Some physico-chemical properties, fatty acid compositions, macro-micro minerals and sterol contents of two variety tigernut tubers and oils harvested from East Mediterranean region

Erman DUMAN*  

Abstract
This research on Yellow sugar and Honey tuber varieties tigernut tubers’ oils physico-chemical properties were investigated to collected from Adana province in Turkey. The 1000 seed weight, width, length, moisture, ash and ascorbic acid values of yellow sugar and honey wax tubers were established as 343,496 and 327,524 g, 7.61 and 8.76 mm, 17.07 and 15.23 mm, 6.41 and 6.45%, 1.19 and 1.38% and 67.34 and 58.81 mg/kg, respectively. Free fatty acidity, peroxide values, refractive index, density, iodine number, total sterol, saponifiable matter, unsaponifiable matter, mineral values of yellow sugar and honey tuber tigernut oils were determined. The palmitic, stearic, oleic and linoleic acid contents of yellow sugar and honey tuber oils were determined as 13.02 and 12.76, 3.92 and 3.94, 69.34 and 69.91, and 11.93 and 11.80%, respectively. Campesterol, sitigmasterol and β-sitosterol contents of yellow sugar and honey tuber tigernut oils were determined. According to results, chufa tubers have high oil, oleic acid, sterol and some minerals suggest that these tubers may be very useful effect on human health and tuber oils can be use in the vegetable oil industry.

Keywords: tigernut; variety; minerals; fatty acid composition; sterols; food.

Practical Application: Two types of tigernut tubers nutritional properties and detail oil specifications.

1 Introduction
Tigernut (Cyperus esculentus L.) widely cultivates in some countries around the world for example Greece, Italy, Turkey, Israel and West Africa countries (Temple et al., 1989; Eteshola & Oraedu, 1996). Tigernut called as chufa, earth almond is an edible perennial plant (Özcan et al., 2010; Yoon, 2015). Tigernut seed has abundant in oil amount (17-33%) and it is a potential new source for edible oil (Addy & Eteshola, 1984; Sánchez-Zapata et al., 2012; Yoon, 2015). Futhermore, Chufa oil can be used for many industrial purposes (Barninas et al., 2001). Tigernut has important components in terms of nutritionally such as oil amount, antioxidant matters and it used as raw, roasted, dried, baker (Belewu & Belewu, 2007; Oladele & Aina, 2007). Tuber is used in many for food, such as powder, delicious nut-like flavour, beverage. In addition it is used in cooking and in making soap (Facciola, 1990). Tigernut oils have content rich palmitic and oleic acids in terms of fatty acid composition (Eteshola & Oraedu, 1996; Yoon, 2015). In addition, tiger nut tubers have been used in the field of health against various diseases such as aphrodisiac, carminative, diuretic (Chopra et al., 1986). The aim of this research is to determine from wide angle some physical properties, fatty acid composition, sterol contents and mineral contents of chufa tuber and oils harvested from East Mediterranean region in Turkey.

2 Materials and methods

2.1 Material

The chufa seeds (Cyperus esculentus L.) varieties used in the research were provided from Eastern Mediterranean Agricultural Research Institute in Adana province in Turkey in 2017. For this purpose, 10 kg of chufa seed collected from in Adana province region were kept in air-permeable cardboard boxes to prevent mold and sweating. All samples dried for one week on the sun. On addition, samples variety were identified as Sarı şeker (yellow sugar) and Bal yumru (honey tuber) varieties by Adana Eastern Mediterranean Agricultural Research Institute.

2.2 Physical analysis

Tuber weight
For each species, a certain number of both chufa tubers were removed and weighed on a scale with a precision of 0.01 g counted to 1000 seeds (Türk Standartları Enstitüsü, 2014).

Tuber dimensions
In the large, medium and small sized tubers, width and length measurements were made with digital calipers and average of the results was taken as mm.
Moisture

Approximately 10 g of the sample was previously dried at 105 °C and weighed in intact vessels. After weighing, the product at 105 °C was dried to constant weight. The amount of moisture was calculated as % weight loss (American Association of Cereal Chemists International, 1999).

Ash

Approximately 3 g of sample from each sample was previously dried at 105 °C and weighed to the ash crucible cooled. It was then placed in the ash oven at 700 °C and the samples were expected to fully sideways (until the ash was completely whitened). The time for complete combustion of the samples ranged from 5 to 6 hours. The amount of ash was calculated as percent (American Association of Cereal Chemists International, 1999).

Oil extraction

About 5-10 g of dried, ground tigernut tuber was placed in the cartridge and the cartridge was placed in the extractor. By connecting the balloon and the extractor, the petroleum ether was put on the cartridge until it was able to circulate and was removed after 6 hours. The petroleum ether was distilled off. The glass balloon was heated to 103 °C for a maximum of 3 hours and then cooled in a desicator and weighed then it was calculated as percent oil value based on the dry matter (Uylaşer & Başoğlu, 2000).

Determination of ascorbic acid

The ascorbic acid determination was carried out according to the method reported by Vázquez-D eriz et al. (1994). For determination of ascorbic acid used below conditions with HPLC.

Instrument: SHOMADZU GC-2025
Injection volume: 10 µL
Reverse phase separation: Zorbax ODS (4.6 × 250 mm, 5 µ)
Flow rate: 0.5 mL/min
Detection wave length: 245 nm

Determination of mineral contents

The mineral contents determination was carried out according to the method reported by Skujins (1998). After the sample burning and preparation steps according to the specified method. Analysis was performed using a ICP-AES (Varian-Vista). RF Power used 0.7-1.5 kw (1.2-1.3 kw for Axial). Plasma gas flow rate used (Ar): 10.5-15 L/min (Skujins, 1998).

Physico-chemical analysis of oils

Tigernut tuber oils some physical and chemical analysis; density, free fatty acidity, peroxide value, iodine value, refractive index, saponification value, unsaponification matter, and total sterol values were analysed according to AOCS methods (American Oil Chemists’ Society, 1998; Food Safety and Standards Authority of India, 2015).

Oils colour

15-20 mL Tigernut tubers oils were filtered by using Whatman (No. 22) paper, and placed into separate containers of 1 inch. The color of oils were measured by Lovibond PFX tintometer 880 for 22 °C. The measurements of the color of each oil was carried out through three different reading (American Oil Chemists’ Society, 2017)

Fatty acid composition

The fatty acid composition determination was carried out according to the method reported by Hisıl (1998). After the samples methyl esters (FAME) and preparation steps according to the specified method. For determination of fatty acid composition used below conditions with GC.

Working condition of gas chromatography

Instrument: SHIMADZU GC-2025
Silica capillary column (RTX-2330): (100 m × 0.25 mm i.d.; film thickness 0.20 micrometer)
Support material: Chromosorb W(AW-DMCS) (60-80 mesh)
Dedector: FID (Flame Ionization Detector)

Temperature

Column: 180 °C
Enjector: 200 °C
Dedector: 200 °C

Flow

Carrier gas (N₂): 30 mL/min.
Combustible gas (H₂): 28 mL/min.
Dry air: 220 mL/min.
Printer: Chromatopac CR 6A (Shimadzu)
Enjection volume: 1 µL

Sterol composition

The sterol composition determination was carried out according to the method reported by Phillips et al. (2005).

Working conditions of gas chromatography;

Instrument: Agilent GC
Silica capillary column (30 m × 0.32 mm i.d.; film thickness 0.25 micrometer).
**Deductor: FID (Flame Ionization Detector)**

**Temperature**
Coloum: The column temperature was increased from 0 to 60 °C (2 min) and then from 60 to 220 °C (18 min) and finally held at 220 °C (35 min).

**Flow**
Carrier gas (He): 45 mL/min.
Combustible gas (H₂): 45 mL/min.
Dry air: 45 mL/min.
Enjection volume: 1 μL

**2.3 Statistical analysis**

Statistical analyzes were performed using SPSS 17.0 for Windows. One-way ANOVA was used for analysis of variance of the results of the research, and importance scores between the mean were determined by Duncan Multiple Comparison Test. All analyses were carried out three times and the results are mean ± standard deviation of independent tigernut oils (Püskülcü & İkiz, 1998).

**3 Results and discussion**

The physico-chemical properties of yellow sugar and honey tuber tigernut samples are given in Table 1. Yellow sugar and honey tuber tigernut samples contained 22.59% and 21.52% oil, respectively. The 1000 seed weight, width, length, moisture, ash and ascorbic acid values of yellow sugar and honey tubers were established as 343.496 and 327.524 g, 7.61 and 8.76 mm, 17.07 and 15.23 mm, 6.41 and 6.45%, 1.19 and 1.38% and 67.34 and 58.81 mg/kg, respectively.

The physico-chemical properties of yellow sugar and honey tuber tigernut oil samples are given in Table 2. Free fatty acidity, peroxide values, refractive index, density, iodine number, total sterol, saponifiable matter, unsaponifiable matter values of yellow sugar and honey tuber tigernut oils were determined as 2.69 and 3.25%, 20.43 and 14.13 meqO₂/kg, 1.4624 and 1.4680 n², 0.899 and 0.893, 81.10 and 81.41, 2612 and 2012 mg/kg, 1.4674 refractive index for tigernut oil.

Mineral contents of yellow sugar and honey tuber tigernut are presented in Table 3. P, K, Ca, Mg and S contents of tubers were the major minerals of both yellow sugar and honey tuber tigernut samples. While yellow sugar contains 13443 mg/kg P, 8673 mg/kg K, 1159 mg/kg Ca, 1203 mg/kg Mg and 1297 mg/kg S, honey tuber tigernut contained 14615 mg/kg P, 7456 mg/kg K, 1648 mg/kg Ca, 1431 mg/kg Mg and 1266 mg/kg S. In addition, both yellow sugar and honey tuber tigernut tubers contained 85 mg/kg and 104 mg/kg Fe, 8.6 mg/kg and 8.6 mg/kg Cu, 4.9 mg/kg and 6.9 mg/kg Mn, 10.3 mg/kg and 10.0 mg/kg Zn and 2.9 mg/kg and 3.2 mg/kg B, respectively. When results were compared, some micro and macro elements; as P, Ca, Mg, Fe, Mn and B were found in honey tuber tigernut tubers higher than yellow sugar tigernut tubers. Also, Glew et al. (2006) reported 52.9 ug/g Fe, 188 ug/g Ca, 763 ug/g Mg, 11.2 ug/g Zn, 55732 ug/g K.

Tocopherol amounts of yellow sugar and honey tuber tigernut oils are are shown in Table 4. α-tocopherol and β-tocopherol were the key tocopherol of tigernut tuber oils. The tocopherol amounts of tuber oils changed depending on varieties. The α-tocopherol and β-tocopherol contents of yellow sugar and honey tuber oils were determined as 17.54 and 7.04, 15.89 and 9.44 mg/100 g, respectively. When results were compared, β-tocopherol amounts were found in honey tuber tigernut tubers higher than yellow sugar tigernut tubers. Yeboah et al. (2012) were determined α-tocopherol (86.73) and β-tocopherol (33.37 μg/g) for tigernut oil tocopherol amounts.

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### Table 1. Physico-chemical properties of yellow sugar and honey tuber tigernut tubers.

| Variety          | 1000 tuber weight (g) | Width (mm) | Length (mm) | Moisture (%) | Ash (%) | Ascorbic acid (mg/kg) | Oil (%) | Free fatty acidity (%) | Peroxide value (meqO₂/kg) |
|------------------|-----------------------|------------|-------------|--------------|---------|-----------------------|---------|------------------------|--------------------------|
| Yellow sugar     | 343.49 ± 1.18a        | 7.61 ± 0.31b | 17.07 ± 0.32a | 6.41 ± 1.21ab | 1.19 ± 0.36b | 67.34 ± 1.48a | 22.59 ± 2.45a | 6.41 ± 0.36a | 20.43 ± 1.39a |
| Honey tuber      | 327.52 ± 2.37b        | 8.76 ± 0.23a | 15.23 ± 0.15b | 6.45 ± 0.97a | 1.38 ± 0.41a | 58.81 ± 1.57b | 21.52 ± 1.29b | 3.25 ± 0.84a | 14.13 ± 1.45b |

± mean standard deviation. Values within each column followed by different letters are significantly different (p < 0.05).

### Table 2. Physico-chemical properties of yellow sugar and honey tuber oils tigernut tubers.

| Variety          | Refractive index (20 °C) | Density (20 °C) | Iodine number | Total sterol (mg/kg) | Saponifiable number (mgKOH/g) | Unsaponifiable matter (%) | Color |
|------------------|--------------------------|-----------------|---------------|----------------------|-------------------------------|---------------------------|-------|
| Yellow sugar     | 1.4624 ± 0.0034a         | 0.899 ± 0.006a  | 81.10 ± 2.56ab | 2612 ± 12.57a        | 164.76 ± 13.45a              | 0.59 ± 0.09a              | Red   |
| Honey tuber      | 1.4680 ± 0.0021a         | 0.893 ± 0.003ab | 81.41 ± 3.84a | 2012 ± 28.36b        | 158.98 ± 4.86b              | 0.40 ± 0.03b              | Yellow|

± mean standard deviation. Values within each column followed by different letters are significantly different (p < 0.05).
Macro and micro element contents of yellow sugar and honey tuber tigernuts tubers (mg/kg).

| Variety       | P      | K      | Ca     | Mg     | S      | Fe     | Cu     | Mn     | B      | Zn     |
|---------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Yellow sugar  | 13443 ± 27.58b | 8673 ± 46.11a | 1159 ± 13.27b | 1203 ± 10.23b | 1297 ± 16.82a | 85 ± 3.28b | 8.6 ± 0.9a | 4.9 ± 0.3b | 2.9 ± 0.3b | 10.3 ± 0.7a |
| Honey tuber   | 14615 ± 104.12a | 7456 ± 28.96b | 1648 ± 9.85a | 1431 ± 11.87a | 1266 ± 14.76ab | 104 ± 7.56a | 8.6 ± 1.1a | 6.9 ± 0.7a | 3.2 ± 0.9a | 10.0 ± 1.3ab |

± mean standard deviation. Values within each column followed by different letters are significantly different (p < 0.05).

Tocopherol contents of yellow sugar and honey tuber tigernuts tubers (mg/100 g).

| Tocopherol | Yellow sugar | Honey tuber |
|------------|--------------|-------------|
| α-tocopherol | 17.54 ± 0.07a | 15.89 ± 0.10b |
| β-tocopherol | 7.04 ± 0.13a | 9.44 ± 0.09b |

± mean standard deviation. Values within each column followed by different letters are significantly different (p < 0.05).

Fatty acid composition of tigernut (Cyperus esculentus L.) tuber oils (%).

| Fatty acids          | Yellow sugar | Honey tuber |
|----------------------|--------------|-------------|
| Myristic             | 0.26 ± 0.05a | 0.07 ± 0.01b |
| Myristoleic          | 0.02 ± 0.00a | 0.01 ± 0.00a |
| Palmitic             | 13.02 ± 0.17a | 12.76 ± 0.28b |
| Palmitoleic          | 0.06 ± 0.01a | 0.06 ± 0.01a |
| Heptadecanoic        | 0.04 ± 0.01ab | 0.05 ± 0.03a |
| Stearic              | 3.92 ± 0.27a | 3.94 ± 0.56a |
| Oleic                | 69.34 ± 0.31a | 69.91 ± 0.63a |
| Linoleic             | 11.93 ± 0.44a | 11.79 ± 0.56ab |
| Linolenic            | 0.02 ± 0.00a | 0.02 ± 0.00a |
| Y-linolenic          | 0.19 ± 0.05ab | 0.22 ± 0.07a |
| Arachidic            | 0.59 ± 0.09a | 0.58 ± 0.44ab |
| Cis-11-eicosenoic    | 0.23 ± 0.07a | 0.22 ± 0.09ab |
| Cis-11,14-eicosadienoic | 0.12 ± 0.03a | 0.11 ± 0.03b |
| Cis-8,11,14-eicosatrienoic | + | 0.01 ± 0.00 |
| Arachidonic          | 0.03 ± 0.01a | 0.03 ± 0.01a |
| Lignoceric           | 0.20 ± 0.03a | 0.19 ± 0.07ab |
| Nervonic             | 0.03 ± 0.01a | 0.03 ± 0.01a |

± mean standard deviation. Values within each column followed by different letters are significantly different (p < 0.05).

Sterol compositions of tigernut (Cyperus esculentus L.) tuber oils (mg/kg).

| Sterols               | Yellow sugar | Honey tuber |
|-----------------------|--------------|-------------|
| Delta 7 Avenasterol   | 13.06 ± 0.09a | 9.25 ± 0.13b |
| Delta 7 Stigmasterol  | 23.24 ± 0.11a | 17.10 ± 0.09ab |
| Eritritol-Uvaol       | 5.48 ± 0.07a | 3.42 ± 0.03ab |
| Campesterol           | 427.58 ± 1.17a | 337.01 ± 0.98a |
| Cholesterol           | 2.35 ± 0.03a | 1.00 ± 0.01b |
| Stigmasterol          | 575.42 ± 1.18a | 426.14 ± 1.23b |
| Beta-sitosterol       | 1559.39 ± 2.57b | 1212.05 ± 1.78a |
| Brassicasterol        | 5.48 ± 0.07b | 6.03 ± 0.09a |

± mean standard deviation. Values within each column followed by different letters are significantly different (p < 0.05).

Fatty acid compositions of yellow sugar and honey tuber tigernut oils are presented in Table 5. Palmatic, stearic, oleic and linoleic acids were the key fatty acids of tigernut oils. The fatty acid compositions of tuber oils changed depending on varieties. The palmatic, stearic, oleic and linoleic acid contents of yellow sugar and honey tuber oils were determined as 13.02 and 12.76, 3.92 and 3.94, 6.9 ± 0.7a, and 11.80%, respectively. Other fatty acids were found at minor levels, and under < 0.6%.

Generally, peroxide value, density, total sterol, saponifiable matter and unsaponifiable matter values and color values of yellow sugar tigernut oil were found partly higher compared to results of honey tuber tigernut oil. This results were found to some similar with Muhammad et al. (2011) and El-Naggar (2016). Özer et al. (2010) fatty acid composition reported 14.6-14.1% palmitic acid, 0.2-0.3% palmitoleic acid, 3.0-3.3% stearic acid, 71.7-73.5% oleic acid, 8.7-9.1% linoleic acid, 0.4% linolenic acid, 0.2-0.5% arachidic acid. Tigernut oils fatty acid compositions were found similar with literatures (Derman, 1998; Özer et al., 2010).

The sterol compositions of tigernut oils (yellow sugar and honey tuber varieties) are shown in Table 6. It was observed statistically significant differences between sterol contents of both tigernut oils (p < 0.05) in Table 6. When results were compared, total sterol amounts were found in yellow tuber tigernut tubers (2612 mg/kg) higher than honey sugar tigernut tubers (2012 mg/kg). Campesterol, stigmasterol and β-sitosterol contents of yellow sugar and honey tuber tigernut oils were determined as 427.58 and 337.01mg/kg, 575.42 and 426.14 mg/kg and 1559.39 and 1212.05 mg/kg, respectively. Yeboah et al. (2012) said that dominated by β-sitosterol (517.25 μg/g) and stigmastrol (225.25 μg/g) for tigernut oil sterol composition.

Acid value that is used in the free fatty acidity measurement of the free fatty acid and an important quality parameter in the edible oil industry (Belewu & Belewu, 2007; Muhammad et al., 2011). Peroxides are give an indication of the process of lipid peroxidation (Shaker et al., 2009). Özcan et al. (2010) determined as 78.0 mg/kg of chufa ascorbic acid content. In a research done in Egypt, Adel et al. (2015) reported that tigernut tuber contained 7.30% moisture, 22.14% oil and 4.33% crude protein. Also, Adeyuyitan (2011) reported that tigernut tuber contained 7.30% moisture, 22.14% oil and 4.33% crude protein. In the physico-chemical properties of tari and tigernut tubers showed partly differences depending on results of literature (Mokady & Dolev, 1970; Arafat et al., 2009). These differences can be probably due to species, climatic factors, growing conditions, harvest time, location and analytical conditions.
Ca contents of both tubers were found similar. Tubers were found abundant in some minerals such as Ca (100.0 mg/100 g), Fe (4.12 mg/100 g), K (486.0 mg/100 g), Mg (94.4 mg/100 g), Na (34.3 mg/100 g) and P (219.0 mg/100 g) (Ekeanyanwu & Ononogbo, 2010). Calcium, potassium and phosphorus are the major components of bone and teeth development. As bromine, cadmium, lead and lithium elements are very important for biological process (Macrae et al., 1993a). Cadmium is toxicological properties (Macrae et al., 1993b). These inorganic elements weren’t found in this research. The key sterols were campesterol, sitigmasterol and β-sitosterol for both tigernut variety’s oils. Also, this study aims to contribute in terms of mineral components of these tubers and sterol components of tubers oils.

4 Conclusion
As a result, this study shows that the physical and chemical properties of the two different types of chufa tubers and oils can be different properties. These differences can be caused by growing conditions, environmental conditions. Also, in both cultivars oils were found to nutritive in terms of fatty acid and sterol compositions. The high oil contents, some minerals, unsaturated fatty acids, and sterols suggest that this tuber and oil can be use in the food industry.

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