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Effects of Extraction Techniques on the Functional and Quality Properties of Tigernut (*Cyperus esculentus*) Oil

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Abstract
This research studied the functional quality of oil extracted from Tiger nut using two different methods (Mechanical extraction and Chemical solvent extraction methods) and the results revealed Moisture: 0.394 ± 0.0021 and 0.226 ± 0.0077, Fatty acid; 0.408 ± 0.0098 and 0.311 ± 0.0005, Impurity; 0.045 ± 0.0021 and 1.7025 ± 0.0106, Saponification; 206.765 ± 4.859 and 203.745 ± 0.7707, Extraction Efficiency; 41.555 ± 0.077 and 88.85 ± 0.353, iodine value; 91.236 ± 0.0047 and 87.00 ± 0.466, Peroxide; 8.381 ± 0.0707 and 0.7635 ± 0.012,Specific gravity: 0.908 ± 0.0035 and 0.880 ± 0.015, Flash Point: 147.845 ±0.021 and 156.355 ± 0.077, Pour Point: 4.005 ± 0.0071 and 4.00 ± 0.00, Cloud Point: 9.050 ± 0.0707 and 7.95 ± 1.343, Refractive Index: 1.416 ± 0.028 and 1.718 ± 0.0056, Colour: Golden Yellow and Dark-brown were obtained for mechanically and chemically extracted oil respectively. The results defined the two different oils and the values are within the WHO permissible limits for the various characterization factors. However either of the oil has some advantages and disadvantages over the other based on the values obtained.

Keywords: Effect, Mechanical, Chemical, Tigernut oil, Quality properties, Extraction

Introduction

Tiger nuts have gained significant attention in developed countries for their impressive health benefits. Rich in soluble glucose and oleic acid, these remarkable tubers also offer a robust energy profile, including starch, fats, sugars, and proteins. Additionally, they are abundant in essential minerals such as phosphorus, potassium, calcium, magnesium, and iron, all vital for maintaining strong bones, repairing tissues, supporting muscle function, and facilitating overall growth and development. Furthermore, they provide a generous supply of vitamins E and C, enhancing overall health (Adejuyitan, 2011).

In the bustling Nigerian market, tiger nuts are available in fresh, semi-dried, and dried forms. However, many consumers remain unaware of their nutritional benefits and the wide range of delightful products that can be created from this versatile tuber. From nutritious tiger nut flour to delicious bread, creamy oil, and refreshing milk, the options are plentiful (Bamishaiye & Bamishaiye, 2011).

Embracing tiger nuts means embracing a powerhouse of health and culinary potential. Tiger nut flour has a special sweet taste, which is suitable for various uses. It is an alternative to many other flours produced, from other Agricultural products because it is a gluten free diet (Belewu, 2006). It has also been recorded that tiger nut milk called kunnu-aya by Northern Nigerians is a healthy drink with nourishing and energetic power, (Mohammed, et al., 2011). Tiger nut oil (TNO) has a golden colour and a nutty aroma, which makes it ideal for different uses in cosmetic and domestic applications. It is used in deep frying because of it's exceptional resistant to chemical decomposition at high temperature and its low absorption than other frying oils (El-Naggar, 2016). Warra (2013) emphasized that the edible and stable properties of tiger nut oil position it as a formidable competitor to olive, corn,

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soybean, and cottonseed oils, making it a highly effective substitute during shortages. Its ability to remain in a consistent liquid form at refrigeration temperatures makes it ideal for salad preparation. Ezebor et al. (2005) affirmed that the oil's high oleic acid content, combined with its low levels of polyunsaturated fatty acids and low acidity, enhances its suitability for body creams. Additionally, Shaker et al. (2009) asserted that tiger nut oil is considered high quality due to its extraction process, which requires no external heat, and its natural resistance to chemical decomposition. Furthermore, research clearly shows that tiger nut oil boasts a substantial amount of monounsaturated fatty acids (exceeding 60%) and is rich in valuable phenolic and non-phenolic antioxidant compounds, making it a superior alternative to olive oil (Ezeh et al., 2014).The functional quality of oil and other valuable components is strongly dependent on the extraction method. The objective of this paper is to characterize some of the functional quality properties of tiger nut oil to establish the effect of extraction methods (mechanical and chemical) on the quality of the oil products using tiger nut obtained from vendors in Ikot Ekpene, Akwa Ibom State.

Materials and Methods

Sample Collection and Preparation

The tiger nut used for the analysis was gotten from Sani Ogun in Ikot Ekpene Local Government Area, Akwa Ibom State. The sample tubers were sorted to remove the unwanted ones. The sample were washed, dried and grind into powder, the ground sample were stored in an air tight container for the various analysis.

Chemical solvent Extraction

The sample of tiger nut was ground using a blender to produce a fine powder. Subsequently, 100 grams of the powdered tuber (Cyperus esculentus) was measured and placed into an extraction thimble. The thimble was securely closed with cotton wool at the top. Petroleum ether was introduced into the extraction flask, and the thimble was positioned within the extractor. The entire apparatus was maintained at a temperature of 50°C for a duration of one hour. Upon completion of the extraction, the process was terminated, and the oil was recovered from the petroleum ether, followed by drying in an oven. The percentage yield of the extracted material from the initial 100 grams was then calculated (Awulu et al., 2018).

Mechanical Method of Extraction

The tiger nut tubers were toasted using a gas cooker for three minutes and then crushed with a fish oil extraction machine at the Department of Chemistry, Ritman University, situated on Umuahia Road in Ikot Ekpene Local Government Area. The tiger nut sample was securely wrapped in a transparent cloth and placed in a mechanical press. Tiger nut oil (TNO) was extracted by continuously pressing two portions of the ground tiger nuts, which were positioned side by side on a locally constructed hydraulic laboratory press. Pressure was applied until the oil started to drip and the limits of the cloth sieve material were reached. The efficiency of this extraction technique was then determined, and the oil was stored for subsequent analysis (Awulu et al., 2018).

Analysis of Fnctional Properties of Tiger Nut Oil

The functional quality properties of the oil were done using a standard method described by AOAC (1990 and 1998).

Determination of Moisture Content

The cleaned silica dish was dried in an oven, cooled in a desiccator, and 5g of each sample was accurately weighed into the dish. Each dish was then placed in an oven at 105°C for 1 hour to dry, cooled in the desiccator, and weighed again. This process continued until a constant weight was reached, and the moisture content was calculated using:

$$\% \text{ Moisture} = \frac{W_3 - W_1}{W_2 - W_1 1} \times \frac{100}{}$$

Determination of Free Fatty Acid Value

Ten grams of Cyperus esculentus oil from both mechanical and chemical extraction methods were measured into 25ml conical flasks. Each flask received 50ml of a 50% ethanol and 50% petroleum ether mixture, followed by 6ml of phenolphthalein indicator. The solutions were titrated with 0.1M potassium hydroxide while shaking continuously

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until a stable pink color appeared for at least 15 seconds. This procedure was conducted for both oil samples.The acid values were determined using the formular

$$\text{Acid Value} = \frac{\text{Titre Value x Malarity x Molar Mass}}{\text{Mass of Sample}}$$

Determination of iodine Value

A total of 0.2g of Cyperus esculentus oil was measured and placed in a dry flask for both mechanical and chemical treatments. The oil was then dissolved in 20ml of carbon tetrachloride and 25ml of Wiji Solution in each instance. After swirling the flask to mix the contents, it was left in a dark cupboard for 1 hour at room temperature. Once removed, 20ml of 15% potassium iodine was gradually added until there was no longer any color. At that point, 2ml of starch indicator was introduced, resulting in a blue color, which was eliminated by further slow additions of thiosulphate. This procedure was also repeated using the methyl esters of Cyperus esculentus. The iodine values were then calculated using the formula provided.

$$\text{Iodine value} = \frac{(\text{Titre – Bank}) \times 0.03175 \times 100}{0.2}$$

Determination of Saponification Value

A 2g oil sample from each *Cyperus esculentus* was transferred to a 250ml conical flask, followed by the addition of 25ml of a 0.5m ethanolic potassium hydroxide solution. The flask was equipped with a condenser and heated for one hour, with frequent swirling. While still hot, 1ml of phenolphthalein was introduced, and any excess alkali was titrated with 0.5M hydrochloric acid. A blank titration was conducted under identical conditions without the samples. The same procedure was also applied to the biodiesel samples of Cyperus esculentus. The saponification value was obtained by this formula:

$$\text{Saponification Value} = \frac{(\text{Blank – Tire}) \text{ x Molarity x Molar Mass}}{\text{Weight of Sample}}$$

Determination of % Impurity in Tiger Nut

10g tiger nut oil in already weighed 250ml conical flask was added to 100ml light petroleum ether, stirred vigorously and allowed to stand for 30minutes. Obtained solution filtered using Whatman paper and residue washed with 50ml ether to remove all oil. The filter paper with residue dried at 105^{O}C until constant weight. Amount of impurities present in the oil increases the weight of the filter paper. A percentage impurity is expressed as:

$$\% \text{ Impurity} = \frac{Wr - Wp}{Ws} \times 100$$

Wr = Weight of the residue

Wp = Weight of Paper

Ws = Weight of Sample

Determination of Specific Gravity by Density Bottle Method

In each case, the density bottle was washed, dried in an oven and the weight were taken 10ml of the oil sample were weighed into the bottle and the weight of the sample and the bottle were also taken and then calculations were done to determine the density of each oil.

$$\text{Specific gravity (density)} = \frac{\text{Mass of Sample}}{\text{Volume of the Sample}}$$

Determination of Flash Point

The oil sample was poured in an aluminum container and heated over a burner flame at the rate of 60 revolution per minute. A thermometer was placed inside the oil and at each 5 minutes temperature, a small flame was passed over the oil surface until a flash appears.

Determination of Fire Point

Each sample undergoing flash point determination was subjected to further heating after obtaining the flash point until the small flame is passed over the oil surface at 5 intervals produced continuous burning flame for a minimum

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of five(5) seconds. The fire point for each sample was then read on the thermometer and recorded. (JECRC, 2020; Awulu, 2018)

Determination of Cloud Point

Cloud point is determined by cooling a sample of the substance and visually observing the point at which the first haze or cloudiness appears. This temperature is typically reported in degrees Celsius (°C). The lower the cloud point, the better the low-temperature performance of the substance, as it indicates a reduced tendency to form solid particles or crystals. Each of the oil samples was introduced into separate test tubes, with thermometer inside and a cork cover and placed in the refrigerator so as to cool the oil samples. The test tubes were observed at intervals until it became cloudy. The temperature at which each oil sample becomes cloudy was recorded respectively as the cloud point.

Determination of Pour Point

The pour point test involves cooling a sample of the substance and tilting it periodically to determine if it can still flow. The temperature at which the substance stops flowing is recorded as the pour point. pour point is typically reported in degrees Celsius (°C). The oil sample was allowed to cool and the test tube were bended at an interval to observe flow of the oil till there is no flow of oil sample. The temperature of the oil sample was then observed and recorded.

Results

The results of the chemo- physical properties of extracted tigernut oil is presented in Table 1 below**.**

Table 1: Results of the functional properties of tigernut oil produced by two extraction methods

Oil Properties	Mechanical Method	Chemical Method	WHO Standard
Moisture (%)	0.394 ± 0.0021	0.226 ± 0.0077	0.05% - 0.4%
Fatty Acid (%)	0.408 ± 0.0098	0.311 ± 0.00056	0.6mgKOH/g
Impurity (%)	0.45 ± 0.0021	1.7025 ± 0.0106	0.3%
Saponification (mg/g)	206.765 ± 4.8598	203.745 ± 0.0770	194 – 265KOH/1g
Extraction Efficiency (%)	41.555 ± 0.0777	88.85 ± 0.3535	Nil
Refractive Index	1.416 ± 0.0028	1.718 ± 0.0056	1.465 – 1.469
Iodine (g/100g)	91.236 ± 0.0047	87.00 ± 0.4666	125 – 140/100g
Peroxide (mEq)	8.381 ± 0.0707	0.7635 ± 0.0120	10mEq
Specific gravity (g/cm^3)	0.9085 ± 0.0035	0.880 ± 0.0155	0.910 – 0.920g/cm
Flash Point (^{O}C)	149.845 ± 0.0212	156.355 ± 0.0777	145 – 150
Pour Point (^{O}C)	4.005 ± 0.0070	4.00 ± 0.0000	3.0 – 4.0
Cloud Point (^{O}C)	9.050 ± 0.0707	7.95 ± 1.3435	6 – 11
Colour	Brownish	Dark brown	Nil

The result shows the mean ± Standard deviation of the analysis.

Discussion

The Table 1 above shows the results of the Effects of Extraction Method of Tiger Nut oil extracted mechanically and chemically. The functional properties of the oil shows some variations. From the analysis, the colour of the extracted oil from the mechanical method was golden yellow while that of chemical method was Dark-brown.

Moisture Content: Oil extracted mechanically contained 0.394 ± 0.00219 and the oil extracted chemically contained 0.226 ± 0.0077. In these cases, since moisture enhances the bacterial growth of a substances, it means that oil from chemical method can be preserved for a short period of time compared to that of the chemical method. However, these are within the range of values (0.375% and 0.265% reported by Awulu et al. (2018) for mechanically and chemically produced oil respectively. The values for moisture in this work are higher than the permissible limit of WHO (2020) of (0.05 -0.4)% each of the oil would definitely require some level of de-moisturisation before storage.

Fatty Acids: Total fatty acid value was 0.4080 ± 0.0098 and 0.311 ± 0.0005 for mechanically and chemically extracted oil. This shows the total amount of free fatty acid in the oil. These values fall within the range of value reported by Hu et al. (2020) for oil obtained SE (0.21±0.00 – 75.06 ±0.12) and MUAAEE (0.07 ± 0.00 – 75.24 ±

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0.13) though higher than 0.06 + 0.00 -76.16 +0.12 reported by Hu et al. (2018) for oil extracted by MAE process. However the values are in accordance with the WHO standard (0.6mgKOH/100g). The level of fatty acid in the oil also determines the refractive index and iodine values of the oil.

The refractive index and iodine value of oils are related to fatty acid composition because high unsaturated fatty acids results in large refractive index. While high saturated fatty acids reduce the iodine value (Gu et al., 2017). They determined that tiger nut oil had a refractive index of 1.465–1.467.

Percentage impurity of the oils: The percentage impurity of the oil extracted mechanically and chemically were 0.0450 ± 0.0021 and 1.7025 ± 0.0106 respectiely. It shows that oil from chemical methods contains many much impurities than the oil from mechanical methods. This is due to the presence of the chemical (solvent) and other extracted components in the oil. Compare to 0.036% and 1.396% ,respectively, by Awulu et al, (2018). The mechanically extracted oil is lower while the chemically extracted oil is greater than the WHO (2020) standard (0.3%).

Saponification values were 206.765 ± 4.859 and 203.745 ± 0.7707 for the oil extracted mechanically and chemically. These means that the oils can be used for soap production perfectly. Though oil from the mechanical method shows the tendency of producing a better soap, chemical method oil can also produce a good soap. These values are higher than the values (287.25 ±1.42 and 187.52±1.23) for oils extracted via SE and MUAAEE, respectively (Huet al., 2020). Also higher than the 174.53±0.62, 175.33±1.61 and 176.71±0.81) obtained for oils extracted by ME, SC-CO_2 and SBE techniques respectively (Guo et al., 2021). However, both values are within the WHO permissible range of 194 – 265KOH/g. Furthermore, the saponification values were similar to the reported values (199.13–202.57) mg KOH/g. for all the oils extracted by Aljuhaimi et al. (2018).

The Extraction Efficiency : The results of the two oils were 41.555 ± 0.0770 and 88.85 ± 0.353 for the oil extracted mechanically and chemically. This means that chemical method of extraction yield more than mechanical method of the analysis. This is therefore encouraging for commercial purpose and profitability.

Iodine Content: 91.235 ± 0.0047 and 87.00 ± 0.466 were obtained for mechanically and chemically extracted oil. Since the number of grams of iodine taken up by the fat is high. Therefore, the oil is unsaturated. Iodine value is an important indicator of the degree of lipid unsaturation that enables the oil to be divided into non-drying oil (below 130gI_2/100g), semi- drying oil (100-130gI_2/100g) and drying oil above (130gI_2/100g). Therefore these oil extracted in this research are non-drying oil. However they are lower than the WHO permissible range (125 – 140/100g). But these extraction technique has produced oils with better Iodine values than (84.78, 85.41,and 66.16) reported for oil extracted via SE, MAE and SBE (Hu, et al., 2020; Hu et al., 2018; Guo et al., 2021) respectively. Aljuhaimi et al. (2018) reported an increasing changes in iodine values of their samples among 71.17–72.84 g/100 g,

Peroxide value was shown to be 8.38±0.0707 and 0.7635±0.012 for the oil extracted mechanically and chemically respectively. The peroxide value of the oil extracted mechanically 8.38±0.0707 was high which shows that higher rate of rancidity will be associated with the mechanically produced oil compared to 0.7635 mEq of the chemically extracted oil which is free of rancidity. The peroxide value can better reflect the degree of rancidity in the initial stage of oil during oxidation. The active peroxides are produced when oil is oxidized, leading to increased peroxide values (Azlan et al., 2010). The MAE and MUAAEE extracted oils showed low peroxide values (2.26±0.17 and2.35± 0.13), indicating that the oil obtained by those two methods contains less free fatty acids and have strong oxidative stability though not as strong as the oil produced chemically in this research. They are within the WHO range (10mEq).
The above results are in accordance with that reported by Zhang et al. (2022) where all their resuts demonstrated the superior storage stability of LPO and SEO.

Specific gravity: 0.908 ± 0.0035 and 0.880 ± 0.015 were obtained for mechanically and chemically extracted oil respectively. It shows that oil from mechanical extraction is denser than that of chemical extraction. The results and the values are within the WHO range (0.910 – 0.920g/cm).

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The refractive index values 1.416 ± 0.003 recorded for mechanical extraction is in accordance with the WHO permissible limit while 1.718±0.006 recorded for chemically extracted oils is higher than the WHO limit (1.465-1.469). however these values are also related to 1.465- 1.467 reported by Gu et al. (2017)

Flash Point: 147.845 ± 0.0212 and 156.355 ± 0.077 were obtained for mechanically and chemically extracted oil. This is within the range of values within the WHO range. The flash point point is the lowest temperature at which the oil gives momentary flash of light when a small flame is brought near it (Patni 2020). Therefore, chemically extracted oil is better with respect to flash point.

Pour points: The results obtained for pour points were 4.005 ± 0.007 and 4.00 ± 0.00 for mechanically and chemically extracted oils, respectively.The pour point is defined as the temperature at which a product becomes so viscous that it can no longer flow freely. It is a critical parameter, as it determines the lowest temperature at which the product can be pumped and used effectively. The pour point indicates the minimum temperature at which a substance can still flow or be poured under specific testing conditions. This measurement reflects the fluidity and mobility of a substance at low temperatures. The pour point is especially important for lubricating oils, greases, and other viscous materials (Mazui, 2020). In this study, the technique employed does not impact the pour point. Additionally, these values fall within the permissible limits set by the WHO. Table 1. Cloud Point values for mechanically and chemically extracted oils were 9.05^{O}C ± 0.0707 and 7.95 ± 1.343 respectively. The cloud point of a substance is the definitive temperature at which small solid particles or crystals initiate their formation as the substance cools. This parameter is vital for diesel fuels and other petroleum products, as it indicates the presence of waxes or solid contaminants that can cause fuel filter blockage and operational challenges in cold weather. In essence, the cloud point represents the temperature at which waxy substances in the product begin to generate a cloudy appearance, marking the onset of solidification.Thus, the chemical extraction process is more advisable in considering the cloud point. In summary, the cloud point is an early indication of potential issues (Kulkarni, 2021).

Conclusion

The oil extracted from both mechanical and chemical methods of extraction was pale brown and Dark-brown in Colour. The chemical method of oil extraction produced more oil with higher extraction efficiency, Higher impurity, flash point andreractve index. While all the other parameters were higher for the oil extracted mechanically Thus either of the oils has some advantages and disadvantages over the other

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