CHAPTER IV

EFFECTS of INDUSTRIAL REFINING STAGES ON CORN OIL's QUALITY AND BIOACTIVE COMPONENTS DETERMINED USING GAS CHROMATOGRAPHY AND INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY

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1. Introduction

To be able to use crude corn oil as an edible food, its ingredients, including free fatty acids, coloring agents, waxes, aldehydes, and ketones, must be extracted and the oil must then be refined. Refining is the process by which adhesive agents, soap, color substances, wax, aldehydes, and ketones in the crude oil are removed through treatments that neutralize, bleach, winterize, and deodorize the oil (El-Mallah, El-Shami, Hassanien, & Abdel-Razek, 2011: Yemişçioğlu, Özdikicierler, & Gümüşkesen, 2016). During the chemical refining process, after removing the adhesive agents in the crude oil with water, the free fatty acidity in the oil is decreased by adding phosphoric acid and caustic, and then the color of the oil is lightened by bleaching the soil. Pearlite is then added while cooling to winterize the product. Finally, a vacuum is used at high temperatures to separate the aldehydes and ketones to deodorize the oil (Ortega-Garcia et al., 2006: Yıldırım, Erdoğdu, & Yorulmaz, 2019).

The adhesive agents in the corn oil are composed of phospholipids; the coloring agents are carotene, xanthophyll, and chlorophyll. Waxy substances are formed by stearins, and effluvial substances are composed of aldehydes and ketones (Hamm, & Hamilton, 2000). It has been reported that pressure, temperature, steam flow, and time parameters applied during the refining process cause reductions in the oil's components (e.g.,

tocopherols, sterols, and vitamins) (Ruiz-Mhdez, Mirquez-Ruiz, & Dobarganes, 1997).

Viscosity, one physical property of vegetable oils, is an important parameter of the rheological properties in fluid foods such as oils, as well as in the design and process of reactors, and pipes (Abramovic, & Klofutar, 1998: Rodenbush, Hsieh, & Viswanath, 1999). Moreover, the fatty acid composition of vegetable oils is significant for human health and changes during the refining processes (Mohdaly et al., 2017). Some of these that affect health are oleic, linoleic, and linolenic unsaturated fatty acids, which are also available in corn oil (Karaca, ve Aytaç, 2011).

Furthermore, phytosterols, which are bioactive components in vegetable oils, protect the oils against oxidation and are also beneficial for human health. Phytosterols have antibacterial, antifungal, anti-inflammatory, and antitumor properties, and are also used to determine adulterations by using the sterol composition of vegetable oils (Yıldırım, Erdoğdu, & Yorulmaz, 2019). In the neutralizing stage of refining, the sterols are substantially the non-saponifiers (Ferrari et al., 1996). The aim of this study was to determine the effects of the chemical refining stages (neutralizing, bleaching, winterizing, and deodorizing) on the quality characteristics and bioactive components of corn oil, such as free fatty acidity, peroxide value, iodine number, viscosity, color, saponification value, and the amount of unsaponifiable matter, and the composition of fatty acids, sterols, and minerals.

2. Materials and Methods

2.1. Materials

The corn oil samples used in this study were obtained from an oil refinery that is operated as a continuous system of neutralizing, bleaching, winterizing, and deodorizing stages, respectively, to the same samples of crude oil. Our study investigated the changes of nine properties comprising seven quality characteristics and different bioactive components in each of the five samples studied with three parallels after each stage.

2.2. Methods

2.2.1. Physico-Chemical Analyses

Free fatty acidity, peroxide value, viscosity, saponification value, and the amount of unsaponifiable matter in the corn oil samples were analyzed according to American Oil Chemists' Society (AOCS) methods (Anonymous, 1990).

2.2.2. Color Analyses

The corn oil samples used were filtered through filter paper and readings were done on a Lovibond PFX 880L tintometer using 1- and 5.25-inç cuvettes (Mehlenbacher, Walker, Walker R., & Link, 1985).

2.2.3. Analyses of Fatty Acıd Composition

The fatty acid compositions in the corn oil samples were determined based on the AOCS method. The samples were first treated with potassium hydroxide and n-heptane to esterify them after which 1 µL was taken from the esterified samples. Gas chromatography (GC) using the Shimadzu GC-2025 (Shimadzu Corporation, Kyoto, Japan) was applied to determine the fatty acid composition after each refining stage under the following conditions (Hışıl, 2004): flame ionization detector, RTX-230; column, 60 m long with an inner diameter of 0.25 mm; and film thickness, 0.20 µm. The column temperature was 180°C, the injection temperature was 200°C, and the detector temperature was 200°C. The flow of gases was as follows: carrier gas (N2), 30 mL/min; combustible gas (H2), 28 mL/min; dry air, 220 mL/min; and injection, 1 µL.

2.2.4. Sterol Analyses

The sterol composition in each corn oil sample was determined using the method of Lencher et al. (1999). The Shimadzu GC-2025 gas chromatography system (HP-5, column length, 30 m, column diameter, 0.25 mm, and film thickness, 0.25 um) was used. The flow rate of all gases was set at 45 mL/min. The column temperature was first increased from 0 to 60°C, then from 60 to 220°C, and the results were read at 220°C.

2.2.5. Mıneral Substance Analyses

Corn oil samples (0.5 g) were put into the heating container after which 15 mL pure HNO3 was added and heated to 200°C in a microwave oven. The solution was then diluted with 100 mL ultrapure water and filtered through ashless filter paper (Macheres-Nagel MN 640W, black band, 110-mm diameter). The concentrations of the mineral substances were determined by reading the prepared samples on the ICP-AES (Varian Vista) (Skujıns, 1998).

2.2.6. Statistical Analyses

The data obtained in the study were analyzed using SPSS ver. 22.0 (IBM Corp., Armonk, NY, USA). In evaluating the data, the mean ± standard deviation was used as the descriptive statistical result and the one-way analysis of variance and Duncan's multiple range test were used to compare quantitative continuous data between more than two independent groups (Püskülcü, & İkiz, 1998: Soysal, 1998).

3. Results and Discussion

The quality characteristics of the chemically refined corn oil are shown in Table 1. As seen in the table, free fatty acid content varied between 2.50 (crude oil) and 0.08% (deodorized oil) and peroxide values ranged from 3.13 meq O2/kg (crude oil) to 0 meq O2/kg (deodorized oil). We determined that the free fatty acid levels significantly decreased after the neutralizing stage and the peroxide value considerably decreased after the deodorizing stage. In a study by Aksoy (2015) the acidity in crude corn oil was determined to be 2.20, 0.12, 0.10, and 0.09% after the neutralizing, bleaching, winterizing, and deodorizing stages, respectively. In addition, Jung Yoon and Min (1989), Young (1983), Loft (1990), and Moo et al. (1995) have reported that free fatty acid levels and peroxide values decreased after vegetable oils were refined. It has been reported that the amount of oil loss increased with increasing free fatty acids during the refining process and the rancidity increased with increasing peroxide values (Tekin, 2005).

Table 1. Changes in the Quality Characteristics of Corn Oil During Chemical Refining.

Process Stages	**Acidity (%)**	**Peroxide value ($meqO_2/kg$)**	**Iodine number ($mgI_2/100 g$)**	**Saponification (mgKOH/g)**	**Amount of unsaponifiable material (mgKOH/g)**
Crude oil	$2.50^{a} \pm 0.03$	$3.13^{a} \pm 0.30$	$119.94^{c} \pm 0.71$	$207.48^{a} \pm 0.03$	$1.91^{a} \pm 0.03$
Neutralizing	$0.09^{d} \pm 0.03$	$2.58^{b} \pm 0.20$	$120.28^{b\,c} \pm 0.45$	$206.19^{b} \pm 0.02$	$0.69^{d} \pm 0.02$
Bleaching	$0.11^{c} \pm 0.02$	$1.54^{c} \pm 1.00$	$122.97^{b} \pm 0.29$	$204.89^{c} \pm 0.01$	$1.29^{c} \pm 0.01$
Winterizing	$0.14^{b} \pm 0.01$	$0.92^{d} \pm 0.20$	$122.26^{b} \pm 0.62$	$203.52^{d} \pm 0.03$	$1.40^{b} \pm 0.02$
Deodorizing	$0.08^{d} \pm 0.02$	$0.0^{e} \pm 0.00$	$124.29^{a} \pm 0.88$	$202.88^{e} \pm 0.04$	$0.23^{e} \pm 0.03$
Crude oil	$2.50^{a} \pm 0.03$	$3.13^{a} \pm 0.30$	$119.94^{c} \pm 0.71$	$207.48^{a} \pm 0.03$	$1.91^{a} \pm 0.03$
Neutralizing	$0.09^{d} \pm 0.03$	$2.58^{b} \pm 0.20$	$120.28^{b\,c} \pm 0.45$	$206.19^{b} \pm 0.02$	$0.69^{d} \pm 0.02$
Process Stages	**Viscosity (Pa-s)**	**Color**			
		Red	**Yellow**	**Blue**	**Dark**
Crude oil	$8.16^{a} \pm 0.30$	9.8^{a} (1″)	70.0^{a}	0.0^{a}	0.9^{a}
Neutralizing	$7.25^{b} \pm 0.20$	3.6^{c} (1″)	70.0^{a}	0.0^{a}	0.1^{bc}

Bleaching	$7.13^{c} \pm 0.10$	5.4^{b} (5.25″)	70.0^{a}	0.0^{a}	0.0^{c}
Winterizing	$7.13^{c} \pm 0.20$	5.5^{b} (5.25″)	70.0^{a}	0.0^{a}	0.2^{bc}
Deodorizing	$6.84^{d} \pm 0.02$	3.1^{d} (5.25″)	46.0^{b}	0.0^{a}	0.6^{b}

Notes: $^{a–e}$ values marked with different letters are statistically different from each other ($p < 0.005$).

The viscosity of corn oil after refining varied between 8.16 (crude oil) and 6.84 Pa-s (deodorized oil) at 22°C. The highest change in viscosity was observed at the end of the neutralizing stage, and the crude corn oil became more fluid than that after this stage. In a study conducted by Noureddini et al. (1992) viscosity levels changed in the refined corn oil with temperature changes and were reported to be 52.3, 30.8, 22.7, 15.7, and 9.28 Pa-s at 23.9, 37.8, 48.9, 60, and 62.2ºC, respectively. In the same study, the viscosity values for refined soybean oil were determined to be 54.3, 31.8, 23.3, 16.1, and 9.51 Pa-s, respectively, at the same temperatures. In another study on the chemical refining process of sunflower oil, viscosity levels were reported to be 7.03, 7.18, 8.00, 6.67, and 6.58 Pa-s for crude, neutralized, bleached, winterized, and deodorized oils, respectively (Ergönül, 2013).

Color is an important parameter in vegetable oil technology. The color values of refined corn oil were determined to be 9.8 red, 70.0 yellow, and 0.9 dark for crude oil and 3.1 red, 46 yellow, and 0.6 dark for deodorized oil; the color of the corn oil was the lightest at the end of bleaching stage. On the other hand, Aksoy (2015) has examined the changes in red corn oil during the refining stages and reported it as 16 in crude oil, 9 after neutralizing, 5.2 after bleaching, 5 after winterizing, and 2.7 after deodorizing.

The saponification number for corn oil varied between 207.48 (crude oil) and 202.88 mg KOH/g (deodorized oil) and decreased gradually during the refining process. The amount of unsaponifiable material increased and decreased within a range of 1.91 (crude oil) and 0.23 g/kg (deodorized oil) depending on the refining stage. The highest decrease in the amount of unsaponifiable corn oil was observed during the deodorizing stage. It was stated that during vegetable oil refining, the free fatty acid, peroxide value, and saponification number decreased, while the amount of unsaponifiable material increased from the crude to the refined oil (Onyema, & Ibe, 2016).

As seen in Table 2, when examining the changes in fatty acid composition during the corn oil refining, the total saturated fatty acids varied between 13.77 (crude oil) and 12.44% (deodorized oil), and total unsaturated fatty acids varied between 86.22 (crude oil) and 87.55%

(deodorized oil). Palmitic and stearic saturated fats and oleic and linoleic unsaturated fats were the dominant fatty acids in corn oil. It was determined that the fatty acid composition of the corn oil did not change with refining. In their study on corn oil, Ferrari et al. (1996) have determined that the palmitic acid concentration is 13.1, 12.4, 12.8, and 12.5% at the neutralizing, bleaching, winterizing, and deodorizing stages, respectively. The palmitoleic acid content was calculated as 0.2% at all stages of refining. Among the unsaturated fatty acids, the amount of oleic acid was determined to be 34.1% in the crude oil and 34.6, 34.6, 34.9, and 34.8% at the neutralizing, bleaching, winterizing, and deodorizing stages respectively.

Table 2. Changes in the Fatty Acid Concentrations of Corn Oil During Chemical Refining.

SFA	**Crude oil (%)**	**Neutralizing (%)**	**Bleaching (%)**	**Winterizing (%)**	**Deodorizing (%)**
Myristic	0.03	0.03	0.03	0.03	0.04
Palmitic	11.15	11.08	10.01	10.27	9.44
Stearic	1.98	1.97	2.33	2.23	2.41
Arachidic	0.43	0.43	0.38	0.39	0.35
Lignoceric	0.18	0.15	0.16	0.16	0.17
∑	13.77[a]	13.67[a]	12.94[b]	13.10[ab]	12.44[c]
MUFA					
Palmitoleic	0.06	0.06	0.06	0.06	0.05
Heptadecanoic	0.04	0.04	0.03	0.04	0.03
Oleic	33.23	33.22	31.38	31.91	30.49
Nervonic	0.01	0.01	0.01	0.01	0.01
PUFA					
Linoleic	52.70	52.78	55.25	54.59	56.58
Linolenic	0.01	0.09	0.09	0.08	0.01
Cis-11.14-eicosadienoic	0.09	0.12	0.25	0.21	0.32
Arachidic	0.05	0.05	0.05	0.05	0.04
∑	86.22[c]	86.32[bc]	87.06[a]	86.90[b]	87.55[a]

Notes: [a–c] values marked with different letters are statistically different from each other ($p < 0.005$).

SFA: Saturated Fatty Acids, MUFA: Monounsaturated Fatty Acids, PUFA: Polyunsaturated Fatty Acids

It has been reported in studies conducted on corn and other vegetable oils that there is no significant change in the fatty acid composition during the refining process (Wilding, Rice, & Mattil, 1963: Morrison, & Robertson, 1978: Jinsuk, & Seung, 1993: Çalışkan, 2008: Aksoy, 2015). On the other hand, it has been reported that the total amount of monounsaturated fatty acids increases and the amount of saturated fatty acids decreases as a result of the refining process (Taşan, 1999: Mohdaly et al., 2017).

Table 3. Changes in the Sterol Composition of Corn Oil During Chemical Refining.

Sterol Composition	Crude Oil	Neutralizing	Bleaching	Winterizing	Deodorizing
1-Campesterol (%)	25.87^{b}	27.25^{a}	21.73^{c}	25.46^{b}	9.00^{d}
2-Stigmasterol (%)	6.46^{c}	6.92^{b}	6.13^{c}	6.76^{b}	12.63^{a}
3-Δ5.23-Stigmastadienol (%)	nd.	nd.	nd.	nd.	0.80^{a}
4-Cholesterol (%)	0.45^{c}	0.43^{c}	0.45^{c}	0.53^{b}	0.84^{a}
5-β-sitosterol (%)	67.21^{b}	65.39^{c}	61.25^{d}	67.22^{b}	71.87^{a}
6- Δ5-Avenasterol (%)	nd.	nd.	4.01^{a}	nd.	nd
7- Δ5.24-Stigmastadienol (%)	nd.	nd.	0.60^{a}	nd.	nd.
8- Δ7-Stigmastenol (%)	nd.	nd.	5.08^{a}	nd.	4.82^{b}
9- Δ7-Avenasterol (%)	nd.	nd.	0.72^{a}	nd.	nd.

Notes: $^{a–d}$ values marked with different letters are statistically different from each other ($p < 0.005$). nd. Not detected.

As seen in Table 3, campesterol, stigma sterol, cholesterol, and ß-sitosterol were determined at all stages of the refining process and that Δ5.23-stigmastadienol, Δ5-avenasterol, Δ5.24-stigmastadienol, Δ7-stigmastenol, and Δ7-avenasterol were determined in different amounts at different stages of the refining process. The concentration of ß-sitosterol was highest, changing from 67.21 (crude oil) to 71.87% (deodorized oil). The chemical refining process was statistically ($p < 0.005$) effective on the sterol composition of the corn oil. The studies in the literature have indicated that the refining process has affected the sterol composition of vegetable oils. The results of a study conducted on corn, soybean, and rapeseed oil showed that the total amount of sterols in all three oils decreases by 36, 18, and 24%, respectively, as a result of the refining process (Ferrari et al., 1996).

Table 4. Changes in the Mineral Composition of Corn Oil During Chemical Refining.

Mineral	Crude Oil (ppm)	Neutralization (ppm)	Bleaching (ppm)	Winterization (ppm)	Deodorization (ppm)
Li	0.028^{a}	0.000^{b}	0.000^{b}	0.000^{b}	0.000^{b}
B	0.000^{c}	0.040^{b}	0.110^{a}	0.123^{a}	0.080^{b}
Na	0.000^{e}	764.74^{a}	109.61^{d}	141.75^{b}	130.68^{c}
Mg	16.693^{a}	6.773^{b}	2.145^{c}	1.976^{d}	1.790^{e}
Al	0.000^{e}	0.446^{d}	1.133^{c}	2.212^{a}	1.163^{b}
Si	20.312^{e}	37.30^{d}	39.15^{c}	43.84^{a}	39.78^{b}
P	165.41^{e}	1029.32^{d}	1084.56^{b}	1202.45^{a}	1046.08^{c}
K	19.451^{a}	0.000^{b}	0.000^{b}	0.000^{b}	0.000^{b}
V	0.000^{b}	0.001^{a}	0.000^{b}	0.001^{a}	0.00^{b}
Cr	0.000^{e}	0.018^{d}	0.040^{b}	0.066^{a}	0.039^{c}
Mn	0.283^{a}	0.062^{c}	0.081^{b}	0.081^{b}	0.043^{d}
Co	0.000^{c}	0.000^{c}	0.002^{b}	0.011^{a}	0.002^{b}
Cu	0.045^{b}	0.000^{c}	0.000^{c}	0.049^{b}	0.071^{a}
Ga	0.000^{a}	0.000^{a}	0.000^{a}	0.001^{a}	0.001^{a}
As	0.001^{c}	0.001^{c}	0.011^{b}	0.020^{a}	0.013^{b}

Se	12.94[b]	12.07[b]	11.99[c]	14.40[a]	11.98[c]
Rb	0.006[a]	0.000[a]	0.000[a]	0.000[a]	0.000[a]
Sr	0.018[b]	0.022[a]	0.024[a]	0.023[a]	0.025[a]
Pd	0.000[b]	0.000[b]	0.000[b]	0.002[a]	0.000[b]
Ag	0.000[c]	0.000[c]	0.000[c]	0.021[a]	0.003[b]
Cd	0.054[a]	0.027[b]	0.000[c]	0.000[c]	0.000[c]
In	0.000[d]	0.000[d]	0.048[b]	0.063[a]	0.043[c]
Sn	0.000[e]	4.998[d]	32.42[b]	40.34[a]	25.78[c]
Sb	0.000[e]	0.002[b]	0.002[b]	0.021[a]	0.001[c]
Te	0.000[b]	0.008[a]	0.000[b]	0.000[b]	0.000[b]
Ba	0.000[e]	0.004[c]	0.022[b]	0.046[a]	0.024[b]
Pb	0.000[d]	0.000[d]	0.005[c]	0.055[a]	0.014[b]

Notes: [a-e] values marked with different letters are statistically different from each other ($p<0.001$).

As seen in Table 4, the elements with the highest composition in the corn oil samples were Mg, Si, P, K, and Se as a result of the chemical refining process and their compositions were 16.693 (crude oil) and 1.790 ppm (deodorized oil) for Mg, 20.312 (crude oil) and 39.78 ppm (deodorized oil) for Si, 165.41 (crude oil) and 1046.08 ppm (deodorized oil) for P, 19.451 (crude oil) and 0 ppm (deodorized oil) for K, and 12.94 (crude oil) and 11.98 ppm (deodorized oil) for Se; therefore, the chemical refining process was statistically significant ($p < 0.001$) on the mineral composition in the corn oil. Ostric et al. (1980) have reported that the amounts of Fe and Cu in sunflower oil are 0.75 and 0.70 ppm after neutralizing and 0.70 and 0.63 ppm after bleaching, respectively. Aksoy (2015) has examined Na, Mg, K, P, Ca, and Fe levels based on the refining stages and determined them to be 37.75, 21.15, 12.97, 107.36, 107.48, and 4.68 ppm, respectively, in the crude oil. These values were found to be 9.84, 1.12, 1.14, 1.04, 31.13, and 3.12 ppm, respectively, after the neutralizing stage; 9.69, 0.29, 6.38, 0.91, 36.61, and 1.46 ppm, respectively, after the bleaching stage; 8.24, 0.91, 10.27, 1.09, 54.80, and 0.77 ppm, respectively, after the winterizing stage; and 8.26, 1.38, 9.82, 0.20, 8.11, and 0.54 ppm, respectively, after the deodorizing stage. Several researchers who have determined the effect of the refining process on the mineral composition of corn and other oils (Crapiste, Brevedan, & Carelli, 1999: Dimic, Karlovic, & Turkulov 1994: Kamyshin & Derevyanko, 1972:

İskander, 1993: Yüksel, 2010: Lepri et al., 2011: De Leonardis, Macciola, & Felice 2000) have indicated that the levels of minerals in the oils change with the refining process and that this change is the result of the chemical structure of the oilseed, the contents of the wash water, and the process applied during refining.

During the refining stages of the corn oil, acidity, peroxide value, viscosity, color, and saponification number decreased, while the number of unsaponifiable materials increased or decreased depending on the refining stage. No dominant changes in fatty acid composition were determined. During the refining stages, palmitic, stearic, and oleic acids were observed in higher amounts in the corn oil compared to levels of other fatty acids. Sterol levels in the corn oil varied depending on the refining stage. Accordingly, stigmasterol and ß-sitosterol contents increased, while campesterol decreased. Mg, K, Mn, Se, and Rb concentrations decreased in the refined corn oil, while Na, Al, Si, P, Cr, Sr, and Ba increased. According to our results, the chemical refining process had different effects on the quality and bioactive components of corn oil, such as the acidity, peroxide value, viscosity, color, saponification number, and on the number of unsaponifiable materials, sterols, and minerals.

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