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Cold-pressed tiger nut (*Cyperus esculentus* L.) oils: chemical and aromatic profiles, sensory properties, and consumer preferences

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SUMMARY: In this study, tiger nut oils produced by cold pressing were characterized by means of physicochemical, compositional, and sensory analyses. The major fatty acids were oleic (70.4%), palmitic (13.3%), and linoleic (11.9%) acids. The main sterols were β-sitosterol and stigmasterol (58.3 and 20.5 mg/100 g), and the main tocopherol was α-tocopherol (234.78 µg/g). Syringic acid, apigenin and vanillin were the major phenolic compounds quantified. The cold-pressed oils crystallized at -9.12 °C and melted at -1.87 °C. A sensory panel described the oil with 5 sensory descriptive (almond, nutty, roasted, straw, sweety, soil) terms. A consumer test indicated that appearance, smell/aroma, and taste/flavor scores were above 4.0 on a 5-point hedonic scale. In conclusion, tiger nut oils with retained nutrients and specific aroma could be produced by the cold-pressing technique. Further studies for food and functional food applications of this gourmet oil are anticipated.

KEYWORDS: *Aroma; Cold-pressed oil; Composition; Consumer; Sensory profiles; Tiger nuts.*

RESUMEN: ***Aceites de chufa (*Cyperus esculentus *L.) prensados en frío: perfiles químicos y aromáticos, propiedades sensoriales y preferencias del consumidor.*** En este estudio, los aceites de chufa producidos mediante prensado en frío se caracterizaron mediante análisis fisicoquímicos, composicional y sensorial. Los principales ácidos grasos fueron el oleico (70,4%), palmítico (13,3%) y linoleico (11,9%). Los principales esteroles fueron β-sitosterol y estigmasterol (58,3 y 20,5 mg/100 g), y el principal tocoferol fue α-tocoferol (234,78 µg/g). El ácido siríngico, la apigenina y la vainillina fueron los principales compuestos fenólicos cuantificados. Los aceites prensados en frío cristalizaron a -9,12 °C y se fundieron a -1,87 °C. El panel describió el aceite con cinco términos descriptivos sensoriales (almendra, nuez, tostado, paja, dulzor, tierra). Las pruebas de consumo indicaron que la apariencia, el olor/aroma y el sabor/sabor obtuvieron puntuaciones superiores a 4,0 puntos en una escala hedónica de 5 puntos. En conclusión, mediante la técnica de prensado en frío se podrían producir aceites de chufa con nutrientes y un aroma específico. Se anticipan más estudios sobre aplicaciones alimentarias y funcionales de este aceite gourmet.

PALABRAS CLAVE: *Aceite prensado en frío; Aroma; Chufas; Composición; Consumidor; Perfiles sensoriales.*

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1. INTRODUCTION

The tiger nut (*Cyperus esculentus* L.) is a tuberous edible plant belonging to the *Cyperaceae* family, and is also known as "Chufa" and "Zulu". The tiger nut grows all over the world, and is widely utilized due to its high yield. The main products derived from the tubers are tiger nut milk, flour and oil (Maduka and Ire, 2018; Asare *et al*., 2020; Yu *et al*., 2022).

Tiger nuts contain approximately 26-30% starch, 21-25% fat, 8-10% dietary fiber, 3-8% protein, and phosphorus (P), potassium (K), iron (Fe), calcium Ca), and vitamins E and C. The main fatty acids found in tiger nut oil are myristic (0.6-0.8%), palmitic (11-12%), palmitoleic (0.2-0.3%), stearic (2-4%), oleic (68-75%), linoleic (8-12%), α-linolenic (0.1-0.3%), arachidonic (0.3-1.0%), and eicosenoic (0.2-0.3%), respectively (Warra *et al*., 2017; Vega-Morales *et al*., 2019; Asare *et al*., 2020; Yu *et al*., 2022). Zhang *et al*. (2022) reported the presence of α-, β-, γ-, and δ-tocopherols and tocotrienols with the dominancy of δ-tocopherols (150.30-164.42 mg/kg). Furthermore, sitosterol as the main sterol (112.43-137.02 mg/100 g oil) with the presence of campesterol and stigmasterol were reported.

Various methods, including solvent and supercritical carbon dioxide extraction have been used to obtain tiger nut oils, but studies on the cold-press technique are limited (Warra *et al*., 2017; Roselló-Soto *et al*., 2019). Miao *et al*. (2022) used a high-pressure hydraulic press (40-60 MPa) to obtain the tuber oils. Although they called this type of oil cold-pressed oil, the temperature of the oil extraction process was not reported.

The aims of this study were to produce cold-pressed oil from the tiger nuts and to fully characterize the oil to possibly extend its functional food applications. The novelty of this study is based on data (thermal properties, aromatics volatiles composition, sensory descriptive analysis, and consumer tests) that provided new contributions to the existing literature.

2. MATERIALS AND METHODS

2.1. Materials

Tiger nuts (*Cyperus esculentus* L. var. Honey Tuber) were obtained from FiFarm Tarım Gıda Danışmanlık Co. (Adana, Turkey) from the 2020 harvest and processing season. The registered Bal Yumru (Honey Tuber) variety was cultivated in the Eastern Mediterranean Agricultural Research Institute (Adana, Turkey). After the necessary cleaning process, 2 lots of approximately 5 kg tiger nuts were stored in cloth bags until pressing. All other chemicals and standards used were of analytical grade, and were purchased from Merck (Darmstadt, Germany) and Sigma-Aldrich Chem. Co. (St. Louis, USA).

2.2. Biometric characteristics of the tiger nuts

The dimensions of ten randomly selected tiger nuts were measured with a digital caliper and the results were averaged in millimeters. A Minolta colorimeter (CR-400, Osaka, Japan) calibrated with a white tile was used for color measurements, and the L, a*, b* values of the samples were recorded. For the determination of 1000-tiger nut weight, 10 randomly selected nuts were weighed and the number was multiplied by 100 for several times. The moisture content of the ground nuts (10 g), was measured with the Ohaus MB45 (Ohaus, Pine Brook, USA) rapid moisture analyzer at 105 °C. The amount of crude oil in the nuts was determined according to the AOAC 920.39 method (AOAC, 2002) using a soxhlet extractor, and the amount of ash was determined according to the AOCS Ba 5a-49 method (AOCS, 1998) using a burning furnace (Protherm Furnaces, PLF 110/15, Turkey).

2.3. Cold-pressing of the tiger nuts

A laboratory scale (12 kg seed/hour capacity, single head, 2 hp, 1.5 kW power) cold-press machine (ESM 3710 model, Koçmaksan Co., İzmir, Turkey) was used for the cold-pressing of the tiger nuts. For optimum oil production, 20 rpm screw rotation speed, no. 12 outlet tip and 40 °C oil outlet temperature were used as constant operating parameters. After collecting the oil, the suspended particles and moisture were removed by filtering and centrifugation (Sigma 2-16K, Sartorius, Germany) at 6797 xg at 20 °C for 10 minutes. The oils were put in brown glass bottles, flashed with nitrogen gas and capped. They were stored in a refrigerator until analysis.

2.4. Physicochemical properties of tiger nut oil

The specific gravity and specific absorbance of the oils were determined by AOCS Cc 10c-95 and

Ch 5-91 methods (AOCS, 1998) at 25 °C, respectively. The refractive index with an Abbe 5 (Bellingham and Stanley, UK) refractometer, and the apparent viscosity with a Brookfield Viscometer (model DV II+Pro with Reocalc software, Brookfield Eng. Lab., Inc., MA, USA) equipped with an LV-SC4-18 spindle rotating 30 rpm were measured at 25 °C. The color values of L, a* and b* were measured with a Minolta Colorimeter CR-400 (Minolta Camera Co., Osaka, Japan). The free fatty acidity and acid values of the samples were measured using Ca 5a-40 and Cd 3d-63 methods, and similarly, peroxide number, *p*-anisidine value and iodine number were measured using Cd 8-53, Cd 18-90 and Cd 1-25 methods, respectively (AOCS, 1998). Saponification numbers and unsaponifiables were determined using methods Cd 3-25 (AOCS, 1998) and ISO 3596 (ISO, 1991), respectively. The total carotenoid content in the oils was determined spectrophotometrically according to the method of Franke *et al.* (2010). The chlorophyll content measurements were made at three different wavelengths (630, 670 and 710 nm) according to the spectrophotometric method based on the AOCS 13i-96 method (AOCS, 1998).

2.5. Composition of tiger nut oil

The fatty acid methyl esters were prepared according to AOCS method Ce 2-66 (AOCS, 1998). Fatty acid compositions were determined using a Gas Chromatograph-FID (Agilent Technologies 7890B, Palo Alto, CA, US) with a HP 88 capillary column (100 m × 0.25 mm ID × 0.2 μm film thickness, J&W Scientific Co., CA, USA). The working conditions of Gas Chromatography (GC) were as follows: 1 μL injection volume, 1:50 injector split ratio, 2 mL/min flow rate, hydrogen as carrier gas, hydrogen (40 mL/min) and dry air (450 mL/min) as detector gases, 280 °C inlet temperature and 280 °C detector temperature. The analyses condition was 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. Fatty acids were quantified using a FAME standard mixture (37-components, 4-24, Supelco, Bellefonte, PA, USA) through comparison of the retention times and peak areas.

The sterol composition of the tiger nut oils was detected according to the ISO 12228 method (TSE, 1999). Unsaponifiable matters were attained, and then sterol fractions were distinguished using Thin Layer Chromatography (TLC). After separating the sterol fraction on TLC, the analysis was managed with the same GC and DB5 capillary column (30 m × 0.25mm ID × 0.1 μm film thickness, J &W Scientific Co, CA, USA). The working conditions of GC were as follows: 1/100 injector split ratio, 1 μL injection volume, 0.8 mL/min flow rate, hydrogen as carrier gas, hydrogen (30 mL/min) and dry air (400 mL/min) as detector gases, 290 °C inlet temperature, and 300 °C detector temperature. The analysis conditions were as follows: oven temperature, 60 °C for 2 min, 60-220 °C (40 °C/min) for 1 min, and 220-310 °C (5 °C/min) for 30 min. The relative retention times of commercially available standards (cholesterol, brassicasterol, stigmasterol, and β-sitosterol) were used under the same conditions to identify and quantify the sterols in the samples.

The tocopherol contents in the tiger nut oils were determined according to Grilo *et al*. (2014). First, a sample (200 μL) was diluted to 5 mL with dichloromethane. The mixture was vortexed for 30 min and then transferred into a vial for injection. Reverse-phase HPLC (Shimadzu Corporation, Kyoto, Japan), equipped with an LC-20AT HPLC pump, SIL-20AHT degasser, CTQ-10ASVP column oven, RF-20A diode array detector, and auto sampler (SIL-20AHT) into the Inertsil ODS-3 column (250mm x 4.6mm x 5μm, GL Sciences Inc., Japan) were used for the determination of the tocopherol compositions of the oils. A methanol:water (97:3, v/v) mixture was used as the mobile phase. Isocratic elution with a flow rate of 1.6 mL/min was used. The detector wavelengths were adjusted to 290 and 330 nm for excitation and emission, respectively. Quantification was applied using commercial tocopherol standards (Merck, Darmstadt, Germany).

The phenolic compounds of the tiger nut oils were extracted according to the technique of Vallverdú-Queralt *et al*. (2014.) through a SPE cartridge (CNWBOND Poly-Sery, GmbH, Germany). Then, the method of Moulehi *et al*. (2012) was used with minor modifications to analyze the phenolic compounds in the oil samples. Reverse-phase HPLC (Shimadzu Corporation), equipped with a Zorbax Eclipse Plus C18 column (250 mm × 4.6 mm × 5 μm), CTQ-10ASVP column oven, LC-20AT HPLC pump, SIL-20AHT degasser and a SPD-M20A diode array detector (280 nm) were used for the determination of the phenolic compositions of the oils. Sulfuric

acid: water (2:998, v:v) (A) and acetonitrile (B) were used as the mobile phase and the flow gradients used in this analysis were as follows: 0. min 0% A, 0.1–18 min 80% A, 18–24 min 70% A, 24–30 min 67.5% A, 30–36 min 45% A, 36–40 min 0% A, 40–45 min 60% A, and 45–47 min 80% A. Commercial standards with minimum 95–98% purity (Sigma-Aldrich and Fluka Chem Co., St. Louis, MO, USA) were used under the same condition to identify and quantify the phenolic compounds in the samples.

2.6. Thermal properties of tiger nut oil

Differential scanning calorimetry (DSC, Perkin-Elmer DSC 4000 series, USA) was used to determine the thermal parameters of the oils. 5-7 mg of oil sample were weighed into aluminum cups, hermetically sealed, and placed in the sample compartment of the device. The method used in the analysis was as follows: heating from 20 ºC to 110 ºC at 10 ºC/min rate, then cooling to -70 ºC at the rate of 10 ºC/min, and maintaining this temperature for 3 minutes for complete crystallization. Finally, it was reheated to 50 ºC at the rate of 5 ºC/min. From the obtained thermograms, melting temperatures (Tm), melting enthalpies (ΔHm), crystallization temperatures (Tc) and crystallization enthalpies (ΔHc) were calculated by the the Pyris1 Manager software program of the device. In addition, DSC was used to determine the oxidative induction times (OIT). OIT is the time required for an oil sample to start oxidation under oxygen atmosphere at a certain temperature. 5-10 mg of oil sample were weighed into aluminum cups and put into the sample compartment of the device with its pan open. The pan was heated from 30 °C to 150 °C at 20 °C/min under 99.99% pure nitrogen. Then, it was held at 150 °C for 30 minutes under 99.99% pure oxygen. Pyris1 Manager Software was used for the calculations.

2.7. Volatile aromatic compounds in tiger nut oil

The volatile aromatic compounds were determined following the Krist *et al*. (2006) method with minor modifications. Five milliliters of oil sample were set in an amber-colored vial, and then 1 g of NaCl and 20 µL of internal standard (1 µL of 2-methyl-3-heptanone dissolved in 10 mL methanol) were added. The vial was kept in a water bath at 60 °C for 15 min. In order to collect the volatile components in the headspace and take them up using SPME (2 cm, 50/30 µm DVB/Carboxen/PDMS, Supelco, Bellafonte), a fiber was inserted and kept for 15 min in a water bath at 60 °C. At the end of this period, the headspace fiber was taken off and inserted into the injection port of the gas chromatography. A gas chromatograph (Agilent 6890HB, Agilent Technologies, Wilmington, DE, ABD), equipped with an MS detector (Agilent 5875C, Agilent Technologies, Wilmington, DE, ABD) and HP5 MS column (30 m x 0,25 mm i.d. x 0,25-µm film thickness; J&W Scientific, Folsom, CA, ABD) was used to measured volatile compounds. The analyss conditions of the GC were as follows: 1:2 split ratio, 1.2 mL/min flow rate, with helium as carrier gas. The working conditions of the MS detector were as follows: 280 °C capillary direct interface temperature, 70 eV ionization energy, 35–350 amu mass range, and 4.45 scans/s scanning rate. The analysis conditions were as follows: oven temperature set at 40 °C for 1 min, heating to 200 °C (4 °C/min), heating to 230 °C (7 °C/min), and holding at that temperature for 15 min. For identification of the volatile compounds, the mass spectra of unknown substances were compared with the libraries of Wiley, Nist, Tutor, and FFNSC. The identified and quantified volatiles are presented with retention times and % area values.

2.8. Sensory properties of tiger nut oil

A descriptive sensory analysis was carried out according to the flavor panel evaluation method for vegetable oils, Quantitative Descriptive Analysis (QDA) with Cg 2–83 (AOCS, 1998; Meilgaard *et. al*., 1991). Five female and four male panelists aged 20-51 were voluntarily trained for more than 15 hours in separate sessions on consecutive days. Moderated by the panel leader, the panelists identified sensory descriptive terms for different samples. There were six descriptive terms selected to describe the oils, and the standards used to calibrate the panelists are shown in Table 1. Linear scales anchored from 0 at the left end for minimum intensity to 10 at the right end for maximum intensity were used. In each session, the oil samples were coded with three-digit numbers and placed in a clear glass with a round base and a thin cap closed with a lid. The oil samples were presented to the panelists at room temperature under sunlight. A signed consent form was prepared indicating that the oil sample was safe, but

Table 1. Descriptors and references used for the sensory Quantitative Descriptive Analysis (QDA) of cold-press produced tiger nut oils.

Attribute	Description	Standards
Almond	Flavor and aroma of fresh almonds	Fresh almond, Benzaldehit
Nutty	Flavor of roasted hazelnut and pistachio	Roasted hazelnut
Roasted	Flavor of roasted snacks	Roasted chickpea
Straw	Aromatics associated with dry straw	Dry straw
Sweet	Sweet smell perceived from honey	Flower honey
Soil	Smell and odors associated with wet soil	Wet soil

panelists were suggested to not swallow it during the test. Drinking water, unsalted crackers and expectoration cups were also provided during the tests.

2.9. Consumer test of tiger nut oil

The sensory attributes (appearance, smell/aroma, taste/flavor, general acceptance) of the oils were assessed on a 5-point hedonic scale (1 = disliked extremely to 5 = liked extremely). The tiger nut oil sample set was tested by a total of 100 different voluntary consumers, male and female, between the ages of 20 and 60. The same consent form and aids were provided during the tests.

2.10. Statistical analyses

Cold pressing of the tiger nuts was replicated twice. For each replicate sample, the analyses were performed twice for composition analyses and thrice for other analyses. The collected data were provided as mean values with standard errors, and as means with standard deviation for the sensory data.

3. RESULTS and DISCUSSION

3.1. Biometric characteristics of tiger nuts

Some properties of the tiger nuts are presented in Table 2. The L, a* and b* values for the nuts were 47.96, 11.37 and 22.31. The length, thickness, width, 1000-nut weight values for the tiger nuts were established as 16.98, 10.10, 8.33 mm, and 619.20 g, respectively. Codina-Torrella *et al*. (2015) reported similar results for color and dimension parameters in their studies. Except for the 1000-nut weight value, the measured properties were in good agreement with a previous study which used the same Honey Tuber variety (Duman, 2019). The proximate composition was also measured (Table 2). Compared to the literature, there were small differences, possibly due to variety and cultivation differences. Previously, a tiger nut moisture content of 8.5%, oil of 30.01%, and ash of 2.23% were reported (Ahmed *et al.,* 2019). In another study, 6.38-8.66% moisture, 25.35-35.21% total oil, 1.60-1.97% ash were determined (Codina-Torrella *et al*., 2015). In this study, the amounts of moisture, ash, and oil in tiger nuts were detected as 8.21, 28.42 and 1.59%, respectively, as shown in Table 2. The results were considered to be similar to previous studies, even with minor differences due to variety and cultivation conditions.

Table 2. Biometric characteristics of the tiger nuts used.

	Tiger Nut (Mean ±SE, n = 6)
Dimension (mm)	
Lenght	16.98 ± 0.01
Thickness	10.10 ± 0.74
Width	8.33 ± 0.33
1000-nut weight (g)	619.20 ± 1.91
Color	
L	47.96 ± 0.51
a*	11.37 ± 0.10
b*	22.31 ± 0.55
Moisture (%)	8.21 ± 0.42
Oil (%)	28.42 ± 1.08
Ash (%)	1.59 ± 0.24

3.2. Physicochemical properties of tiger nut oil

The physicochemical properties of the oil samples are presented in Table 3. A lower specific gravity (0.893 g/ml at 20 °C) and similar refractive index (1.4680) were reported for the same Honey Tuber variety (Duman, 2019). Literature (Yoon, 2016; Duman, 2019; Miao *et al*., 2022) reported color values of around 1.5-4.0 Red, 24.4-72.9 Yellow and 0.1-2.9 Blue for this oil, and we measured 28.19, 2.17, 12.70 of L, a* and b* values. Clearly, this oil is of a yel-

Table 3. Some physicochemical properties of the cold-press produced tiger nut oils.

	Tiger Nut Oil (Mean ± SE, n = 6)
Specific gravity (g/ml, 25 °C)	1.04 ± 0.03
Refractive index (25 °C)	1.47 ± 0.00
Specific extinction K_{232}	1.60 ± 0.21
Specific extinction K_{270}	0.41 ± 0.07
Viscosity (cP, 25 °C)	89.00 ± 0.00
Color L	28.19 ± 1.21
Color a*	2.71 ± 0.21
Color b*	12.70 ± 0.16
Free fatty acidity (% linoleic acid)	1.82 ± 0.01
Acid value (mg KOH/g oil)	3.63 ± 0.02
Peroxide value (meq O_2/kg oil)	2.64 ± 1.14
p-anisidine value	12.66 ± 1.33
Iodine number (g I/100 g oil)	82.20 ± 0.24
Saponification number (mg KOH/g oil)	160.01 ± 1.76
Unsaponifiable matter (%)	0.51 ± 0.05
Total carotenoid (mg/100g oil)	0.60 ± 0.12
Total chlorophyll (mg pheophytin a/kg oil)	0.08 ± 0.03

lowtone. The *p*-anisidine value and specific absorbance at K_{232} and K_{270} of the cold-pressed tiger nut oils were measured as 12.66, 1.60 and 0.41, respectively. In one study (Zhang *et al*., 2022), K_{232} values of 1.17-1.22 and K_{286} values of 0.12-0.22 were reported. The free fatty acidity (FFA) and acid value of the oil was 1.82% linoleic acid and 2.69 mg KOH/g, and very similar data were presented in the available literature (Yoon, 2016; Duman, 2019; Miao *et al*., 2022). The peroxide number (2.64 meq O_2/kg oil) was different from one (20.43 meq O_2/kg oil) reported by Duman (2019), but was quite similar to those reported by others (Yoon, 2016; Miao *et al*., 2022). The measured iodine and saponification numbers as well as the unsaponifiable matter contents were in good agreement with the existing literature cited above. Total carotenoid content of 0.60 mg/100g oil, and total chlorophyll content of 0.08 mg pheophytin a/kg oil were measured as new data for the literature. In another study, the total carotenoid and total chlorophyll contents in cold-pressed grapefruit seed oils were reported as 2.35 mg/100g oil and 0.08 mg pheophytin a/kg oil (Aydeniz Güneşer, 2016). Generally, the physicochemical properties of the cold-pressed tiger nut oils were found to be in good agreement with similar oils in the available literature.

3.3. Composition of tiger nut oil

The fatty acid, tocopherol, sterol, and phenolic compositions of the tiger nut oils were analyzed and the results are presented in Table 4. Eight different fatty acids (FA) were quantified, and the major fatty acids were oleic (70.4%), palmitic (13.3%), and linoleic (11.9%) acids. The nut variety was reported to cause FA composition differences (Duman, 2019). General-

Table 4. Composition of the fatty acid, sterol, tocopherol and phenolic compounds in the cold-press produced tiger nut oils.

	Tiger Nut Oil (Mean ± SE, n = 4)
Fatty acids (%)	
Myristic acid (C14:0)	0.7 ± 0.05
Palmitic acid (C16:0)	13.3 ± 1.2
Palmitoleic acid (C16:1)	0.4 ± 0.1
Stearic acid (C18:0)	3.1 ± 0.5
Oleic acid (C18:1)	70.4 ± 2.8
Linoleic acid (C18:2)	11.9 ± 1.3
Linolenic acid (C18:3)	0.1 ± 0.1
Arachidic acid (C20:0)	0.1 ± 0.01
Tocopherols (μg/g oil)	
α-Tocopherols	234.78 ± 5.50
β- Tocopherols	75.13 ± 2.30
γ-Tocopherols	4.61 ± 0.32
δ-Tocopherols	1.18 ± 0.35
Sterols (mg/100 g)	
β-sitosterol	58.3 ± 2.5
Brassicasterol	0.5 ± 0.01
Delta-5-avenasterol	3.7 ± 0.5
Campesterol	14.7 ± 2.6
Cholesterol	1.2 ± 0.9
Stigmasterol	20.5 ± 3.2
Phenolic Compounds (μg/g)	
Syringic acid	5.27 ± 0.70
Vanillin	3.68 ± 0.50
p-Coumaric acid	0.31 ± 0.05
Ferulic acid	0.43 ± 0.00
Benzoic acid	1.89 ± 0.20
o-Coumaric acid	0.07 ± 0.05
Rosmarinic acid	1.37 ± 0.01
Cinnamic acid	0.38 ± 0.00
Apigenin	4.95 ± 0.50

ly, the FA composition data concurred with the literature (Ahmed *et al*., 2019; Duman, 2019; Yoon, 2016). The oil is characterized by high oleate content, and this type of oils was found suitable for frying due to its thermal stability (Ahmed *et al*., 2019).

Four tocopherols (µg/g oil) were quantified as α-tocopherol (234.78), β-tocopherol (75.13), γ-tocopherol (4.61) and δ-tocopherol (1.18), (Table 4). The total tocopherol accounts for around 315.7 µg/g in this oil. Yoon, (2016) reported the tocopherol contents as α-tocopherol (37.5-42.4%), γ-tocopherol (25.2-31.00%), δ-tocopherol (29.01-34.3%) in different refining steps of tiger nut oils. In another study (Duman, 2019), α-tocopherol (15.89 mg/100g) and β-tocopherol (9.44 mg/100g) were found in the same variety of tiger nut. In general, the findings of this study concur with the literature.

The sterols β-sitosterol (58.3%), brassicasterol (0.5%), delta-5-avenasterol (3.7%), campesterol (14.7%), cholesterol (1.2%) and stigmasterol (20.5%) were quantified in the oil samples (Table 4). In addition to these, three different streols, delta-7-avenastrol, delta-7-stigmasterol and eritrodiol-uvaol were found in a previous study (Duman, 2019). It has been observed that β-sitosterol and stigmasterol were the dominant sterols, among others. In another study, β-sitosterol (517.25 µg/g) and stigmasterol (225.25 µg/g) was reported (Yeboah *et al*., 2012).

Nine different phenolic compounds were quantified (µg/g), and the phenolics were syringic acid (5.27), vanilin (3.68), *p*-coumaric acid (0.31), ferulic acid (0.43), benzoic acid (1.89), *o*-coumaric acid (0.07), rosmarinic acid (1.37), cinnamic acid (0.38) and apigenin (4.95), (Table 4). It could be observed that syringic acid and apigenin occurred in higher concentrations, and *p*-coumaric acid was in the lowest proportion. It was reported (Oladele *et al*., 2017) that the phenolic composition of tiger nut oils changes with plant variety. The ferulic and *p*-hydroxybenzoic acids (58.38 and 29.12 mg/100 g) were the main phenolic compounds in yellow tiger nuts, and vanillic and *p*-coumaric acids (15.20 and 17.25 mg/100 g) were the predominant compounds in the brown variety.

3.4. Thermal properties of tiger nut oil

The thermal properties of the tiger nut oil samples are presented in Table 5. The oil started to crystallize at around -7.19 °C and crystallization was completed at around -9.12 °C. The enthalpy of crystallization was measured as -7.11 J/g, while the enthalpy of melting was 72.05 J/g. In addition, the exac crystallized oil samples (held for 3 min at –70 °C), started melting at around -11.04 °C, and melting was completed at around -1.87 °C. In this study, the thermal properties were provided in the literature for the first time. The oxidation induction time (OIT) value is an indicator of oil stability against oxidation. Lower OIT values indicate that the oil is more susceptible to oxidation or has a shorter shelf life. This oil had around 6.3 min OIT (Table 5) value. The contents of natural antioxidants and ratio of unsaturated fatty acids govern the OIT value. (Aydeniz Güneşer, 2016).

Table 5. Thermal properties of the cold-press produced tiger nut oils.

	Tiger Nut Oil (Mean ± SE, n = 4)
Crystallization	
$Onset_c$ (°C)	-7.19 ± 0.33
T_c (°C)	-9.12 ± 0.32
ΔH_c (J/g)	-7.11 ± 0.51
Melting	
$Onset_m$ (°C)	-11.04 ± 0.09
T_m (°C)	-1.87 ± 0.21
ΔH_m(J/g)	72.05 ± 0.69
OIT (min)	6.3 ± 0.50

$Onset_c$: crystallization start temperature; T_c: crystallization peak temperature; ΔH_c: crystallization enthalpy; $Onset_m$: melting start temperature; T_m: melting peak temperature; ΔH_m: melting enthalpy

3.5. Volatile aromatic compounds in tiger nut oil

Forty different aromatic volatile compounds were identified in the tiger nut oil samples and are listed in Table 6. The volatile compounds with higher amounts were furfural (24.27%), 2,5-dimethyl pyrazine (15.86%), acetic acid (13.66%), 5-methyl-2-furfural (6.24%), methyl pyrazine (3.78%), 2-acetylfuran (3.52%), 2-acetyl pyrrole (3.19%), nonanal (3.45%), and 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one (3.28%), respectively. These compounds were usually described with almond, sweet, caramel, roasted, nutty, fruity, walnut, bread and chocolate definitions. It was observed that the results were similar to the sensory analysis results, with the panelists reporting that they felt these components intensely with higher scores for nutty, almond and roasted descriptors (Table 7). In aroma science, the concentration of an individual compound does not

Table 6. Composition of the volatile aromatics in the cold-press produced tiger nut oils.

No	RT† (min)	Volatile Compound	Aroma Definition††	Peak Area (Mean ± SD, n = 4)	Peak Value (%)
1	1.299	Acetaldehyde	Fresh, fruity	154162.0 ± 12541.2	0.46
2	1.370	Ethyl alcohol	Alcoholic, medicinal	73035.5 ± 2429.8	0.22
3	1.873	Acetic acid	Vinegar, sour	4616750.0 ± 22538.9	13.66
4	2.253	3-Methylbutanal	Sour, fruity, chocolate	61053.5 ± 1782.6	0.19
5	2.305	Acetol	Sweet, caramel	574337.5 ± 527.7	1.70
6	2.650	Propionic acid	Acidic, vinegar	32406.5 ± 4609.9	0.10
7	2.688	2,3-Pentanedione	Butter, sweet, nut, caramel	152652.5 ± 3959.7	0.46
8	3.287	Pyrazine	Roasted, nutty	29353.0 ± 1185.8	0.09
9	3.962	Acetol	Sweet, caramel, roasted	246117.0 ± 25169.1	0.73
10	4.351	Butanoic acid	Butter, fruity, rancid	34621.5 ± 1922.6	0.11
11	4.655	Hexanal	Vegatable, fresh, herbal	275055.0 ± 15640.4	0.82
12	5.264	Methyl pyrazine	Chocolate, nut, peanut	1276116.0 ± 8253.5	3.78
13	5.484	Furfural	Almond, sweet, caramel	8212205.0 ± 212627.1	24.27
14	6.197	2-Furanmethanol	Bread, sweet, caramel	541472.5 ± 3394.5	1.60
15	6.615	Ethylene diacetate	Flower, vegetable	177882.5 ± 1821.8	0.53
16	7.742	Heptenal	Vegetable, butter, almond	95235.5 ± 4315.9	0.28
17	7.941	2-Acetylfuran	Sweet, almond, nutty, caramel	1188998.0 ± 3465.3	3.52
18	8.060	2,5-Dimethyl pyrazine	Roasted, nutty	5356709.0 ± 20387.7	15.86
19	9.736	*trans*-2-Heptenal	Fruity, butter	53083.5 ± 2787.4	0.16
20	9.833	5-Methyl-2-furfural	Bread, sweet, caramel	2114048.0 ± 92688.7	6.24
21	10.998	Furan, 2-pentyl-	Vegetable, soul, fruity	93139.0 ± 274.8	0.28
22	11.110	Furfuryl acetate	Banana, fruity	59594.5 ± 1830.8	0.18
23	11.276	Pyrazine, 2-ethyl-6-methyl-	Roasted, hazelnut	281671.5 ± 261771.5	0.84
24	11.508	Octanal	Butter, soap, grass, citrus	582663.5 ± 5257.1	1.73
25	11.984	2-Methyl-6-vinyl pyrazine	Nut	269892.0 ± 23185.8	0.80
26	12.173	Pyrazine, 2-ethenyl-5-methyl-	Coffe	78992.5 ± 4491.2	0.24
27	12.270	Corylon	Caramel	130084.5 ± 1792.4	0.39
28	12.463	L-Limonene	Vegetable, pine odor	68576.0 ± 13312.5	0.21
29	13.755	2,5-Dimethyl-4-hydroxy-3(2H)-furanone	Roasted, caramel	86089.5 ± 297.6	0.26
30	13.849	2-Acetyl pyrrole	Bread, nut, walnut, sweet, fruity	1081452.0 ± 69301.9	3.19
31	14.327	3-Ethyl-2,5-dimethylpyrazine	Roasted, nutty	520291.5 ± 18202.4	1.54
32	15.446	Nonanal	Cucumber, citrus, grass	1167439.0 ± 46597.9	3.45
33	15.734	1-(6-Methyl-2-pyrazinyl)-1-ethanone	Popcorn, coffe, roasted	31889.0 ± 339.5	0.10
34	15.980	2-Acetyl-3-methylpyrazine	Hazlenut, vegetable, caramel, roasted	58838.5 ± 4477.6	0.18
35	17.570	*trans*-2-Nonenal	Butter, melon, cucumber	51860.5 ± 6578.0	0.15
36	18.150	Caprylic acid	Butter, waxy, rancid, vegetable	71565.5 ± 747.9	0.22
37	19.340	Decanal	Soap, orange peel, butter	44456.0 ± 4884.9	0.13
38	21.395	*trans*-2-Decenal	Butter, waxy, soul	202675.5 ± 14715.8	0.60
39	25.019	2-Undecenal	Sweet, fruity, waxy	105422.5 ± 886.2	0.32
40	26.016	Vanillin	Vanilya, cream, chocolate	43059.5 ± 2936.1	0.13

†RT: Retention time.
††Aromatic definitions of the volatile compounds are found in the web pages of http://www.thegoodscentscompany.com/index.html#

provide information about how much that compound contributes to the perceived aroma intensity. To determine how an aromatic compound contributes to the perceived aroma of a sample, the odor (aroma) threshold value for that compound must be known. By definition, the odor threshold identifies the minimum concentration at which the specific aroma of a compound could be defined by human subjects. It is well known that some volatiles produce intense aroma sensations at very low levels and some cannot be perceived at even higher concentrations. Consequently, how an individual compound contributes to the perceived aroma of a sample could only be known with the odor threshold and actual concentration values together. Odor threshold values also depend on the matrix (aqueous, oil or other) in which the release kinetics could be different. Unfortunately, only a limited number of odor threshold values in specified matrices for some very common volatiles are known. Thereby, the characteristic aroma of a food sample is rather a cumulative effect of all aromatic volatiles present (Meilgaard *et al*., 1991). We could find only one study reporting the volatile constituents of roasted tiger nut oil (Lasekan, 2012). In that study, the tubers were roasted at 150 °C for 15 min before supercritical fluid extraction. Therefore, it differs from our sample, which was produced by the cold-pressing technique without any roasting. They identified 75 compounds, and found vanillin, 5-ethylfurfural, 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one, phenyl acetaldehyde, ethanone and 1-(4-hydroxy-3-methoxyphenyl) as the most active odor compounds. Further, they investigated chocolate, caramel, spicy, honey-like, and faint vanilla as the most common descriptors for the odor-active compounds identified. Although the nuts were not roasted and only cold-pressed in this study, the most common aroma compounds seem quite similar. On account of this, our study significantly contributes to the literature.

3.6. Sensory properties of the tiger nut oil

The Quantitative Descriptive Analysis (QDA) results for the tiger nut oil samples are summarized in Table 7. The oil was described according to six defined, panel-selected descriptors, (Table 1), which were almond, nutty, roasted, straw, sweet and soil. Among the six sensory descriptors, nutty (7.0), almond (5.2) and roasted (4.3) were the dominant attributes. The results of the QDA clearly match well with the volatile aromatic data presented in Table 6. Further, similar sensory descriptors (chocolate-like, butterscotch-like, roasted-coffee, peanut-like, and nutty sweet) were used in the study by Lasekan (2012) for roasted tuber seed oils. Roasting obviously yielded some new aroma and flavor attributes. Generally, the sensory properties of cold-pressed tiger nut oils provided in this study indicate that this oil would be used for edible purposes, since no offensive compound or attribute was present.

Table 7. The Quantitative Descriptive Analysis (QDA) test results of the cold-press produced tiger nut oils.

	Tiger Nut Oil (Mean ± SD, n = 6)
Almond	5.2 ± 0.5
Nutty	7.0 ± 1.0
Roasted	4.3 ± 0.7
Straw	2.0 ± 0.5
Sweet	2.0 ± 0.3
Soil	1.0 ± 0.1

3.7. Consumer test of tiger nut oil

The 5-point hedonic scale (1= dislike extremely to 5= like extremely) was used to assess the appearance, smell/aroma, taste/flavor, and general acceptance of the oil sample with 100 volunteer consumers participating to the study (Table 8). The mean scores for appearance, smell/aroma, taste/flavor and general acceptance were 4.19, 4.20, 4.29 and 4.17, respectively. For all measured attributes, the scores were above 4.0 points, indicating that the samples were well-liked by the consumers. In this context, this study provides the first results on consumer preferences for this oil.

Table 8. The consumer hedonic test results of the cold-press produced tiger nut oils.

	Tiger Nut Oil (Mean ± SD, n = 6)
Apperance	4.19 ± 0.69
Smell/aroma	4.20 ± 0.77
Taste/flavor	4.29 ± 0.62
General acceptance	4.17 ± 0.76

Hedonic scale: 1 = Dislike extremely to 5 = Like extremely

4. CONCLUSIONS

In this study, tiger nut oils were produced by a cold-pressing technique and analyzed for their physicochemical and thermal properties, aromatic volatile compositions, sensory descriptions, and consumer preference. The physicochemical properties were appropriate and acceptable. The main components were oleic acid, α-tocopherol, and α-sitosterol. The oil included some nutty, almond and vanillin-like aromatics, and sensory tests approved the attributes. In general, the consumers liked this oil. The high oil contents, unsaturated fatty acids, and presence of bio-active compounds suggest that this oil can be used in the edible oil and functional food industries.

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6. DECLARATION OF COMPETING INTEREST

The authors of this article declare that they have no financial, professional or personal conflicts of interest that could have inappropriately influenced this work.

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