International Journal of Food Science and Nutrition
ISSN: 2455-4898
Impact Factor: RJIF 5.14
www.foodsciencejournal.com
Volume 3; Issue 4; July 2018; Page No. 212-216

Physicochemical characteristics and nutritional benefits of Nigerian *Cyperus esculentus* (Tigernut) oil

Nwozo Sarah O[1*], Nwawuba Stanley U[2]
[1, 2] Nutritional and Industrial Biochemistry Unit, Department of Biochemistry, College of Medicine, University of Ibadan, Ibadan, Oyo State, Nigeria, 200005

Abstract
Tiger nut oil, although not entirely new, still largely remains unexploited in major parts of the world and underutilized even in regions where it is cultivated, especially in Nigeria. Therefore, the aim of this study is to investigate the physiocochemical characteristics and nutritional benefits of tigernut oil. From 4 kg ground powder soxhlet extracted using n-hexane there was oil yield of 32.95%. The pale yellow oil had a specific gravity refractive index of 1.452, relative density 0.919 g/cm^3, acid value 2.21 mg/KOH/g, saponification value 143.05 mg/KOH/g, iodine value 80.37g/100g and ester value 62.68. Fat soluble vitamin content analysis revealed vitamin A (72.93UI), vitamin D (49.17UI), vitamin E (55.44UI) and vitamin K (15.3mcg) and Ca, Mg, K, Na, P, Mn, I, Ni, and Zn were found in varying proportions all of which was found to fall within the daily recommended intake limit. GC/MS revealed that it contains noticeable levels of oleic acid (15.05%), tetrasiloxane (9.245%), β-sitosterol (8.860%), n-hexadecanoic acid (7.753%) and 9-octadeceadienoic acid (7.649%). Thus, tigernut is oil rich tuber, could find application as an industrial raw material and could be used for cooking.

Keywords: tigernut oil, GC/MS, physicochemical characteristics, vitamins, minerals

1. Introduction
Tiger nut (*cyperus esculentus*) is commonly known as earth almond, *chufa*, yellow nut sedge, *zulu* nuts, locally here in Nigeria "*Aya*" (Hausa), "*ofio*" (Yoruba) and "*Akiausa*" (Igbo). Three varieties (black, brown and yellow) are cultivated; just like other sedges, the plant is most frequently found inhabiting wet marshes, edges of water bodies such as streams, ponds where it grows in coarse tufts and the tubers are daily ingredients of the diet of many people in North Africa and Spain [1]. It is widely distributed in the temperate regions within South Europe as its probable origin, and has become naturalized in Ghana, Nigeria and Sierra Leone. Interestingly, in Nigeria tiger nut is available in fresh, semi-dried and dried form in the markets where it is sold locally and consumed even uncooked [2]. Tiger nut has been cultivated as a feed for both livestock and humans; it can be eaten wholly in different forms as raw, roasted, grated, and baked and it is used in addition to other food ingredients in making ice cream and beverages [3].
Tiger nut oil is golden brown in color with a rich nutty taste and it is a good component of beauty products for both the skin and hair due to the high content of oleic acid and tocopherol [4]. It has been reported that tiger nut intake reduces low density lipoprotein-cholesterol (LDL-C), triglycerides and increases high density lipoprotein-cholesterol (HDL-C) thereby reducing the risk of arteriosclerosis [5]. It also stimulates the absorption of calcium, due to short and medium chain fatty acids, oleic acid, essential fatty acids it is recommended for both infants and the elderly because of its high content in Vitamin E and its antioxidant benefits in the cell membrane [6].
The tiger nut oil also has high monounsaturated fatty acids, similar to olive, avocado and hazelnut oil [7]. This monounstaturated oil has high unsaponifiable matter, phospholipids and other bioactive compounds such as tocopherols, phytosterols and polyphenols [8, 7]. The small round tubers found along the roots have a slightly almond flavor and are eaten raw or cooked, or made into a traditional chufa drink called *orxata*. These tubers contain high levels of protein, carbohydrate and oleic acid [9, 2], and 20 to 28 percent in the form of tiger nut oil. They have been a number of techniques reported thus far for the extraction of oil from tigernut. [10] extracted tigernut oil using both enzyme-aided pressing (EAP) and aqueous enzymatic extraction (AEE) methods and heating temperature however has no significant effect on the specific gravity, density, refractive index, saponification value and iodine value of the extracted oil [11].
Tiger nut oil shows antioxidant and scavenging activities of hydroxyl radicals [12]. It also contains phytosterol, vitamin E and β-carotene [13]. Such substances, together with the unsaturated fatty acids of tigernut oil, are responsible for the overall antioxidant activity. Additionally, Tiger nut oil is also a fantastic component of beauty products having high oleic acid content and low acidity hence excellent for the skin and it is used for soap making [14].
Tiger nut oil, although not entirely new, still remains unknown, largely unexploited in major parts of the world and underutilized even in regions where it is cultivated and a lot of people eat the tiger nut without knowing the nutritional benefits and products that can be obtained from it. Despite its high nutritional value, tiger nut oil is hardly used in food industries compared to other vegetable oils such as olive and peanut oil [7]. Therefore, further research is required to emphasize its benefit in food industries, domestic food

preparation and possible impact health. Thus, this study aims to investigate the physiocochemical properties and nutritional benefits of tigernut oil.

2. Materials and methods

Plant Collection

Tiger nut was harvested freshly from a local farm in Maiduguri, Borno State, Nigeria and identified at Botany Department, University of Ibadan. The tubers were inspected; spoilt ones were removed by hand picking. Matured healthy tubers were washed, air dried at room temperature under the ceiling fan for 2 weeks, ground using Hammer mill into coarse powder and stored in sealed cellophane bags prior to extraction.

Oil Extraction

4kg of the powdered tubers was transferred into a glass container and 7.5 L of redistilled commercial grade n-hexane as added for 72 hours, the filtrate was removed using muslin cloth. To the residue was added another 5litres of pure n-hexane and macerated in the cold for 3 days, thus the process was repeated. The combined n-hexane extract was then further filtered using filter paper and was concentrated using rotary evaporator at 30 °C to remove the solvent and was dried further using a vacuum oven set at 30°C and with a pressure of 700 mg/Hg. The obtained oil was stored in dark bottle in the refrigerator till needed for analysis.

The weight of the sample was calculated using the formula below

Weight of initial sample	=	X
Weight of bottle	=	Y
Weight of bottle + extract	=	Z
Weight of extract (W)	=	(Z-Y) =W

Determination of percentage oil yield

Solvent was freed from the oil obtained after extraction was placed over a water bath at 70 °C for 30 minutes and the volume of oil was recorded and expressed as oil content (%) as calculated below:

$$\text{Oil content} = \frac{\text{Weight of oil}}{\text{Weight of sample}} \times 100$$

Determination of physicochemical parameters

Determination of relative density

A specific density bottle was washed, dried and weighed (W0). It was filled with distilled water and weighed (W1). The water was poured off and the bottle was dried to it previous constant weight and then filled with the oil sample and weighed (W2).

$$\text{Relative density} = \frac{W2 - W0}{W1 - W0}$$

Refractive index principle determination

The prism of the instruments was cleaned thoroughly and two drops of the oil sample was placed on the prism. The temperature of the oil was allowed to equilibrate with that of the thermos stated fluid and reading of the thermometer was noted. The knobs of the instrument were set and the fluid was demarcated by a sharp line dividing the field of view into two equal halves and when line conceded with the spot marks "X" in the field of the view. The reading was taken at this point that is at normal temperature of 25°C but 40-60°C is for high melting fats [15, 16].

$$\text{Refractive index (Ri)} = R + K(T_1 - T_2)$$

Where

Ri = refractive index reduced to standard temperature (25 ^{0}C)

R = Reading obtained at temperature T^3

T_1 = Standard temperature 25 °C

T_2 = Temperature at which reading was taken.

K = substituting factor (Constant) 0.000385 for oils 0.000365

Saponification value determination

The method described by [16] was adopted. 2.0 g of the oil sample was weighed into 200 ml conical flask and 25ml of 0.5 M of ethanolic potassium hydroxide solution was added. The flask was configured to a condensing set-up and heated on a water- bath for 1 hour with frequent shaking and the content was allowed to cool. To the solution was added 1% phenolphthalein indicator and 0.5M hydrochloric acid was titrated into it. Equivalent titration was performed for the blank and generated values were employed for computation according to the following equation;

$$\text{Saponification value } \frac{A-B}{Q} \times 28.05$$

Where

A = Volume of 0.5M of Hydrochloric acid used in the blank titration.

B = Volume of 0.5M of Hydrochloric acid used in the sample titration.

Q = Weight in grams of the oil sample.

28.05 = Conversion Factor

Iodine value determination

Wij's solution was prepared; 8.0g of iodine monochloride was dissolved in 200 cm^3 glacial acetic acid. 9.0g of Iodine crystals was dissolved in 300cm^3 of carbon tetrachloride (CCl_4) the two solutions were then mixed and made up to the mark with glacial acetic acid. 10g of oil sample was weighed into a clean dry 250cm^3 conical flask and 10 cm^3 of CCl_4 was added followed by 20cm^3 of the prepared Wij's solution. The flask was stopped and kept in a dark cup board for 30 minutes at room temperature; 15ml of 10% of potassium iodide (KI) solution and 100cm^3 of distilled water was added. This was titrated against 0.2ml sodium thiosulphate solution using starch as an indicator. A blank titration was also conducted under the same conditions without the sample.

$$\text{Iodine value} = \frac{(A - B) \times (N \text{ of } Na_2S_20_35H_20) \times 12.69}{Q}$$

Where

A = volume of 0.1 M $Na_2S_20_35H_20$ solution used for the blank titration.

B = volume of 0.1 M $Na_2S_20_35H_20$ solution used for the sample titration.
Q = Weight in gram of the oil sample
12.69 = Conversion Factor.
N = Normality

Proximate analysis
The proximate analyses of *Cyperus esculentus* oil were determined according to Standard procedure [17].
Determination of Vitamins
The separation and detection of the vitamins were performed using atomic absorption spectrophotometer (AAS) according to method described by [18]. Sample was analyzed for vitamin A, D, E & K.

Determination of mineral content
Minerals content were analyzed using atomic absorption spectrophotometric methods according to [19]. Sample were analyzed for sodium (Na), potassium (K), calcium (Ca), iron (Fe), magnesium (Mg), zinc (Zn), copper (Cu) and phosphorus (P) content.

Determination of fatty acid composition
Fatty acids were determined using gas chromatography/ mass spectrometry (GC-MS) Agilent technology 7890 GC system and the model of the detector is Agilent technology 5975 MSD (Mass Spect. Detector). The column model is HP5 MS with length 30 m, internal diameter 0.320 mm, while the thickness was 0.25 µm. The oven temperature program was initial temperature of 80 ^oC held for 1 minute. It increases by 10^0 per minute to the final temperature of 240 ^0C to hold for 6 minutes. The injection volume was 1 microlitre and the heater or detector temperature is 250 ^0C. The tigernut oil extracted was put in a vial bottle and the vial bottle was placed in auto injector sample compartment. The automatic injector injected the sample into the liner. The mobile phase pushes the sample from the liner into the column where separation took place into different components at different retention time. The MS interpret the spectrum MZ (mass to charge ratio) with molar mass and structures.

3. Results

Table 1: Physicochemical Characteristics of Tigernut oil

Properties	Result
Colour	Light yellow
Texture at 37%	Liquid
pH value	4.45
Refractive index	1.452
Relative density (g/cm^3)	0.919
Acid value (mg/KOH/g)	2.21
Saponification value (mgKOH/g)	143.05
Iodine value (g/100g)	80.37
Ester value	62.68

Table 2: Proximate analysis of Tigernut oil

Parameter (%)	Tigernut oil
Crude protein	0.37±004
Crude fat	0.012±0.01
Fat	92.78±0.01
Moisture content	0.70±0.51
Ash	0.10±0.002

Results are expressed as Mean ± SD for triplicate measurement

Table 3: Vitamin composition of Tigernut oil

Vitamins	Yield (mg/100g)
Vitamin A	2.21±0.03
Vitamin D	1.49±0.05
Vitamin E	1.68±0.08
Vitamin K	1.53±0.30

Results are expressed as Mean ± SD for triplicate measurement

Table 4: Mineral composition of Tigernut oil

Mineral elements	Yield (mg/l)
Calcium	1820±20
Magnesium	241±20
Potassium	32.5±2.50
Sodium	60.5±1.80
Phosphorus	0.063±0.001
Manganese	0.75±0.02
Iron	93.00±0.5
Zinc	3.30±0.015
Nickel	5.60±0.02

Results are expressed as Mean±SD for triplicate measurement

Table 5: Fatty acid composition of Tigernut oil as determined by Gas chromatography-mass spectrometry analysis

Compound identified	Retention time (min)	%
2-Decenal	7.790	4.90
2-Dodecenal	9.204	4.51
Hexadecanoic acid, methyl ester	16.059	4.07
n-Hexadecanoic acid	16.757	7.75
Hexadecanoic acid, ethyl ester	16.97	2.30
9,12-Octadecadienoic acid	18.365	2.65
11-Octadecenoic acid, methyl ester	18.456	7.65
Oleic Acid	19.274	15.05
Linoleic acid	19.343	3.19
9-Octadecenoic acid, methyl ester	19.440	5.48
Tris(tert-butyldimethylsilyloxy)arsane	31.15	1.36
Cyclododecanone	31.485	2.41
.beta.-Sitosterol	31.783	8.86
N,N-Dimethyl-4-nitroso-3 (trimethy lsilyl)aniline	32.29	5.40
1,4-Bis(trimethylsilyl)benzene	34.5	0.32
4-Methyl-2-trimethylsilyloxy-acetophenone	34.6	4.66

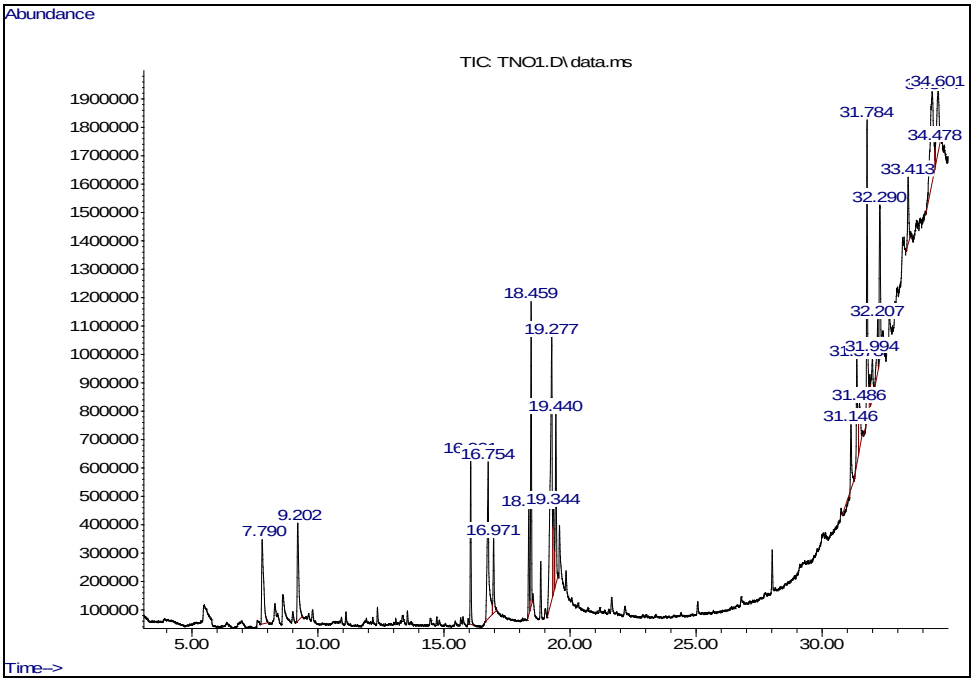

Fig 1: GC-MS Chromatogram of Tigernut oil.

4. Discussion

Physicochemical analysis of Tigernut oil revealed relative density (0.919g/cm^3) and refractive index (1.452) at 25 °C (Table 1) which falls within the range of recommended values of 1.445-1.470 refractive index for edible vegetable oils [20]. The saponification value of 143.05mgKOH/g was obtained and saponification values have been reported to be inversely related to the average molecular weight of the fatty acids present in oils [21] and a large saponification number increases the suitability of the oil for soap making. Thus qualifying tigernut oil an excellent candidate for soap making. The iodine value was found to be 80.37g/100g, tigernut oil is non-drying lipid suitable for paint making as drying oils have an iodine value above 100g/100g [22]. The ester and acid values of 62.68 and 2.21mg/KOH/g respectively is in the range (0.00 to 3.00 mgKOH/g) recommended for oil suitable for cooking [23], hence Tigernut oil could an excellent candidate for food preparation.

Proximate analyses of tigernut oil (Table 2) show that it contains crude protein (0.37±004), crude fat (0.012±0.01), fat (92.78±0.01) moisture (0.70±0.51) and ash content (0.10± 0.002) respectively. Vitamin content (Table 3) revealed vitamin A (2.21±0.03mg/100g), vitamin E (1.68±0 .08mg/100g) and vitamin D 49±0.05mg/100g equivalent to 72.93, 49.17 and 55.44 UI respectively. Vitamin K level was found to be 1.53±0.30mg/100g equivalent to (15.3mcg) and all of which was within the recommended daily intake limits (5000UI, 400UI, 30UI and 80mcg respectively) by U.S Food and Drug Administration (FDA). Vitamins are essential food components, deficiency of vitamin A and D causes blindness and rickets in children [24, 25]. Vitamin E has antioxidant properties [26], vitamin K is required to minimize blood loss in case of injury deficiency in it increases calcium deposition which could lead to coronary artery calcification and the development of heart disease [27]. Hence tigernut oil is a fat soluble vitamin rich source and could be very beneficial nutritionally.

Table 4 shows that tigernut oil contains Ca (1820±20mg/l), Mg (241±20mg/l), K (32.5±2.50mg/l), Na (60.5±1.80mg/l), P (0.063±0.001mg/l), Mn (0.75±0.02mg/l), Fe (93.00±0.5mg/l), Zn (3.30±0.015mg/l) and Ni (5.60±0.02mg/l) respectively. Mg, K, Na, P, Mn, Fe, and Zn values are within range of the daily intake recommendation by U.S Food and Drug Administration (FDA) 400mg, 3500mg, 2400mg, 1000mg, 2mg, 18mg and 15mg respectively.

The components present in Tigernut oil were identified by GC-MS analysis. The GC-MS chromatogram of 21 peaks of compounds detected were shown in figure 1. The retention time (RT), compound identified and concentrations (% peak area) are presented in table 5. The most prevailing compounds are 4.7% hexadecanoic acid methy ester, 2.65% 9, 12-octadecadienoic acid, 9-octadecenoic acid methyl ester 7.65% and 7.75% n-hexadecanoic acid (palmitic acid). Also, oleic acid was found to be 15.05% and People consuming the highest amounts of oleic acid were 89% less likely to have ulcerative colitis than those consuming the least amount of oleic acid [28] and a diet high in oleic acid may reduce the inflammation seen in obesity and non-insulin dependant obesity [29]. Linoleic acid was found to be 3.19% and linoleic acid has been shown to prevent the development of atherosclerosis, reduce body fat while improving lean body mass, and modulate immune and/or inflammatory responses [30]. However, the level of oleic acid and linoleic acid is inconsistent with the reports of [31, 10, 7] and this may be as result of the humus content of the soil were it was harvested, climatic variation, processing or storage technique.

5. Conclusion

Tiger nut oil is the fixed oil obtained from the tubers of *Cyperus esculentus* and the results of this study have

demonstrated that the oil finds application as an industrial raw material, essential food supplement and important nutraceutical.

6. References

1. Oladele KA, Osundahunsi FO, Adebowale AY. Influence of Processing Techniques on the Nutrients and anti-nutrients of Tigernut (*Cyperus esculentus* L.). World J. Dairy & Food Sci. 2009; 2:88-93.
2. Bamishaiye EI, Bamishaiye OM. Tiger nut: as a plant, its derivatives and benefits. African Journal of Food, Agriculture, Nutrition & Development. 2011; 11(5):5157-5170.
3. Sanful RE. The use of tiger-nut (Cyperus esculentus), cow milk and their composite as substrates for yoghurt production. Pak. J Nutr. 2009; 8:755-758.
4. Belewu MA, Abodunrin OA. Preparation of Kunnu from unexploited rich food source: Tiger nut (Cyperus esculentus). Pak J Nutr. 2008; 7(1):109-111
5. Belewu MA, Abodunrin OA. Preparation of Kunnu from unexploited rich food source: Tiger nut (Cyperus esculentus). World Journal of Dairy and Food Sciences. 2006; 1(1):19-21.
6. David AB. Tiger nut. A Dictionary of Food and Nutrition. Encyclopedia.com: http://www.encyclopedia.com/doc/1O39-tigernut.html. 2005; Accessed May, 2010.
7. Ezeh O, Micheal HG, Niranjan K. Tiger nut oil (*Cyperus esculentus* L.): A review of its composition and physico-chemical properties. Eur. J Lipid Sci. Technol. 2014; 166(7):783-794.
8. Sanchez-Zapata E, Fernández-López J, Angel Pérez-Alvarez J. Tiger nut (*Cyperus esculentus*) commercialization: health aspects, composition, properties, and food applications. Compr. Rev. Food Sci. Food Saf. 2012; 11(4):366-377.
9. Warra AA. Quality characteristics of oil from Brown and Yellow *Cyperus esculentus L.* Tubers. Research and Reviews: Journal of Botanical Sciences. 2013; 3(1):23-26.
10. Ezeh O, Keshavan N, Michael HG. Effect of Enzyme Pre‐treatments on Bioactive Compounds in Extracted Tiger Nut Oil and Sugars in Residual Meals. J. Am. Oil Chem. Soc. 2016; 93(11):1541-1549.
11. Adejumo BA, Olorunsogo ST, Omodiaye SL. Qualities of Tigernut Oil as Influenced by Heating Temperature. International Journal of Emerging Technologies and Engineering. 2015; 2(3):49-52.
12. Jing S, Ouyang W, Ren Z, Xiang H, Ma Z. The in vitro and in-vivo antioxidant properties of Cyperus esculentus oil from Xinjiang China. Journal Science of Food and Agriculture. 2013; 93(6):1505-9.
13. Ozcan MM, Gumuscu A, Er F, Arslan D, Ozkalp B. Chemical and fatty acid composition of Cyperus esculentus. Chemistry of Natural Compounds. 2010; 46:276-277.
14. Adejuyitan JA. Tiger nuts processing: its food uses and health benefits. American Journal of Food Technology. 2011; 6(3):197-201.
15. AOAC. Method of analysis of official analytical chemists. 14th edition. Association of official analytical chemists, Washington DC, 1980, 516-697
16. Pearson. Composition and analysis of foods, 9th edition, 1991, 509
17. AOAC. International. Official methods of analysis of AOAC international. 17th edition. Gaithersburg, MD, USA, Association of Analytical Communities, 2000.
18. Ruales J, Nour BM. Content of fat, vitamins and minerals in quinoa grains. Food Chemistry. 1993; 48:31-36.
19. AOAC. International. Official methods of analysis of AOAC international. 2 vols. 16th edition, Arlington, VA, USA, Association of Analytical Communities, 1995.
20. Pearson D. Fats and Oils" Composition and Analysis of Food 9th edition. Church Hill living Stone London, 1991, 480-497.
21. Olaoluwa R, Abolanle O, John SA, Efere AOO, Olatunji MO, Adedayo SO, *et al*. Refining, Toxicology Study and Biodiesel Potentials of Used Vegetable Oils. American Journal of Food Science and Technology. 2017; 5(3):78-88.
22. Dosumu MI, Ochu C. Physicochemical Properties and Fatty Acid Composition of Lipids extracted from some Nigeria Fruits and Seeds. Global Journal of Pure and Applied Science. 1995; 1:45-47.
23. Oderinde RA, Ajayi IA, Adewuyi A. Characterization of seed and seed oil of Hura crepitans and the kinetics of degradation of the oil during heating. Elect. J Env. Agr. Food Chem. 2009; 8(3):201-208.
24. World Health Organization. Global Prevalence of Vitamin A Deficiency in Populations at Risk 1995-2005: WHO Global Database on Vitamin A Deficiency. Geneva, 2009.
25. Kebashni T, John M. The roles of vitamin D and dietary calcium in nutritional rickets. Science Direct. 2018; 8:81-89.
26. Traber MG, Vitamin E. In: Shils, M.E., Shike, M., Ross, A.C., Caballero, B., Cousins R, eds. Modern Nutrition in Health and Disease. 10th ed. Baltimore, MD: Lippincott Williams & Wilkins, 2006, 396-411.
27. Hartley L, Clar C, Ghannam O, Flowers N, Stranges S, Rees K. Vitamin K for the primary prevention of cardiovascular disease. The Cochrane Database of Systematic Reviews. 2015; 9(9):CD011148.
28. De Silva PS, Luben R, Shrestha SS, Khaw KT, Hart AR. Dietary arachidonic and oleic acid intake in ulcerative colitis etiology: a prospective cohort study using 7-day food diaries. Eur. J Gastroenterol Hepatol. 2014; 26(1):11-8.
29. Vassiliou EK, Gonzalez A, Garcia C, Tadros JH, Chakraborty G, Toney JH. Oleic acid and peanut oil high in oleic acid reverse the inhibitory effect of insulin production of the inflammatory cytokine TNF-α both *in vitro* and *in vivo* systems. Lipids Health Dis. 2009; 26(8):25.
30. Jun HK, Yoo K, Young JK, Yeonhwa P. Conjugated Linoleic Acid: Potential Health Benefits as a Functional Food Ingredient. Annual Review of Food Science and Technology. 2016; 7:221-244.
31. Adel AAM, Awad AM, Mohamed HH, Iryna S. Chemical composition, physicochemical properties and fatty acid profile of Tiger Nut (*Cyperus esculentus* L) seed oil as affected by different preparation methods. International Food Research Journal. 2015; 22(5):1931-1938.