RESEARCH ARTICLE

Effect of different extraction methods and refining steps on fatty acid distributions of triacylglycerol and physicochemical property of sesame oil

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Sesame oils are subjected to different extraction methods including compression, aqueous extraction, and refining that includes degumming, alkali refining, bleaching, and deodorizing. This research investigated the fatty acid distribution and composition of triacylglycerols (TAGs) in the sesame oils and the acid value (AV), peroxides value (PV), sesamin, sesamolin, and total tocopherols. The results showed that sesame oil obtained by aqueous extraction had a higher content of sesamin, sesamolin, and tocopherols but lower free fatty acids (FFA) and peroxides. Aqueous extraction of sesame oil had the highest percentage of polyunsaturated fatty acids (PUFA) and lowest content of saturated fatty acids (SFA). Refining sesame oil had the lowest and the highest contents of PUFA and SFA, respectively. After different extraction methods and refining steps, the fatty acids' positional distribution's main specifications were retained. The composition of TAGs showed no significant difference between the compression and aqueous extraction of sesame oil, while the TAGs composition of refining sesame oil had a small but significant difference in comparison to the compression and aqueous extraction of sesame oil (P < 0.05) because of the high temperature in refining. After refining saturated-unsaturated-saturated triacylglycerol (SUS), saturated-unsaturated-unsaturated triacylglycerol (SUU) decreased, while saturatedsaturated-unsaturated triacylglycerol (SSU) and unsaturated-saturated-unsaturated triacylglycerol (USU) increased. This research confirmed that the aqueous extraction method produced the best quality and the highest nutritional value sesame oil, while the extraction and refining sesame oil by pressing method had the lowest nutritional value. In addition, analyzing the composition of fatty acids and TAGs could serve as a basis for determining the production process of sesame oil.

Keywords: sesame oil; extraction methods; refining steps; physicochemical property; fatty acid distribution; triacylglycerol.

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Introduction

Sesame oil has been widely consumed because of its characteristic flavor and richest source of nutrients such as sesamin, sesamolin, and tocopherols. Many studies have indicated that sesamin or sesamolin have diverse functions including anticancer, lowering plasma cholesterol levels, and potentiating tocopherols' antioxidant activity [1, 2]. Another nutritional component group in sesame oil is tocopherols, which is important since their antioxidant features preserve required fatty acids against antioxidants [3]. Factors like alkali, heat, light, and metal contaminants decrease the content of tocopherols. Oil exposure to air and heat can also lead to its reduction. Even though the nutritional components are minor, they give special information on the identity, properties, and processing phases of oil [4].

Oleic acid (38.2 -47.3%) and linoleic acid (31 -40.9%) were the prevailing unsaturated fatty acids of sesame oil, constituting about 80% of that. Human bodies cannot synthesize these essential fatty acids [5]. The contents of palmitic and stearic acids are low, ranging from 8.85 to 12.0% and 6.3 to 8.9%, respectively [6, 7]. Several studies showed that full-linoleic acid diets have decreased serum cholesterol by 25% compared to pre-study values on conventional American diets [8]. In sesame oil, triacylglycerols (TAGs), which glycerol esterifies with three fatty acids, are the main constituents with about 80.3 -88.9% [9]. The TAGs composition of different oils is not alike, so TAG composition is frequently employed to search for authenticity and the geographical and botanical origins [10, 11]. Two types of distribution are available for fatty acids in the TAGs skeleton, including sn-1, 3 (alpha), and sn-2 (beta). Their positional distribution affects these fatty acids' physical and biological properties in TAGs. These features influence the digestion, absorption, and metabolism in human bodies. Also, they may effectively design new fat mixtures for special purposes in children's nutrition [12]. It was found that increasing the saturated fatty palmitic acid level in the sn-2 position of TAG has increased the atherogenic potential in fat rabbits [13].

Compression, aqueous extraction, and refining are three categories of sesame oil production. Solvent extraction sesame oil (crude sesame oil) should be refined including degumming, alkali refining, bleaching, and deodorizing. These are classified as refined sesame oils. Aqueous extraction sesame oil is sold and much more expensive than compression and refining sesame oil for its special flavor and high safety without hexane hazards [14]. It has been revealed that adding refined or pressed sesame oil into aqueous extraction and then labeling it as fully aqueous extraction is an open secret. It is

challenging to distinguish between real aqueous extraction of sesame oil products and fake ones. Since the processing approach and materials used in producing the sesame oils are different, the physicochemical properties, fatty acids distributions, and TAGs composition are expected to be different too. Accordingly, the physicochemical properties, fattv acid distributions, and TAGs composition should be compared to discriminate the quality of products. Different processing steps have effects on chemical composition. However, there is little information about the impacts of different processing methods and steps on the fatty acid distributions and TAGs composition. This study aimed to identify the differences between sesame oils from various extraction methods and explore the differences between sesame oils acquired from each refining step. The differences were elucidated and discussed by determining the fatty acid distributions, TAGs composition, and the levels of sesamin, sesamolin, total tocopherols, acid value (AV), and peroxides value (PV).

Materials and methods

Sesame oil extracted through different methods The sesame seeds were a white species, supplied by the Sesame Research Center, Henan Academy of Agricultural Sciences (Zhengzhou, Henan, China). For compression sesame oil extraction, sesame seeds were chosen, cleaned, roasted at 200°C for 30 minutes, and pressed using a hydraulic press machine (Zhengzhou, Henan, 2-3 minutes China) for according to manufacturer's instructions. For aqueous sesame oil extraction, sesame seeds were selected, cleaned, roasted at 200°C for 20 minutes, and ground using a stone mill at 30 rpm to get sesame slurry before adding 90°C water to the sesame slurry at the ratio of sesame slurry to water at 1:1 (V/V). The admixture was incubated at 50°C with a consistent shaking rate at 80 rpm for 3 hours. The top layer of oil was then separated.

Sesame oil through different refining steps

The crude sesame oil was processed using degumming, alkali refining, bleaching, and deodorizing to obtain the refining sesame oil. The crude sesame oil was obtained by milling sesame seeds and extracting with solvent at the ratio of sesame grinder-to-hexane at 1:3 (V/V) and incubating at room temperature for 6 hours. The solvent was then evaporated in vacuo at 35°C. For degumming sesame oil, the crude sesame oil was warmed to 85°C in a beaker and degummed by adding 1.2% water with gentle excitation for 30 minutes. The oil was settled for 30 minutes. and the water was drained with the dissolved gum. For alkali refining sesame oil, the degumming temperature of sesame oil was increased to 71°C and mixed with a certain amount of sodium hydroxide solution (12%) as per the free fatty acids (FFA) content of the oil with gentle excitation for 15 minutes and then settled for 30 minutes before draining removed the soap stock with residual gum. The neutralized oil was rinsed with 20% hot soft water at 75°C, and residuary soap was taken by settlement. For bleaching sesame oil, alkali refining sesame oil was heated at 90°C under a vacuum of 75 mmHg for 30 minutes to eliminate the trace humidity. The oil was mixed with 3% acid clay (w/w), and the bleaching was done under vacuum at 100-110°C for 10 minutes. The acid clay was removed by filtering after being cooled to 60-70°C. For deodorizing sesame oil, temperature of 240°C and residence time of 100-120 minutes were taken. The steam flow constant rate was 0.25 mL water/min. The system vacuum was kept at 1-3 mm Hg. Finally, deodorized oil was released after cooling to 60-70°C.

Physicochemical properties analysis

The acid value (AV), peroxide value (PV), total tocopherols, sesamin, and sesamolin of sesame oils were analyzed. AV and PV were monitored using the methods described in American Oil Chemist Society (AOCS) (https://www.aocs.org/) Official Method Cd 8b-90 and Cd 18-90, respectively. The tocopherols content was determined by using Waters-e2695 Highperformance liquid chromatography (HPLC) (Waters Inc., Milford, Massachusetts, USA)

following International Union of Pure and Applied Chemistry (IUPAC) (<u>https://iupac.org/</u>) Method 2.432 [15]. Briefly, 0.2 g crushed sesame samples were weighed accurately before adding 10 mL of ethanol and then placed in an ultrasound assisted extraction at 40°C for 20 mins. The reaction was cooled down and supplemented the mass with ethanol solution. After shaking well and standing, the supernatant was sucked out and filtered before placing in the injection bottle for HPLC determination [16, 17]. A total of 0.5 g sesame oil was dissolved in 10 mL of hexane. 5 µL of samples were directly injected into a Nucleosil NH2 chromatograph column (250 mm \times 42.6 mm \times 5 μ m) (Macherey-Nagel, Wiesbaden, Germany) equipped with a RF-10AXL ultraviolet detector (Shimadzu Inc., Kyoto, Japan) at room temperature. The mobile phase was hexane: isopropanol (99:1, v/v) at 1 mL/min flow rate. Tocopherols were measured at 298 nm. The chromatogram's peaks were determined by comparing the retention times of the related tocopherols (α , β , γ , and δ) standards (Sigma, St. Louis, MO, USA). Sesamin and sesamolin were analyzed by using HPLC with a reversed-phase Hypersil BDS Sunfire-C18 chromatography column (250 mm \times 4.6 mm \times 5 μ m) (Thermo Fisher Scientific, Shanghai, China). The mobile phase was an admixture of methanol-deionized water (70/30, v/v) at a 0.8 mL/min flow rate with an injection volume of 10 µL. Absorption was checked at 287 nm. Each sample was replicated two times, and the mean values were the final sesamin or sesamolin content.

Gas chromatography for fatty acids composition analysis

The fatty acids composition of sesame oil was identified using gas chromatography (GC). The samples were converted to methyl esters with boron trifluoride/methanol (1/4, V/V) using the AOCS Ce 2-66 method and analyzed by an Agilent 7890B GC instrument (Agilent Technology, Santa Clara, California, USA) equipped with a HP-88 capillary column (100 m \times 0.25 mm \times 0.1 µm), (Agilent (China) Technology Co., Ltd, Beijing, China) and flame ionization detector. The GC experiments were performed using nitrogen as

the carrier gas with an inlet pressure of 38.4 psi, 1 mL/min, split at 50:1. 1 μ L of sample was injected with the oven temperature at 170°C-no hold, up to 220°C at the rate of 4°C/min-no hold, then up to 235°C at the rate of 1°C/min-no hold. The temperature of the injection port was kept at 250°C, while the temperature of the detector was maintained at 300°C. The fatty acid methyl esters were measured through their relative peak areas recognized in the samples. The total GC running time was 27.5 minutes. The standards of fatty acid methyl ester (mixture of C8-C24, 100 mg) were obtained from Lvyuan Biotech Co., Ltd. (Shanghai, China).

Separation of sn-2 monoacylglycerols (MAGs) from triacylglycerols (TAGs) hydrolyzation

The purified TAGs of sesame oil were segregated by thin-layer chromatography (TLC). The TAGs were isolated, extracted with diethyl ether, and taken as the raw material for composition analysis of sn-2 fatty acid. The TAGs hydrolyzation was done by 1, 3-specificity porcine pancreatic lipase $(3 \times 10^5 \text{ U}, \text{ optimum pH})$ 8.0, optimum temperature 40-45°C) (Lvyuan Biotech Co., Ltd., Shanghai, China) based on Pure and Applied Chemistry Method 2.210 [18]. The sn-2 MAGs were separated from TAGs hydrolyzation by using Florisil solid phase extraction (SPE) column (1,000 mg/6 mL) (Boehner Biotech Co., Ltd., Tianjin, China) with mixed solvents of n-hexane and ether as eluent. Briefly, the TAGs hydrolyzation was injected into the Florisil SPE column followed by injection of 12 mL (85:15, V/V), 16 mL (70:30, V/V), and 12 mL (25:75, V/V) mixed solvent into the SPE column in sequence. The last step elution phase was collected and concentrated to 0.5 mL by an MGS-2200 nitrogen evaporator (Eyela, Japan) to obtain the sn-2 MAGs. The sn-2 MAGs fraction was then converted to methyl esters with boron trifluoride/methanol (1/4, V/V). The fatty acid methyl ester was subject to GC, and the sn-2 fatty acids were determined.

TAGs composition analysis

The International Union of Biochemistry Molecular Biology (IUBMB) (<u>https://iubmb.org/</u>)

recommends using the stereospecific numbering (sn) to describe the stereochemical structure and naming of TAGs. In addition, saturated fatty acids. monounsaturated fatty acids, diunsaturated fatty acids, and triunsaturated fatty acids are represented by S, M, D, and T, respectively. Therefore, according to the degree of unsaturation of the three fatty acids bound to the glycerol skeleton, TAG (XYZ) can be divided types including 1-saturated-2into ten monounsaturated TAG (SM2), 2-saturated-1monounsaturated TAG (S2M), 2-saturated-1diunsaturated TAG (S2D), 3-monounsaturated TAG (M3), 2-monounsaturated-1-diunsaturated TAG (M2D), 1-saturated-1-monounsaturated-1bisunsaturated TAG (SMD), 1-saturated-2disunsaturated TAG (SD2), 3-diunsaturated TAG 1-monounsaturated-1-diunsaturated-1-(D3), triunsaturated TAG (MDT), and 1monounsaturated-2-diunsaturated TAG (MD2). The values of XYZ TAG estimated by the 1, 3random-2-random distribution hypotheses were calculated using the equations below.

%X at 1, 3-positions = (3 × %X at 1, 2, 3 positions - %X at sn-2 position) / 2

%sn - XYZ = 2 × (%X at 1, 3-positions) × (%Y at 2position) × (%Z at 1, 3-positions) × 10^{-4}

Statistical analysis

SPSS 20.0 (IBM, Armonk, New York, USA) was employed for statistical analysis. The amounts of various parameters were represented as the mean ± standard deviations. Analysis of Variance (*ANOVA*) was used to determine the significant differences among the data with *P* value less than 0.05 as the significant difference.

Results and discussion

Physicochemical properties of sesame oils

The physicochemical characteristics of sesame oils obtained from various extraction methods and refining steps were listed in Tables 1 and 2, respectively. AV represented the free fatty acid (FFA) content, implying the oil spoilage level and

	Acid value (mg KOH/g)	Peroxide value (mEq O₂/kg)	Total tocopherols (mg/g)	Sesamin (mg/g)	Sesamolin (mg/g)
Compression	0.26 ± 0.01 ^c	1.61 ± 0.01^{f}	0.42 ± 0.02^{b}	5.83 ± 0.01 ^e	3.43 ± 0.07 ^c
Aqueous extraction	0.15 ± 0.01 ^e	1.40 ± 0.02^{e}	0.40 ± 0.01^{b}	7.12 ± 0.05 ^b	4.08 ± 0.04^{b}
Solvent extraction	1.10 ± 0.07^{a}	3.49 ± 0.11 ^b	0.32 ± 0.32 ^c	3.20 ± 0.04^{h}	1.89 ± 0.02 ^h
Refining	0.10 ± 0.01^{f}	5.26 ± 0.11 ^e	0.15 ± 0.11^{f}	1.84 ± 0.04^{d}	1.09 ± 0.02^{g}

 Table 1. Physicochemical property of sesame oils through different extraction methods.

Note: Different superscripts in the same columns indicated significant differences (P < 0.05).

 Table 2. Physicochemical property of sesame oils through different refining steps.

	Acid value (mg KOH/g)	Peroxide value (mEq O ₂ /kg)	Total tocopherols (mg/g)	Sesamin (mg/g)	Sesamolin (mg/g)
Crude	1.10 ± 0.07 ^a	3.49 ± 0.11 ^b	0.32 ± 0.32 ^c	3.20 ± 0.04^{h}	1.89 ± 0.02^{h}
Degumming	0.82 ± 0.01^{b}	3.52 ± 0.12 ^c	0.30 ± 0.12^{d}	5.06 ± 0.05 ^a	2.60 ± 0.02^{a}
Alkali refining	0.15 ± 0.01^{d}	4.02 ± 0.01^{b}	0.28 ± 0.02^{d}	4.88 ± 0.01^{f}	2.93 ± 0.01 ^e
Bleaching	0.14 ± 0.01 ^{de}	5.14 ± 0.03 ^a	0.21 ± 0.04^{e}	2.26 ± 0.07 ^c	1.28 ± 0.03^{f}
Deodorization	0.10 ± 0.01^{f}	5.26 ± 0.11 ^e	0.15 ± 0.11^{f}	1.84 ± 0.04^{d}	1.09 ± 0.02^{g}

Note: Different superscripts in the same columns indicated significant differences (P < 0.05).

the oil edibility degree. The AV of the sesame oils ranged between 0.15 to 1.10 mg KOH/g, which was a proper domain for edible oil (ISO 660-1996). Compared with compression sesame oil, sesame oil obtained by aqueous extraction and refining had lower AV. Refining sesame oil had the lowest AV due to the neutralization and distillation of FFA in neutralization and deodorization steps, significantly reducing AV from 1.10 to 0.10 mg KOH/g (P < 0.05). PV was the representative of the concentration of peroxides and hydroperoxides created in the initial phases of lipid oxidation. Also, it shows the stability of the oil. The PV was associated with the percentage of hydroperoxides in oil. The high polyunsaturated fatty acid (PUFA) content in sesame oils makes them sensitive to oxidative deterioration. Compared with compression and refining sesame oil, aqueous extraction sesame oil had the lowest PV. The high PV of compression and refining sesame oil might be because of oxidation due to high temperature. The bleaching is the most significant refining phase to decrease peroxides by transformation at oxidation secondary products [19]. However, this study showed that bleaching increased the PV significantly (P < 0.05). Increasing or decreasing

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the PV during this phase pertains to the used clay type and its value [20]. After refining, PV reached a value of 5.26 mEq O₂/kg, which exhibited a significant difference compared with the crude sesame oil (P < 0.05). Tocopherols are important due to their vitamin activities and antioxidant specifications. The results of tocopherol analysis showed that aqueous extraction sesame oil had the highest tocopherol content because there was no high temperature during the process compared with refining sesame oil. The relative contents of tocopherols were reduced constantly during refining, and the loss of total tocopherols due to refining was 53.13%. Tocopherol was considerably decreased during refining steps (P <0.05), and it was mainly decreased in bleaching and deodorization too (Table 2). The reasons could be the absorption by clays within bleaching or distillation under deodorizing in refining. Oil exposure to air or heat might also decrease the tocopherols percentage by oxidation and polymerization [21]. Other groups with minor components of sesame oil were sesamin and sesamolin. The results showed that aqueous extraction sesame oil had the highest percentages of sesamin and sesamolin values as 7.13 and 4.08 mg/g, respectively (Table 1). There

was the disappearance of sesamin and sesamolin during refining processes. The significant chemical changes mainly took place in the bleaching stage using acid clay because the acid clay might have catalyzed the conversion of sesamin and sesamolin [22]. After refining, the content of sesamin and sesamolin reduced 53.62% and 56.08% as 4.88 mg/g and 2.93 mg/g, respectively (Table 2).

Fatty acids composition and distribution of TAGs in sesame oils

The fatty acids distribution of TAGs in sesame oils from different extraction methods and refining steps were shown in Figures 1 and 2. In TAGs, aqueous extraction sesame oil had the highest content of PUFA (47.08%) and lowest content of SFA (14.55%), while refining sesame oil had the lowest content of PUFA (45.89%) and the highest content of SFA (19.59%). Refining decreased the relative values of unsaturated fatty acids (80.41 -85.46%), while it significantly increased the relative values of saturated fatty acids (14.54 -19.59%) (P < 0.05) due to the heating condition [23]. This change occurred mainly during bleaching and deodorization. Most fatty acids of sesame oil were lionleic (C18:2), oleic (C18:1), palmitic (C16:0), and stearic acids (C18:0), whereas several fatty acids like arachidic and linolenic acids, indicated as 'others' were rare ingredients (Figure 1). No significant difference in fatty acids positional distribution was observed between compression and aqueous extraction sesame oil. However, with increasing temperatures in refining, a partial but significant difference was observed in the type of fatty acids positional distribution in the remaining TAGs (P < 0.05) [24]. The amount of linoleic acid was greater than its value in the saturated fatty acids (palmitic and stearic acids), while the amount of oleic acid at the sn-2 position was lower than that before purification. Some researchers stated that the unsaturated fatty acids at the sn-2 position of glycerol TAGs were more stable than the unsaturated fatty acids at the sn-1 or sn-3 positions [25]. The percentages of unsaturated fatty acids in total and sn-2 fatty acids were 85.30 - 85.46% and 93.13 - 97.85%, respectively,

revealing that unsaturated fatty acids were primarily distributed in the sn-2 position. C18:2 was exclusively incorporated into the sn-2 position, while the saturated fatty acids, particularly the palmitic and stearic acids, were mainly distributed in the sn-1, 3 positions [26, 27]. Minor fatty acids (arachidic and linolenic acids) were mostly incorporated into the sn-1 or sn-3 positions. C18:1 was approximately smoothly distributed in sn-1, sn-2, and sn-3 positions per the previous report [28].

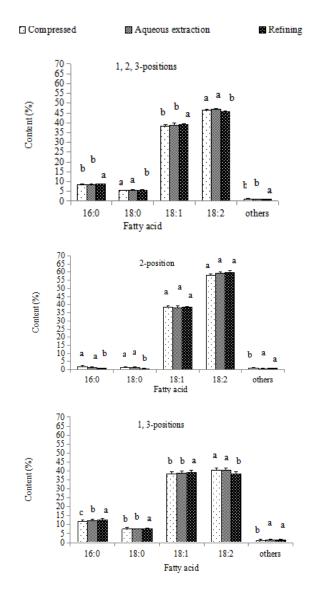


Figure 1. Changes in composition and positional distribution of fatty acids of TAGs in sesame oils obtained from different extraction methods. Each value is the average of three replicates, and vertical bars represent the standard error of the replicate the standard error of the replicate.

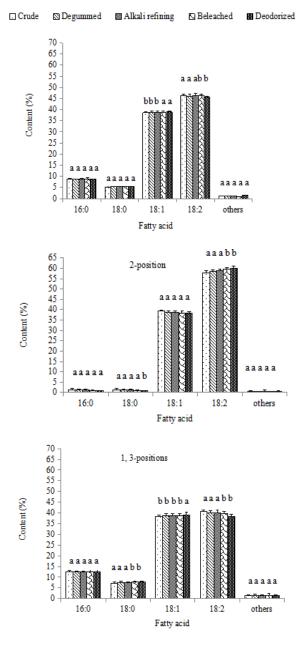


Figure 2. Changes in composition and positional distribution of fatty acids of TAGs in sesame oils obtained from different refining steps. Each value is the average of three replicates, and vertical bars represent the standard error of the replicate the standard error of the replicate.

TAGs composition of sesame oils

The TAGs profiles and the detailed composition in sesame oils obtained from different extraction methods and refining steps were shown in Tables 3 and 4, respectively. The TAGs compositions were calculated through 1,3-random-2-random distribution hypotheses. Sesame oils made through different extraction methods (Table 3) and refining steps (Table 4) comprised 23 kinds of TAGs. The UUU, SUU (UUS), and SUS were the main types of TAGs, OLL (17.49 - 18.49%), OOL (11.74 - 12.66%), LLL (9.25 - 9.92%), OLO (7.96 - 9.02%), LOL (5.95 - 6.95%), OOO (5.52 - 5.80%) were the dominant U of TAGs, which represented almost 62% of the whole TAGs (Figure 3).

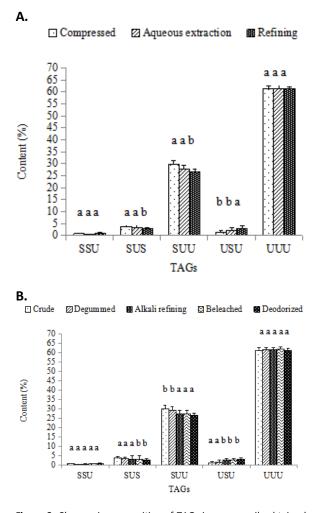


Figure 3. Changes in composition of TAGs in sesame oils obtained from different extraction methods (A) and refining steps (B). S: saturated fatty acids. U: unsaturated fatty acids.

The content of SUU followed UUU, which ranged from 26.38 to 30.16%. The main SUU were PLL, PLO, POL, StLL, POO, and StLO, which were more than 3%. Sesame oil had no or little SSS and SSU types of TAGs. The TAGs compositions showed no significant differences between compression and

TAG	Compression	Aqueous extraction	Solvent extraction	Refining
OLL	18.19 ± 0.17 ^b	18.05 ± 0.10 ^{bc}	17.90 ± 0.22 ^c	17.73 ± 0.00 ^d
OOL	11.94 ± 0.30 ^d	$12.15 \pm 0.00^{\circ}$	$12.24 \pm 0.00^{\circ}$	12.53 ± 0.02 ^b
LLL	9.55 ± 0.00 ^{bc}	9.92 ± 0.00^{a}	9.51 ± 0.01^{bc}	9.44 ± 0.01 ^{bc}
OLO	8.66 ± 0.34 ^b	8.21 ± 0.22 ^d	8.42 ± 0.00 ^c	8.32 ± 0.06 ^c
LOL	6.27 ± 0.00 ^c	6.68 ± 0.00^{b}	6.50 ± 0.72 ^b	6.68 ± 0.11 ^b
000	5.69 ± 0.55 ^b	5.52 ± 0.14^{d}	5.76 ± 0.32 ^b	5.88 ± 0.00 ^e
PLL	5.63 ± 0.00 ^b	5.29 ± 0.00^{bc}	5.78 ± 0.00^{b}	4.84 ± 0.03 ^c
PLO	5.36 ± 0.00 ^a	4.82 ± 0.00^{bc}	5.44 ± 0.00^{b}	4.55 ± 0.00 ^e
POL	3.70 ± 0.01 ^{abc}	3.56 ± 0.00^{cd}	3.95 ± 0.00^{ab}	3.42 ± 0.01^{d}
StLL	3.55 ± 0.00 ^a	3.42 ± 0.00^{bcd}	3.49 ± 0.01^{ab}	3.10 ± 0.00^{d}
POO	3.52 ± 0.00 ^b	3.24 ± 0.06^{b}	3.72 ± 0.00^{ab}	3.21 ± 0.02c
StLO	3.38 ± 0.00 ^a	3.11 ± 0.09^{d}	3.28 ± 0.01^{ab}	2.91 ± 0.04 ^d
StOL	2.33 ± 0.01 ^{de}	2.30 ± 0.00^{b}	2.39 ± 0.01 ^e	2.19 ± 0.00 ^a
StOO	2.22 ± 0.00 ^a	2.09 ± 0.01^{bc}	2.25 ± 0.02^{a}	2.06 ± 0.02 ^d
OPL	0.57 ± 0.04^{ab}	1.04 ± 0.01^{bc}	0.41 ± 0.02^{a}	$1.49 \pm 0.00^{\circ}$
PLSt	1.05 ± 0.02^{ab}	0.91 ± 0.02^{bc}	1.06 ± 0.03^{ab}	0.80 ± 0.05 ^c
PLP	0.83 ± 0.01 ^{de}	0.71 ± 0.04^{b}	$0.88 \pm 0.00^{\rm e}$	0.62 ± 0.06 ^a
POSt	0.69 ± 0.01^{d}	0.61 ± 0.01^{b}	$0.73 \pm 0.00^{\rm e}$	0.56 ± 0.02 ^a
LPL	0.30 ± 0.02^{b}	0.57 ± 0.04 ^c	0.22 ± 0.02^{a}	0.79 ± 0.02 ^c
OStL	0.37 ± 0.01^{d}	0.56 ± 0.01^{b}	$0.30 \pm 0.04^{\rm e}$	0.77 ± 0.00^{a}
POP	0.55 ± 0.00 ^e	0.48 ± 0.01^{b}	$0.60 \pm 0.04^{\rm f}$	0.44 ± 0.04^{a}
OPO	0.27 ± 0.01 ^e	0.47 ±0.02 ^b	$0.19 \pm 0.04^{\rm f}$	0.70 ± 0.01^{d}
LStL	0.19 ± 0.04 ^a	0.31 ± 0.02^{ab}	0.16 ± 0.03 ^a	0.41 ± 0.03^{b}

Table 3. Triacylglycerols composition of sesame oils through different extraction methods (%).

Notes: Different superscripts in the same line indicated significant differences (*P* < 0.05). **O**: oleic acid. **L**: linoleic acid. **P**: palmitic acid. **St**: stearic acid.

aqueous extraction sesame oil, while the TAGs composition of refining sesame oil had a small but significant difference compared with compression and aqueous extraction sesame oil (P < 0.05) because of the high temperature in refining. After refining SUS and SUU decreased 25.84% and 12.53% from 3.87% and 30.16%, while SSU and USU increased 200% and 186.67% from 0.26% and 1.05%, respectively. Bleaching and deodorization were the principal steps in the oil refining which contributed to making the changes in TAGs profiles (Figure 3). If the conditions of bleaching and deodorization was not controlled correctly, the TAGs composition could change significantly. The digestion of TAGs and the absorption of fatty acids would be changed.

Conclusion

This study demonstrated that sesame oil obtained by aqueous extraction had higher sesamin, sesamolin, and tocopherols contents, but lower FFA and peroxides. Aqueous extraction sesame oil had the highest content of PUFA (47.08%) and lowest content of SFA (14.55%), while refining sesame oil had the lowest content of PUFA (45.89%) and the highest content of SFA (19.59%). After different processing steps, the fatty acids' positional distribution's main properties were retained composition of TAGs that showed no significant difference between compression and aqueous extraction sesame oil (P > 0.05), while the TAGs composition of refining sesame oil demonstrated a small but significant difference compared with compression and aqueous extraction sesame oil (P < 0.05) because of the high temperature in refining. After refining, SUS and SUU decreased 25.84% and 12.53% from 3.87% and 30.16%, SSU and USU increased 200% and 186.67% from 0.26% and

TAG	Crude	Degumming	Alkali refining	Bleaching	Deodorization
OLL	17.90 ± 0.22 ^c	17.99 ± 0.10 ^{bc}	17.49 ± 0.32 ^e	18.08 ± 0.12 ^{bc}	17.73 ± 0.00^{d}
OOL	12.24 ± 0.00 ^c	12.11 ± 0.01 ^c	12.66 ± 0.30 ^a	12.42 ± 0.23^{b}	12.53 ± 0.02 ^b
LLL	9.51 ± 0.01 ^{bc}	9.40 ± 0.02 ^{bc}	9.60 ± 0.23 ^{abc}	9.71 ± 0.20^{ab}	9.44 ± 0.01 ^{bc}
OLO	8.42 ± 0.00 ^c	8.60 ± 0.00 ^b	7.96 ± 0.21 ^e	8.42 ± 0.23 ^c	8.32 ± 0.06 ^c
LOL	6.50 ± 0.72 ^b	6.33 ± 0.01 ^c	6.95 ± 0.17 ^a	6.67 ± 0.15 ^b	6.68 ± 0.11^{b}
000	5.76 ± 0.32 ^b	5.79 ± 0.00 ^c	5.76 ± 0.34 ^d	5.78 ± 0.20 ^d	5.88 ± 0.00 ^e
PLL	5.78 ± 0.00 ^b	5.49 ± 0.00 ^{bc}	5.34 ± 0.70 ^{cd}	5.25 ± 0.00 ^e	4.84 ± 0.03 ^c
PLO	5.44 ± 0.00 ^b	5.25 ± 0.02 ^{bc}	4.87 ± 0.43 ^{cd}	4.89 ± 0.01 ^e	4.55 ± 0.00 ^e
POL	3.95 ± 0.00 ^{ab}	3.70 ± 0.04 ^a	3.87 ± 0.01 ^{cd}	3.61 ± 0.21 ^d	3.42 ± 0.01^{d}
S _t LL	3.49 ± 0.01 ^{ab}	3.42 ± 0.01 ^{bc}	3.36 ± 0.00 ^{cd}	3.16 ± 0.30^{d}	3.10 ± 0.00^{d}
POO	3.72 ± 0.00 ^{ab}	3.54 ± 0.07 ^b	3.52 ± 0.43 ^b	3.36 ± 0.45 ^{cd}	3.21 ± 0.02 ^c
S _t LO	3.28 ± 0.01 ^{ab}	3.27 ± 0.00 ^{ab}	3.06 ± 0.60 ^a	2.94 ± 0.09 ^d	2.91 ± 0.04 ^d
StOL	2.39 ± 0.01 ^e	2.30 ± 0.04 ^c	2.43 ± 0.00 ^c	2.17 ± 0.23 ^b	2.19 ± 0.00 ^a
StOO	2.25 ± 0.02 ^a	2.20 ± 0.00 ^{bc}	2.21 ± 0.71 ^{bc}	2.02 ± 0.69 ^{cd}	2.06 ± 0.02 ^d
OPL	0.41 ± 0.02 ^a	0.72 ± 0.15 ^{abc}	0.86 ± 0.01 ^{bc}	1.09 ± 0.33 ^{bc}	1.49 ± 0.00 ^c
PLSt	1.06 ± 0.03 ^{ab}	1.00 ± 0.06^{abc}	0.93 ± 0.41 ^{abc}	0.85 ± 0.52 ^{bc}	0.80 ± 0.05 ^c
PLP	0.88 ± 0.00 ^e	0.80 ± 0.10^{cd}	0.74 ± 0.02^{b}	0.71 ± 0.05^{b}	0.62 ± 0.06 ^a
POSt	0.73 ± 0.00 ^e	0.67 ± 0.07 ^c	0.68 ± 0.33 ^c	0.59 ± 0.00 ^a	0.56 ± 0.02 ^a
LPL	0.22 ± 0.02 ^a	0.37 ± 0.07 ^b	0.47 ± 0.23 ^b	0.58 ± 0.09 ^c	0.79 ± 0.02 ^c
OS _t L	0.30 ± 0.04 ^e	0.43 ± 0.01^{c}	0.45 ± 0.31 ^c	0.77 ± 0.04^{b}	0.77 ± 0.00 ^a
POP	0.60 ± 0.04^{f}	0.54 ± 0.02 ^{de}	0.54 ± 0.61 ^{cd}	0.49 ± 0.02^{a}	0.44 ± 0.04^{a}
OPO	0.19 ± 0.04^{f}	0.34 ± 0.02^{d}	0.39 ± 0.04 ^c	0.51 ± 0.06^{b}	0.70 ± 0.01^{d}
LS _t L	0.16 ± 0.03^{a}	0.22 ± 0.01 ^a	0.24 ± 0.01^{ab}	0.42 ± 0.00^{b}	0.41 ± 0.03^{b}

Table 4. Triacylglycerols composition of sesame oils from different refining steps (%).

Notes: Different superscripts in the same line indicated significant differences (*P* < 0.05). **O:** oleic acid. **L:** linoleic acid. **P:** palmitic acid. **St:** stearic acid.

1.05%, respectively. Bleaching and deodorization were the principal refining steps contributing to the effects. The results concluded that, among the three oil production processes, the nutritional value of water-based sesame oil was the highest, and there was no significant difference in nutritional value between pressed sesame oil and water-based sesame oil. The nutritional value of refined sesame oil was relatively the worst one. In addition, there were significant differences in the triglyceride composition between sesame oil obtained by refining method and sesame oil obtained by pressing method and water substitution method, which could be used as a basis for determining the production process of sesame oil.

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References

- Ghafoorunissa, Hemalatha S, Rao MV. 2004. Sesame lignans enhance antioxidant activity of vitamin E in lipid peroxidation systems. Mol Cell Biochem. 262(1-2):195-202.
- Rogi T, Tomimori N, Ono Y, Kiso Y. 2011. The mechanism underlying the synergetic hypocholesterolemic effect of sesamin and α-tocopherol in rats fed a high-cholesterol diet. J Pharmaol Sci. 115(3):408-416.

- Fernandes L, Casal S, Cruz R, Pereira JA, Ramalhosa E. 2013. Seed oils of ten traditional Portuguese grape varieties with interesting chemical and antioxidant properties. Food Res Int. 50(1):161-166.
- Atabani AE, Silitonga AS, Ong HC, Mahlia TMI, Masjuki HH, Badruddin IA, et al. 2013. Non-edible vegetable oils: a critical evaluation of oil extraction, fatty acid compositions, biodiesel production, characteristics, engine performance and emissions production. Renew Sust Energ Rev. 18(2):211-245.
- Kaur N, Chugh V, Gupta AK. 2014. Essential fatty acids as functional components of food – a review. J Food Sci Technol. 51(10):2289-2303.
- Uzun B, Arslan Ç, Karhan M, Toker C. 2007. Fat and fatty acids of white lupin (*Lupinus albus* L.) in comparison to sesame (*Sesamum indicum* L.). Food Chem. 102(1):45-49.
- Engin Y, Ramazan T, Muharrem G, Bulent U. 2015. Oil content and fatty acid characteristics in mediterranean sesame core collection. Crop Sci. 55(5):2177-2185.
- Fernandez ML, West KL. 2005. Mechanisms by which dietary fatty acids modulate plasma lipids. J Nutr. 135(9):2075-2078.
- Hiremath SC, Patil CG, Patil KB, Nagasampige MH. 2007. Genetic diversity of seed lipid content and fatty acid composition in some species of *Sesamum* L. (*Pedaliaceae*). AJB. 6(5):539-543.
- García-González DL, Luna G, Morales MT, Aparicio R. 2009. Stepwise geographical traceability of virgin olive oils by chemical profiles using artificial neural network models. Eur J Lipid Sci Technol. 111(10):1003-1013.
- Montealegre C, Marina Alegre ML, Garcia-Ruiz C. 2010. Traceability markers to the botanical origin in olive oils. J Agric Food Chem. 58(1):28-38.
- 12. Zhang Y, Li F, Patterson AD, Wang Y, Krausz KW, Neale G, *et al.* 2012. Abcb11 deficiency induces cholestasis coupled to impaired β -fatty acid oxidation in mice. J Bio Chem. 287(29):24784-24794.
- Ramí rez M, Amate L, Gil A. 2001. Absorption and distribution of dietary fatty acids. Early Hum Dev. 65:95-101.
- Kumar SJ, Prasad SR, Banerjee R, Agarwal DK, Kulkarni KS, Ramesh KV. 2017. Green solvents and technologies for oil extraction from oilseeds. Chem Cent J. 11(1):1-7.
- Paquot C. 1982. Standard methods for the analysis of oils, fats and derivatives. 6th Edition. 1st Supplement: Part 4 (1981) Section II, Oils and fats. IUPAC. 54(1):233-245.
- Rangkadilok N, Pholphana N, Mahidol C, Wongyai W, Saengsooksree K, Nookabkaew S, *et al.* 2010. Variation of sesamin, sesamolin and tocopherols in sesame (*Sesamum indicum* L.) seeds and oil products in Thailand. Food Chem. 122(3):724-730.
- Wu WH. 2007. The contents of lignans in commercial sesame oils of Taiwan and their changes during heating. J Agric Food Chem. 104(1):341-344.
- Yoshida H, Tanaka M, Tomiyama Y, Mizushina Y. 2007. Antioxidant distributions and triacylglycerol molecular species of sesame seeds (*Sesamum indicum*). J Am Oil Chem Soc. 84(2):165-172.
- Liu YL: Oil Refining: Decolorization and Deodorization. Oil Production and Processing Technology. Volume 2. 2nd edition.

Edited by Liu YL. Beijing: The Science Publishing Company; 2009:360-406.

- Kim I, Choe E. 2005. Effects of bleaching on the properties of roasted sesame oil. J Food Sci. 70(1):48-52.
- Pimpa B, Kanjanasopa D, Boonlam S. 2009. Effect of addition of antioxidants on the oxidative stability of refined bleached and deodorized palm olein. Int J Agric Nat Reso. 43(2):370-377.
- Huang JN, Song GH, Zhang LX, Sun Q, Lu X. 2012. A novel conversion of sesamolin to sesaminol by acidic cation exchange resin. Euro J Lipid Sci Technol. 114(7):842-848.
- Ortega-García J, Gámez-Meza N, Noriega-Rodriguez JA, Dennis-Quiňonez O, arcía-Galindo HS, Angulo-Guerrero JO, *et al.* 2006. Refining of high oleic safflower oil: Effect on the sterols and tocopherols content. Eur Food Res Technol. 23(6):775-779.
- Yoshida H, Abe S, Hirakawa Y, Takagi S. 2001. Roasting effects on fatty acid distributions of triacylglycerols and phospholipids in sesame (*Sesamum indicum*) seeds. J Sci Food Agri. 80(2):620-626.
- Yoshinaga K, Sasaki K, Watanabe H, Nagao K, Inoue N, Shirouchi B, et al. 2015. Differential effects of triacylglycerol positional isomers containing n-3 series highly unsaturated fatty acids on lipid metabolism in C57BL/6J mice. J Nutr Biochem. 26(1):57-63.
- Karupaiah T, Sundram K. 2007. Effects of stereospecific positioning of fatty acids in triacylglycerol structures in native and randomized fats: a review of their nutritional implications. Nutr Metab (Lond). 4(16):103-120.
- 27. Gao F, Birch J. 2016. Oxidative stability, thermal decomposition, and oxidationonset prediction of arrot, flax, hemp, and canola seed oils in relation to oil composition and positional distribution of fatty acids. Eur J Lipid Sci Technol. 118(7):1042–1052.
- Momchilova S, Nikolova-Damyanova B. 2022. Regio-and stereospecific analysis of triacylglycerols—a brief overview of the challenges and the achievements. Symmetry-Basel. 14(2):247.