



EFFECT OF STORAGE CONDITIONS ON THE QUALITY OF TIGER NUT OIL

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ABSTRACT

The study investigated the effect of storage conditions on the physio-chemical properties of tiger nut oil, a product known for its nutritional and medicinal benefits. Tiger nut oil was extracted using a mechanical expeller and stored under three conditions: room temperature (28°C), refrigeration (4°C), and freezing (-18°C) for two months. Key parameters analyzed before and after storage included moisture content, iodine value, saponification value, free fatty acid content, and percentage impurity. It was revealed that storage temperature had no significant impact on the oil's quality attributes at ($p \leq 0.05$). Moisture content remained stable, ranging from 2.27% to 2.29%. The highest moisture content was observed under freezing condition (2.29%), while the lowest was at room temperature (2.28%), with minimal variation under different temperatures. Percentage impurities ranged from 0.83% to 1.00% and iodine value ranging from 31.69 g I/100g to 31.90 g I/100g, also displayed no significant changes, confirming the oil's resistance to contamination and oxidation. Saponification values varied from 186.313 to 189.643 mg KOH/g without significant impact from either factor; While a slight increase in free fatty acid content was observed at room temperature (4.98%), the values remained within acceptable limits, indicating the oil's resistance to hydrolytic degradation. Tiger nut oil exhibits remarkable stability across various storage conditions, making it suitable for storage under ambient, refrigerated, or frozen environments without compromising quality.

Keywords: Quality, Storage, Conditions, Tiger nut, Oil, Shelf-life

INTRODUCTION

Tiger nut (*Cyperus esculentus*), an edible perennial sedge plant of the *Cyperaceae* family, It is highly valued for its health benefits due to its rich contents of dietary fibres, protein, vitamins, minerals such as potassium, magnesium, and calcium, and natural sugars (Oke, *et al.*, 2019); Awulu, *et al.*, 2018). Versatile in its applications, it can be roasted, dried, or processed into refreshing and nutritious milk known for its health-promoting properties. Despite its numerous benefits, tiger nut remains an underutilized crop. It grows abundantly in Nigeria, other parts of West and East Africa, Spain, and the Arabian Peninsula, where it plays a significant role in traditional diet (Omale, *et al.*, 2020). The oil extracted from these tubers is rich in oleic acid and tocopherols, demonstrates exceptional oxidative stability and quality, comparable to olive oil and other common vegetable oils used in food related application and can be considered a competitive lipid for food preparation anywhere, making it suitable for culinary and industrial application (Aljuhaimi, *et al.*, 2018; Omale, *et al.*, 2023). Tigernut oil contributes to the decrease of cholesterol, having also the potential in reducing the risk of coronary heart disease and atherosclerosis (El-Naggar, 2016). According to Aljuhaimi *et al.*, (2018) Tigernut oil has golden colour and a nutty taste which makes it ideal for different users. The main composition of unsaturated fatty acids in tigernut oil is oleic acid, with a content of 77.60%, which is 3.6 times higher than that of soybean oil, and even higher than that of olive oil (Li, *et al.*, 2021). The high content of oleic acids makes tiger nut oil a very nutritious and health enhancing oil (Nina, *et al.*, 2020). The quality and shelf life of tiger nut oil are highly susceptible to storage conditions. Factors like temperature, light exposure, and oxygen significantly influence its stability, with oxidative rancidity being a primary cause of degradation. Study by Ogori *et al.*, (2021) emphasize on the quality characteristics of stored varieties of tigernut oil and Nina *et al.*, (2020) studied storage condition on the properties of extracted tigernut oil from different cultivars Despite existing research, there is a lack of comprehensive studies detailing how specific storage conditions



impact the physico-chemical properties of tiger nut oil over time. This study aims to address this gap by evaluating the effects of various storage conditions, providing essential guidelines for maintaining oil quality from production to consumption.

MATERIALS AND METHODS

Materials

The following materials were utilized for the study: dried tiger nuts as the primary ingredient for oil production, a mechanical oil expeller for extracting the oil, a weighing balance for precise measurements, storage containers, a fridge, a freezer, a thermometer, a record book, beaker, heating mantle, Aliquots of ethanol, 0.1N NaOH, phenolphthalein indicator, 250mL flask, chloroform, pipette, 30% potassium iodide, distilled water, 0.1N sodium thiosulphate, starch solution, silica dish, oven, dessicator, potassium hydroxide, reflux condenser, 0.5N hydrochloric acid, petroleum ether, Wattman no 4 paper.

Sample Preparation

Tiger nuts was sourced from North Bank Market in Makurdi LGA of Benue State. The tiger nuts were cleaned and sorted thoroughly to remove any impurities, then processed into oil using an existing oil expeller. Three storage conditions were used; room temperature, refrigeration temperature, freezing temperature,, and 500ml of the oil was stored each under three respective conditions in three replications. A total of 4.5 litres of tiger nut oil was used for the study. The oil was stored for two months.

Determination of Free Fatty Acid Value

A clean dry beaker was weighed and 2 g of pre-heated oil (heated to about 50°C) was added and reweighed Aliquots of ethanol was added to the oil to completely free the fatty acids and the ethanol-oil mixture was then titrated with 0.1N NaOH using phenolphthalein indicator. The volume (V) of NaOH required to produce the first permanent pink colour was recorded and the free fatty acid content of the oil was determined from the formula as describe by AOAC (2000).

$$\%FFA = \frac{M \times V \times N}{10 \times m} \quad (1)$$

Where

M = Relative molecular mass of Palmitic acid =256,

V = volume of NaOH used,

N = Normality (concentration) of NaOH used,

m = Weight of oil used,

10= constant.

Determination of Iodine Value

The iodine value (IV) indicates the degree of unsaturation of the oil. It is defined as the number of grams of iodine absorbed by 100 grams of oil (Sadoudi, *et al.*, 2017). Tiger nut oil sample was weighed 0.2g and placed in a 250mL flask, and 20mL of chloroform was then added to the sample. A solution of iodine monochloride (ICI) in acetic acid known as Wijs reagent weighed 25ml was added with the aid of a pipette and the resulting mixture stirred and stored in a dark place at 25°C for 30 minutes before 10mL of 30% potassium iodide was added to the mixture as well as 100mL of distilled water. The mixture was then titrated with 0.1N sodium thiosulphate until the yellow colour almost disappeared. One milliliter of starch solution was then added and the mixture titrated further until the blue starch-iodine colour disappears. A blank titration was also carried out and the Iodine value calculated using the formula below:

$$\text{Iodine value} = \frac{TD \times 1.269}{M} \quad (2)$$

Where TD=Titre difference,

M= mass of sample (g),

1.269= constant

Determination of Moisture Content

Moisture content of tiger nut oil was determined by the AOAC (2000). Silica dish was dried and weighed. Then 5ml of tiger nut oil was added and weighed also. This was heated in an oven at



105°C for 1 hour, cooled in a dessicator containing phosphorus peroxide and weighed. This was repeated until a constant weight obtained.

$$\% \text{ Moisture} = \frac{M_s - M_h}{M_s - M_t} \times 100 \quad (3)$$

Where,

M_s = Weight of moisture dish + Sample (g),

M_h = Weight of moisture dish + sample after heating(g),

M_t = Weight of Tare/moisture dish(g)

Determination of Saponification Value

Saponification Value is the weight of potassium hydroxide, in milligrams, needed to saponify one gram of oil (Sadoudi, *et al.*, 2017). Two (2) grams of sample was accurately weighed and placed in a 250ml flask and 25ml of a mixture of equal volumes of ethanol and potassium hydroxide were added. The mixture was then heated in a water bath (coupled to a reflux condenser from the soxhlet extractor) for 30 minutes while being stirred continuously. One milliliter of phenolphthalein indicator was added and the resulting mixture titrated with 0.5N hydrochloric acid. A blank procedure was carried out and the saponification value calculated using the formula below:

$$SV = \frac{N \times Eq \times (V_0 - V_1)}{P} \times 10^3 \quad (4)$$

Where

SV = saponification value, (mg KOH/ g oil);

V_0 = volume of hydrochloric acid solution required for the blank, (mL);

V_1 = volume of hydrochloric acid solution required for the sample, (mL);

N = normality of HCl solution (0.5N);

Eq = equivalent gram of KOH (56.1 g/mol);

P = weight of the oil sample (g)

Determination of % Impurity in Tiger Nut Oil

To determine percentage impurities, 10g of tiger nut oil in already weighed 250ml Erlenmeyer flash was added to 100ml light petroleum ether, stirred vigorously and allowed to stand for 30 minutes. The solution was then filtered using Wattman no 4 paper and the residue was washed with 50ml ether to remove all oil. The filter paper with residue dried at 105°C until a constant weight. Amount of impurities present in the oil increases weight of the filter paper. Percent impurities is expressed as:

$$\% \text{ impurities} = \frac{W_r - W_p}{W_s} \times 100 \quad (5)$$

Where,

W_r = weight in g of dried residue + filter paper,

W_p = weight in g of filter paper,

W_s = weight in g of test sample

RESULTS AND DISCUSSION

Effect of Storage Conditions on Moisture Content

The moisture content remained relatively stable across all storage conditions, ranging from 2.27% to 2.29%. The highest moisture content was observed under freezing conditions (2.29%), while the lowest was at room temperature (2.28%). Statistical analysis ($p > 0.05$) confirmed no significant differences between the storage groups, emphasizing the stability of tiger nut oil against moisture-induced degradation. Moisture Content in this study is slightly lower and more stable compared to the 2.03% to 2.71% (Nina, *et al.*, 2020) and Finding from Afuape *et al.* (2021) which showed moisture content of varieties of tigernut oil to ranged from 2.54 to 4.43%.

The differences may result from variation in storage conditions and initial processing. Report by Nina *et al.* (2020) observed a gradual increase in moisture content over time, likely due to environmental factors during storage. In contrast, this study stored tiger nut oil under controlled conditions, minimizing moisture absorption. These findings emphasize the importance of reducing



moisture to enhance oil stability and shelf-life. This also implies that the tiger nut oil can be stored under various conditions without moisture-induced degradation over two months.

Effect of Storage Conditions on Percentage Impurities

The percentage of impurities ranged from 0.83% to 1.00%, with the highest level observed at room temperature and the lowest at freezing temperatures. Statistical analysis revealed no significant differences ($P > 0.05$) across the different storage conditions. This stability suggests that tiger nut oil maintains its purity during storage.

Afuape *et al.* (2021), reported percentage impurity 0.15 to 0.24%, lower than that of this study. The percentage impurity reported in this study exceeds the maximum permissible limit of 0.05% set by Codex Alimentarius Commission (CAC, 2009). This discrepancy is likely due to variation in extraction and filtration.

Effect of Storage Conditions on Saponification Value

The saponification value ranged from 186.31 mg KOH/g to 189.64 mg KOH/g, showing slight increases at room temperature. Although the values remained within acceptable limits, the minor rise suggests a potential increase in hydrolysis at higher temperatures. These results are within the range of some other report such as maize oil (189mgKOH/g) and sunflower (190mgKOH/g), as reported by Sani (2015), confirming the stability of tiger nut oil's fatty acids across storage conditions.

Effect of Storage Conditions on Iodine Value

Iodine values remained consistent across conditions, ranging from 31.69 g I/100g to 31.90 g I/100g.



Table 1: Effect of Storage Conditions and Storage Duration on the Physiochemical Qualities of Tiger nut Oil

physiochemical qualities	Storage conditions	Storage duration	mean	Standard deviation	N
Free fatty acid (g/100g)	Room temperature (28°C)	Before storage	4.79	.56	3
		After storage	5.17	.25	3
		Total	4.98	.44	6
	Refrigeration temperature (4°C)	Before storage	4.79	.56	3
		After storage	4.90	.46	3
		Total	4.85	.46	6
	Freezing temperature (-18°C)	Before storage	4.79	.56	3
		After storage	4.85	.35	3
		Total	4.82	.42	6
Iodine value (g I/100g)	Room temperature (28°C)	Before storage	31.72	.63	3
		After storage	31.77	.55	3
		Total	31.74	.53	6
	Refrigeration temperature (4°C)	Before storage	31.72	.63	3
		After storage	31.90	.80	3
		Total	31.81	.65	6
	Freezing temperature (-18°C)	Before storage	31.72	.63	3
		After storage	31.70	.64	3
		Total	31.71	.567	6
Saponification value (mg KOH/g)	Room temperature (28°C)	Before storage	186.53	4.21	3
		After storage	189.64	1.87	3
		Total	188.09	3.37	6
	Refrigeration temperature (4°C)	Before storage	186.59	4.21	3
		After storage	186.31	3.64	3
		Total	186.45	3.52	6
	Freezing temperature (-18°C)	Before storage	186.60	4.31	3
		After storage	186.84	3.31	3
		Total	186.72	3.44	6
Moisture content	Room temperature (28°C)	Before storage	2.27	.000	3
		After storage	2.29	.02	3
		Total	2.28	.02	6
	Refrigeration temperature (4°C)	Before storage	2.29	.03	3
		After storage	2.29	.02	3
		Total	2.29	.02	6
	Freezing temperature (-18°C)	Before storage	2.29	.03	3
		After storage	2.29	.01	3
		Total	2.29	.02	6
Percentage impurity	Room temperature (28°C)	Before storage	.83	.29	3
		After storage	.83	.06	3
		Total	.83	.19	6
	Refrigeration temperature (4°C)	Before storage	.83	.29	3
		After storage	.90	.10	3
		Total	.867	.20	6
	Freezing temperature (-18°C)	Before storage	.83	.29	3
		After storage	.87	.12	3
		Total	.85	.20	6



Statistical analysis indicated no significant differences ($p > 0.05$), confirming that the oil's degree of unsaturation is unaffected by storage conditions. This stability underscores the resistance of tiger nut oil to oxidative degradation.

The iodine value of this study ranging from 31.69 g I/100g to 31.90 g I/100g is observed to be significantly different from the range of 52.62 to 80.20 g I/100g reported by Omale *et al.* (2023) and range of 67.35 g I/100g reported by Guo *et al.*, (2021) and the report by Afuape *et al.* (2021) ranging from 66.11 to 69.75 g I/100g.

This variation likely results from differences in the processing conditions. These findings aligns with the classification of tiger nut oil as a non-drying oil due to its moderate unsaturation (Aremu, *et al.*, 2015)

Effect of Storage Conditions on Free Fatty Acid Content

Free fatty acid levels ranged from 4.790% to 5.167%, with a slight increase observed at room temperature. While the differences were not statistically significant, the trend suggests that elevated temperatures may accelerate lipid hydrolysis. The free fatty acid value reported in the study are higher than the 0.22% to 0.49% observed by Omale *et al.* (2023) and 0.39% to 0.41% by Ogori *et al.* (2021). Differences in the mechanical extraction process including potential oxidative stress during processing could have introduced additional free fatty acid in this study. This observation underscores the critical influence of storage and processing parameters on the quality of tiger nut oil, highlighting the importance of stringent control during production and storage to maintain a low free fatty acid level.

CONCLUSION AND RECOMMENDATION

The study evaluated the effect of storage conditions and duration on the physio-chemical properties of tiger nut oil. Results showed that storage temperature (room temperature, refrigeration, and freezing) had minimal impact on key quality parameters, including moisture content, iodine value, saponification value, impurities, and free fatty acid content. The oil demonstrated strong resistance to hydrolytic and oxidative degradation, with only a slight, acceptable increase in free fatty acids at room temperature.

Given its stability, tiger nut oil is recommended to be stored under various conditions without significant quality deterioration, making it a reliable option for long-term use.

REFERENCES

- Afuape, Z. O., Oke, E. K., Idowu, M. A., Olorode. O. o., and Omoniyi, S. A. (2021). Physical and chemical properties of tigernut oil as influenced by variety and method of extraction. *the annals of the university dunarea de jos og galati, fascicle vi-food technology*, **45**(1):129-140.
- Aljuhaimi, F., Ghafoor, K., Ozcan, M. N., Miseckaite, O., Babiker, E. E., and Hussain, S. (2018). The effect of solvent type and roasting processes on physio-chemical properties of tigernut (*Cyperus esculentus* L.) tuber oil. *Journal of Oleo Science*, **67**(7): 823-828.
- AOAC (2000). Official Methods of Analysis. 17th Edition, The Association of Official Analytical Chemists, Gaithersburg, MD, USA. Methods 925. 10, 65,17. 974.24, 992,16.
- Aremu M. O., Ibrahim H., Bamidele T. O. (2015). Physiochemical characteristics of oils extracted from some Nigerian plant foods-A review. *Chem. Engr. Res.* **32**: 36-52.
- Awulu, J. O., Omale, P. A., and Omadachi, J. O. (2018). Characterization of tiger nut oil extracted using mechanical and chemical methods. *Journal of Science and Multipurpose research*, **10**(2): 13-25
- Codex Alimentarius Commission (CAC,2009). Codex standard for named vegetable oils- codex stan 210-1999 Food and Agricultural Organization of the United Nations and World Health Organization.
- El-Naggar, E. (2016). Physiochemical characteristics of tigernut tuber (*Cyperus esculentus* L.) oil. *Middle East Journal of Applied Sciences*, **6**(4): 1003-1011.



- Gou, T., Wan, C., Huang, F., Wei, C. (2021). Evaluation of quality properties and antioxidant activities of tiger nut (*Cyperus esculentus* L.) oil produced by mechanical expression or/with critical fluid extraction. *LWT*, 141, 110915
- Li, H., Nui X. X., Chai, J. J., Gou, C. L., Sun, Y. H., Li, J. H., Li, C. (2021). Optimization of hot air drying process of tigernut and analysis of fatty acid composition of tigernut oil. *International journal of agricultural and biological engineering*, 14(6): 228-236
- Nina G. C., Ukeyima, M., Ogori, A. F., Hleba, L., Hlebova, M., Glinushkin, A., Laishevtcev, A., Derkanosova, A., Igor, P., Plygun, S., and Shariati, M. A., (2020). Investigation of physiochemical and storage condition on the properties of extracted tigernut oil from different cultivars. *Journal of Microbiology, Biotechnology, and Food Sciences*, 9(5): 988-993
- Ogori, A. F., Nina G. C., and Ukeyima, M. T. (2021). Quality characteristics of stored varieties tiger nut oil. *Food and Life*, 1(1): 29-34.
- Oke, E. K., Tijani, A. O., Abamba, R. N., Odumosu B. O., Abiola O. T., (2019). Tigernut and its food application: A Review. *Researcher*, 11(1): 73-78
- Omale P. A., Aremu, A. K., Omobowale, M. O., Olanrewaju, T. O., and Adedeji, A. O., (2023) Quality evaluation of tigernut (*Cyperus esculentus* L.) oil: Effect of processing pretreatments. 2023 *ASABE Annual International meeting*, 2300098. <https://doi.org/10.13031/aim.202300098>
- Omale P. A., Iyidiobu, B. N., Ibu, E. J. (2020). Effect of drying temperature on the nutritional quality of tiger nut (*Cyperus esculentus*). *International Journal of Engineering Applied Sciences and Technology*. 04. 399-403. 10.33564/IJEAST.2020.v04i09.051.
- Omale, P. A., Aremu, A. K., Omobowale, M. O., Olanrewaju, T. O., Adedeji A. A., (2023). Tiger nut (*Cyperus esculentus* L.) oil quality evaluation as affected by variety and processing conditions. *Journal of Essential Oil and Plant Composition*, 1(3): 228-238
- Sadoudi, R., Ahmed, D. A., Trache, M., Kecili, A. (2017). Studies on physio-chemical characteristics and fatty acid composition of commercially available Algerian frying edible oils. *International Food Research Journal*, 24(1): 60-67
- Sani, I. (2015). Physiochemical analysis and preliminary phytochemical screening of oil extract from, two varieties of *Cyperus esculentus* tubers. *International Research Journal of Pharmaceutical and Biosciences*, 36(2): 10-17