seed; * Mean ± standard deviation; One-way ANOVA analysis was determined (n=3) by Duncan's multiple range tests. Degree of significance: ^{a-b}p-value < 0.05; In each column, means with different letters are significantly different.

that occur during the refining stages of various oils (crude and refined canola, cotton seed, peanut, sunflower, soybean oils), it has been reported that there may be changes in the composition of saturated and unsaturated fatty acids with tendencies to increase and decrease. It has been stated that this may be due to the type of refining process (physical or chemical refining) and the variety of parameters in the stages (El-Mallah *et al.*, 2011; Mohdaly *et al.*, 2017; Shah *et al.*, 2018; Özcan *et al.*, 2021).

As seen in Table 4, the total sterol values for crude and refined CS oil was 4.91–4.03 g/kg, which were close to sunflower, corn, canola, hazelnut and cotton seed oils (2–5 g/kg) in terms of total sterol amount (Lavedrine *et al.*, 1997; Weststrate and Meijer, 1998; Cercaci *et al.*, 2003; Yıldırım *et al.*, 2019). Examining the nutritional and crude and refined CS oil in terms of sterol composition, these amounts found in CS crude and refined oils showed that β-sitosterol and campesterol were dominant and that these sterol values were 71.41–67.65% and 15.91–16.06%, respectively. It was determined that CS oil was similar to palm kernel oil in terms of sterol composition. As a result of the refining process, it was determined that while the β-sitosterol, Δ-stigmasterol, and stigmasterol levels in CS oil decreased, the level of

TABLE 4. Sterol compositions of CS crude and refined oils.

Sterol Composition (%)	Crude Oil	Refined Oil
β -Sitosterol	71.41 \pm 2.25 ^{*a}	67.65 \pm 2.00 ^b
Brassicasterol	nd	nd
Campesterol	15.91 \pm 0.90 ^b	16.06 \pm 0.75 ^a
Δ -7-Stigmasterol	1.08 \pm 0.04 ^a	0.87 \pm 0.08 ^b
Cholesterol	nd	nd
Stigmasterol	1.80 \pm 0.01 ^a	1.72 \pm 0.02 ^b

CS: *Cephalaria syriaca* L. seed; nd: Not determined; * Mean \pm standard deviation; One-way ANOVA analysis was determined (n=3) by Duncan's multiple range tests. Degree of significance: ^{a-b}p-value < 0.05; In each column, means with different letters are significantly different.

campesterol increased. These substances, which are among the most important minor components in oils, play an important role in lowering serum cholesterol levels.

As seen in Table 5, 11 minerals were detected in the crude and refined CS oils. The dominant minerals were sodium, magnesium, potassium, calcium, and iron (Na, Mg, K, Ca, and Fe, respectively). As a result of the refining process, although the mineral substances of Mg, K, Fe, and copper (Cu) decreased, the ratio of Na, aluminum (Al), Ca, chromium (Cr), strontium (Sr), rubidium (Rb), and barium (Ba) in-

TABLE 5. Mineral matter compositions of CS crude and refined oils.

Mineral (ppm)	Crude Oil	Refined Oil
Sodium	11.56 \pm 0.10 ^{*b}	47.10 \pm 0.80 ^a
Magnesium	26.96 \pm 1.10 ^a	14.18 \pm 1.01 ^b
Aluminum	3.58 \pm 0.08 ^b	4.32 \pm 0.05 ^a
Fluorine	52.40 \pm 1.25 ^a	34.66 \pm 1.10 ^b
Calcium	27.60 \pm 1.10 ^b	35.93 \pm 1.15 ^a
Chromium	0.01 \pm 0.01 ^b	0.06 \pm 0.01 ^a
Manganese	0.03 \pm 0.01	nd
Iron	4.25 \pm 0.09 ^a	2.65 \pm 0.06 ^b
Copper	1.46 \pm 0.02	nd
Strontium	0.19 \pm 0.04 ^b	0.32 \pm 0.03 ^b
Barium	nd	0.05 \pm 0.01

CS: *Cephalaria syriaca* L. seed; nd: Not determined; * Mean \pm standard deviation; One-way ANOVA analysis was determined (n=3) by Duncan's multiple range tests. Degree of significance: ^{a-b}p-value < 0.05; In each column, means with different letters are significantly different.

creased. In the literature, there is no information on the mineral composition of CS crude or refined oils. In this respect, our data are the first on this subject. On the other hand, in terms of mineral substance composition, it was determined that CS oils contained lower amounts of minerals at the ppm level compared to that of olive oil (Sayago *et al.*, 2018). According to these results, crude and refined CS oils, which have minerals which are beneficial for human nutrition, can be consumed for the development of bones and teeth and for muscle and nervous system functions. In addition, refined CS oil appears to be a good source of micro and macro minerals as a food ingredient for human nutrition.

4. CONCLUSIONS

As can be seen in the literature, this research is the first work on the refining of crude CS oil. The results obtained from the present study provide important data for future consumption of CS oil, especially for its refined product. It was determined that all physical and chemical properties (except specific gravity and refractive index) in the crude oil from CS seeds changed as a result of the refining process (p < 0.05). It was also determined that CS seeds, in addition to having an average oil ratio comparable to that of other oilseeds, such as sunflower, corn, canola, hazelnut oils, can be refined. It can be a new source of oilseed raw material. In addition to the determined quality properties of the refined oil from CS seeds, it was determined that it is a good source of new crude materials in terms of vitamin E, essential fatty acids, minerals, and sterol composition for use in cosmetics and human nutrition. In future studies, it is recommended that biotechnological studies be conducted on the seeds to reduce the toxicity of myristic acid.

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