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Investigation into Edible and Non-edible Oil Potentials of Tiger Nut (*Cyperus esculentus*) Grown in Nigeria

Oyedele O. A.[1]*, Oladipo I. O.[2] & Adebayo A. O.[3]
[1&2]Department of Agricultural and Bio-Environmental Engineering,
The Federal Polytechnic, Ado-Ekiti, Ekiti State, Nigeria.
[3]Department of Agricultural Engineering,
Ladoke Akintola University of Technology, Ogbomoso, Oyo State
*Corresponding Author

Abstract
Oil is a major source of essential mineral content in the diets of common people in Africa. A large proportion of oil consumed in this part of the world is obtained as agricultural product. Investigation into oil potential of tiger nut was carried out using samples obtained from the Oja-Oba Market in Ado Ekiti, Ekiti State, Nigeria. The nuts were oven-dried at different level of temperature of 50, 55, 60, and 65 and 70°C. The dried samples were milled and Oil was extracted from 100 g of milled sample using soxhlet extractor. Proximate analysis was carried out on the oil to determine its physico–chemical properties. The results from the study showed highest oil yield of 23.7 % at 70°C and lowest values of 18.7% at 50 °C, highest and lowest Acid value of 1.66 mg/KOH/g at 50 °C and 1.01 mg/KOH/g at 70°C. The Free Fatty Acid (FFA) content obtained were 0.84 at 50°C and 0.51 at 70°C, while Saponification values were 68.83 mg/KOH/g at 50°C and 46.26 at 70 °C. The highest Peroxide value of 6.42 was recorded at 70°C which dropped to 4.16 at 55 °C. The Refractive Index value of 1.46 is approximately recorded for the temperature. pH levels of 6.1was recorded for all the temperatures. The moisture content recorded were 27.36% at 50 °C and 17.08% at 70^{0}C. When compared with other oil seeds commonly grown in West African Sub Region, tiger possess a high level of commercial value that could meet the need of domestic and industrial oil demand of this region.
Keywords: tiger nut, oil, proximate, physico–chemical, Saponification

1. Introduction
Tiger nut (*Cyperus esculentus*) is a tough erect fibrous-rooted perennial plant, 1 to 3 ft high, reproducing by seeds and by many deep, slender rhizomes, which form weak runners above the ground, and small tubers or nutlets at the tips of underground stems (Warra, 2013). In the past it was considered an underutilized plant as it is considered as mere weed in the majority of warm countries because of the creeping and rapidly expanding roots (Alias and Linden, 1999). *Cyperus esculentus* can be found as a weed or as a crop, it is cultivated for its edible tubers called earth almonds. It is an annual or perennial plant, growing to 90 cm tall with solitary stems growing from tuber.

Tiger nut is not really a nut but a small tuber, first discovered some 4000 years ago in ancient Egypt and is cultivated today in China, Spain and West Africa (Ofoefule et al., 2013). It has many other names like Zulu nut, yellow nutgrass, and ground almond, edible rush and rush nut, earth chestnut, and edible galingale. In Nigeria, the Hausas call it aya, Yorubas imumu, the Igbos ofio, aki Hausa in southern Nigeria.

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. Tiger nuts are under-utilized due to lack of information on their nutritional potential (Rita, 2009). Tiger nut has been reported to be eaten raw, fermented and processed as beverages. It has the medicinal quality of preventing colon cancer, heart attack and diabetis (Belewu and Belewu, 2007).

Edible oils are derived from animals and plants (Sangha *et.al* 2004). Oils from plants are classified as vegetable oil. The largest sources of vegetable oils are annual plants, which include soybeans, corn, cottonseed, groundnut, sunflower, rapeseed, melon and sesame seed (Frank, 1998, O'Brien 1998). Other sources are oil bearing perennial plants such as olive, coconut, shear, cashew and palm. Tiger nut is prepared medicinally as digestive tonic, having a heating and drying effect on digestive system and alleviating flatulence and also promote urine production and menstruation (Aremu *et al.*, 2006). Tiger nut is also used in the treatment of flatulent, in digestion, colic, diarrhea, dysentery, debility and excessive thirst (Aremu *et al*., 2007).

Tiger nut can be eaten raw, roasted, dried, and baked or be made into a refreshing milk beverage. It is also used as a flavoring agent for ice cream and biscuit, as well as in making oil, soup, starch and flour (Bonanome and Grundy, 1988). The objectives of this work are to investigate oil potential and its characterization at vary roasting temperature and the possibility of recommending it as substitute or complement for common existing vegetable oils.

2. Materials and Methods
2.1 Materials Collection and Preparation
Fresh tiger nut was purchased from a local market in Ado-Ekiti town of Ekiti State, Nigeria. The tiger nut was sorted out by removing decayed, unmatured and unwanted material so as to ensure good quality oil. About 600 g of the sorted tiger nut was then weighed and divided into three parts of 142.0, 136.1, and 140.9 g. The three samples were oven dried at 50^{0}C for 24 hours. It was then kept in the desiccators for natural cooling and then re-weighed and recorded. Also the same procedures were then repeated for four other samples at temperatures of samples of 55, 60, 65, and 70^{0}C

respectively. The five samples were then milled using electric blender to powder form for easy extraction oil (Adebayo *et al.*, 2012) and then stored in an airtight container to prevent moisture absorption.

2.2 Oil Extraction

Solvent extraction method was used to extract oil from milled Tiger using soxhlet extractor (Avalier model). 100 g of the milled sample was wrapped with filter paper and masking tape. This was inserted into the condenser of the extractor. The round bottom flask was filled with the solvent (n-hexane) up to two-third capacity of the flask. The reflux condenser was filled to the top of the extractor and water flow was turned on.

The round bottom flask was placed in the heating mantle and temperature of the mantle adjusted to 150^0C so that the solvent is brought to the evaporation point. Each extraction occurred over a period of 8 hours. When the solvent has just siphoned over the barrel, the condenser was detached and the thimble removed. The filtrate was kept in desiccators and allowed to cool at room temperature and the extracted oil was re-heated to remove the n-hexane from the oil through evaporation. These procedures were repeated for each of the five samples dried at 50, 55, 60, 65 and 70^0C.

2.1.1 Determination of Percentage Yield

The oil obtained from the extraction was transferred into a measuring cylinder which was placed over water bath for 30 min at 70˚C so as to ensure complete evaporation of solvent and volume of the oil was recorded (Warra *et al.*, 2011). The oil yied was calculated using equation (1) (Warra, 2013).

$$\text{Oil content (\%)} = \frac{\text{weight of the oil}}{\text{weight of sample}} \text{ x } 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ (1)}$$

2.1.2 Determination of Colour and Odour

The colour and odour of the oil samples was determined by observation using several independent competent individuals. Oil colour was correlated using colour charts (Okolie et al., 2012).

2.1.3 Determination of Saponification Value

Method of AOAC (1998) was followed in determination of saponification value. 2 g of the oil sample was added to a flask with 30 cm³ ethalonic KOH and the flask was then attached to a condenser for 30 min to ensure the sample was fully dissolved. After the sample has cooled, 1 cm³ of phenolphthalein was added and titrated with 0.2 M HCL until a pink end point has reached. The sample analysis was performed using blank. Blank was also prepared using the same reagents as the sample without the oil in it. Saponification Value (SV) was calculated from the equation (2)

$$\text{SV} = \frac{(S-B)\,X\,M\,X\,56.1}{Sample\ weight\ (g)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ (2)}$$

Where S= sample titre value, B= blank titre value, M= molarity of the HCL, 56.1= molecular weight of KOH.

2.1.4 Determination of Acid Value

AOAC (1990) method of acid value determination was used. The acid value of the sample oil was determined by dissolving about 5.0-5.5 g of the sample oil in a hot mixture of 25 ml diethyl ether and 25ml 95% v/v ethyl alcohol. The hot solution was neutralized with 0.1 M NaOH using phenolphthalein as indicator. Acid Value (AV) was then calculated using relationship in (3)

$$\text{AV} = \frac{\text{AC X TV}}{\text{W}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ (3)}$$

Where M = molar mass of KOH (56.1), C = concentration of KOH (0.1M), TV = titre value, W = weight of oil sample

2.1.5 Determination of Free Fatty Acid

0.5 g of sample oil was boiled with 5cm3 of ethanol allowed to cool and 2 drops of phenolphthalein indicator was added, then titrated with 0.1N NaOH until pink color disappear. Free fatty acid was calculated using the expression in (4) (AOAC, 1998).

$$\text{FFA} = \frac{\text{V X Na x F}}{\text{Ws}} \text{ x } 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ (4)}$$

Where, V = Titre value, Na = normality of acid, F = equivalent weight of free fatty acid, Ws = weight of sample

2.1.6 Determination of Peroxide Value

1 g of the sample was weighted into a clean dry boiling tube and 1 g powdered potassium iodide (KI) plus 20 ml of the solvent mixture (2 volume glacial acetic acid and 1 vol. chloroform) was added. The mixture was boiled vigorously for 1minute using boiling water as the heating source. Then the boiled mixture was transferred quickly into a flask containing 20 ml of 5% KI and the tube was washed twice with 25 ml of distilled water into the flask. The content of the flask was titrated with 0.002 M sodium thiosulphate ($Na_2S_2O_3$) solution using starch as indicator. A blank was determined at the same time and condition. The peroxide value of the sample oil was calculated using equation (5) (AOAC, 1990).

$$\text{peroxide} = \frac{2(a-b)}{w} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ (5)}$$

Where; a = titrate value for the sample, b = titrate value for the blank and w = weight of sample.

2.1.7 Determination of Refractive Index and pH Level

The refractive index was determined using the refractometer (Erma hand refractometer) as used by (Ayo and Agu 2012). It has range of 0-32%. A drop of the oil was placed on the surface of the refractometer and the reading was taken. The oil was characterized for pH using a pH meter.

2.1.8 Determination of Moisture Content

In determining the moisture content of an acid, a clean flat dish of silica, platinum was dried, the cool dish was weighed (W_1) and 5 g of the oil was introduced into the dish and was spread after which it was weighed accurately (W_2). The dish and its content W_2 as transferred into an air oven at 105^0C to dry for about 3 hours. Then a pair of tongs was used to transfer the dish into desiccators, allowing it to cool down before the oven for half an hour and was allowed to cool in the desiccators after which it was then weighed. Finally the dish was returned to the oven for half an hour and was allowed to cool in the desiccators after which it was then weighed. This process was repeated for each of the five samples. Equation (6) was then used to calculate the moisture content.

$$\text{Moisture Content} = \frac{(W_1 - W_2)}{(W_2 - W_1)} \times 100 \quad \ldots\ldots (6)$$

Where: W_1 = weight of the cool dish, W_2 = weight of the cool dish with the sample and W_3 = weight of the dried dish and the sample

3. Results and Discussion

3.1 Results

The results obtained from the study are presented in Tables 1. It shows the values of the oil properties observed from the extracted oil from tiger nut at different levels of drying temperature.

Table1: The Physicochemical properties of Extracted Tiger nut oil

Oil parameters	Drying temperature (^{0}C)				
	50^0C	**55^0C**	**60^0C**	**65^0C**	**70^0C**
Oil yield (%)	18.7	18.8	22.1	23.6	23.7
Acid Value (mg/KOH/g)	1.664	1.402	1.309	1.206	1.010
Free fatty acid (%)	0.84	0.70	0.65	0.56	0.51
Saponification Value (mg/KOH/g)	68.83	68.00	56.10	48.62	46.26
Peroxide value	6.15	4.16	6.27	6.40	6.42
Refractive value	1.464	1.450	1.460	1.452	1.455
pH level	6.10	6.13	6.06	6.10	6.10
Moisture content	27.36	25.95	25.57	17.38	17.08
Freezing point (^{o}C)	−4.3	−4.3	-1.3	−5.2	−6.2
Melting point (^{o}C)	5.5	7.3	21.3	11.7	9.0
Colour	Light yellow	*light yellow*	Light yellow	light yellow	*Golden yellow*
Odour	Pleasant	*Sweet smell*	Sweet smell	Sweet smell	*Sweet smell*

3.2 Discussion

Oil yield: Table 1, shows that the highest oil yields from the tiger nut is 23.7% and lowest 18.7% at drying temperature of 70 and 50^0C respectively. This is higher than the value reported by (Ofoefule, 2013) as 16% but lower than yield of brown and yellow tiger nut reported as 26.15±3.142% and 27.50± 5.721% respectively, by (Warra, 2013). Comparing these values with other seeds oil; the values lower than 36.75% reported for *Nicotiana tabacum* seed oil and recommended production of hair shampoo (Chinweuba, 2013), groundnut oil (47.2 %), safflower (30.5 %) (Casten and Snyder, 2010) and chest nut (41.9%) reported by (Oyedele and Oladipo, 2014). The value is closely to 21.2% and 23.9% optimum yield for pericarp (peels) of avocado apples extracted by indirect and direct extraction using n–hexane respectively, which was recommended for as a potential substitute for most oils used for cosmetics and health care production (Adama and Edoga, 2011). The oil content of the tiger nut is higher than Soybean (21.0 %), corn (4.5 %) and cotton seed (22.9 %) as reported by (Esuoso and Odetokun, 1995). This shows that tiger nut can as well produce similar quantity like Soybean and cotton seed oil. It also indicates that processing of chestnut oil for industrial or edible purpose is possible.

Acid value: The results show that highest Acid values of 1.66 and lowest value of 1.01 mg/KOH/g are recorded at drying temperatures of 50 and 70^0C respectively (Table 1). This is greater than 0.41±0.015 mg KOH/g and 0.48±0.014 mg KOH/g obtained for brown and yellow *Cyperus esculentus* oil by (Warra, 2013); 0.81 ± 0.01 for cotton seed oil

(Warra et al., 2011b) and 0.421 mgKOH/g reported for *Adansonia Digitata* seed oil recommended for use as an ingredient in tooth paste (Chindo et al,. 2010). However, the values of 10.3 mg KOH/g for shea nut butter (Warra, et al., 2009) and 2.34 ± 0.19 mg KOH/g for *Luffa cylindrica* (Linn.) seed oil (Gafar et al., 2012) reported in literature and recommended for soap making.

Free fatty acid (FFA): The highest value of 0.84% FFA at 50^0C (Table 1), obtained from this work is far lower than free fatty acid of 77.00±1.0 and 72.67 ±1.528 (% oleic acid) obtained for the brown and yellow Cyperus *esculentus* tubers oil by (Warra, 2013); 37.96% reported for Nigerian rubber seed oil (Ebewele et al., 2010); 12.76% reported for chest nut (Oyedele and Oladipo, 2014) and also higher than free fatty acid obtained for bread-fruit oil (2.86%) and breadnut oil, 1.89% (Nwinuka et al., 2001). Oils with highly acidic free fatty acid indicate unsuitability for edible purpose except for technical purposes (Ebewele et al., 2010). This therefore suggested that the tiger nut oil extracted is suitable for ebible. Free Fatty Acids can stimulate oxidative deterioration of oils by enzymatic and / or chemical oxidation to form off flavour components. Free fatty acid value is an indication of lipase activity (Ukhun 1986).

Peroxide values: Peroxide Value is an index of rancidity, thus the high peroxide value of oil indicates a poor resistance of the oil to peroxidation during storage (Mohammed and Hamza 2008). The peroxide value of tiger nut oil in this work has highest value of 6.42 mg/KOH/g at a drying temperature of 70^0C and lowest value of 4.16 at 55^0C was recorded. The peroxide value from tiger nut is higher than Cucumeropsis Adulis (white seed melon) 2.85 mg/KOH/g, African star apple 1.57 mg/KOH/g, as reported by (Adebayo et al. 2012); 3.80 ± 0.1 meq H_2O_2 and 4.10±0.153 meq H_2O_2 obtained for the brown and yellow Cyperus *esculentus* tubers oil by (Warra, 2013) and close to 8.33 mEq/Kg reported by (Ofoefule, 2013) falls in the same range with 6.322mgO2/kg reported for Helianthus annuus L. seed oil (Aboki *et al.*, 2012) and lower 11.75 reported for fluted pumpkin (Eddy *et al.*, 2012). The low peroxide values of the oil samples is an indication that the oils are stable and may not be susceptible to oxidative rancidity since they are produced from fresh seeds (Eddy *et al.*, 2012).

Saponification values: Saponification values of 68.83 mgKOH/g and 42.26 mgKOH/g were obtained at 50 and 70^0C respectively which are lower than 193.33±2.121 mgKOH/g and 188.33±0.707 mgKOH/g obtained respectively for brown and yellow Cyperus esculentus tubers oil (Warra, 2013); 199.42 ± 0.53 mgKOH/g for cotton seed oil (Warra *et al.*, 2011b); 213mgKOH/g for neem seed oil (Warra *et al.*, 2011c) and 116.88 ± 0.97 mg KOH/g for Cucumis melo Linn Seed oil (Gafar, *et al.*, 2013) reported and recommended for cosmetic applications. A saponification value of 200 mg/KOH/g indicates high proportion of free fatty acids of low molecular weight. This shows that the oil may not have a potential for use in soap making and in cosmetics industries and or the thermal stabilization of poly vinyl chloride (PVC). This property makes them useful as sources of essential fatty acids required in the body (Akanni, *et al.*, 2005).

pH Level: The high values of pH observed is approximately 6.0 (Table 1) this value shows that the oil is less acidic. This suggests that the oil is a potential source of biodiesel (Bamishaiye and Bamishaiye, 2011).

4. Conclusion

It is concluded from the results obtained that the oil is edible. The oil yield indicates that, it can be produced in commercial quantity. The moisture content obtained was higher which implies good shelf life of the oil and the odour of the oil is pleasant which implies that it will be good for cooking.

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