Journal of Sciences and Multidisciplinary Research
Volume 10, No. 2, 2018
ISSN: 2277-0135

CHARACTERIZATION OF TIGER NUT OIL EXTRACTED USING MECHANICAL AND CHEMICAL METHODS

[1]Awulu, J.O. [2]Omale, P.A. and [3]Omadachi, J.O.

Department of Agricultural and Environmental Engineering,
University of Agriculture Makurdi, Benue State
email:ayeslody4ll@gmail.com

Abstract: The mechanical and chemical methods of extraction of tiger nut oil were investigated using mechanical screw press and N-hexane respectively. Tiger nuts are used for food, medicine and industrial purposes and yet its oil energy properties have not been researched much, which was the reason for this research work. Tiger nut tubers sample was bought from Northbank market Makurdi, Benue state. The colour, iodine, specific gravity, free fatty acid, percentage impurity, moisture content, flash point, fire point, saponification value, peroxide value, extraction efficiency and extraction losses of the oil extracted were characterized. The colour of the extracted oil was dark brown yellow and golden yellow for mechanical and chemical methods respectively, Iodine value was determined using wij's method as 91.508(0.515) g/100g and 91.174(0.127) g/100g for mechanical and chemical method of extraction, specific gravity was determined by density bottle as 0.903(0.016) g/cm^3 and 0.892(0.003) g/cm^3 for mechanical and chemical method of extraction, moisture content was gotten by oven drying method as 0.375(0.045) % and 0.265(0.0586) % for mechanical and chemical method of extraction, free fatty acid was determined using titration method as 0.381(0.010) % and 2.148(0.067) % for mechanical and chemical method of extraction, flash and fire points were determined using flame test method 159(7.810) °C and 169.00(1.732) °C, 170(2.000) °C and 180(2.646) °C for mechanical and chemical method of extraction respectively; while cloud and pour point were determined by refrigeration method as 10.00(1.732) °C and 8.00(1.00) °C, 4.00(1.00) °C and 4.00(1.00) °C for mechanical and chemical method of extraction. Peroxide value was determined by titration method as 7.547(.560) Meq/g and 0.086(0.013) Meq/g for mechanical and chemical method of extraction. Percentage impurity was 0.036(0.004) % and 1.396(0.004) % for mechanical and chemical method of extraction and saponification was 209.91(2.321) mg/g and 209.33(8.168) mg/g for mechanical and chemical method of extraction, the extractillon losses was 4.250(0.303) % and

0.340(0.056) % for mechanical and chemical method of extraction. Result from mechanical and chemical methods showed that extraction efficiency was 77.5% (0.560) % and 87.5% (0.004) % respectively. Oil yield from mechanical and chemical methods using 100g of tiger nut tuber produced 12.25g equivalent to 0.014liters of oil and 17.5g equivalent to 0.02 liters of oil respectively. ANOVA results revealed that Free fatty acid, percentage impurity, peroxide value, extraction efficiency, extraction losses and fire point of tiger nut oil extracted using both the mechanical and chemical methods were all significantly different at $P<0.05$ while iodine value, specific gravity, moisture content, flash point, saponification value and cloud point of the oil extracted mechanically and chemically were not significantly different at p0.05.

Keywords: Tigernut, Extraction, oil, Mechanical, N-hexane

INTRODUCTION

Tiger nut (*Cyperus esculentus*) is an emergent grass- like plant that belongs to the sedge family found to be a cosmopolitan perennial crop of the same genus as the papyrus plant that is commonly grown in the tropical region (Bamishaiye and Bamishaiye, 2011). Tiger nuts have long been recognized by developed countries for their health benefits as they have a high content of soluble glucose and oleic acid, along with high energy content (starch, fats, sugars and proteins), they are rich in minerals such as phosphorous and potassium, calcium, magnesium and iron necessary for bones, tissue repair, muscles, blood stream and for body growth and development, and rich in vitamins E and C (Adejuyitan,ú 2011). In Nigeria tiger nut is available in fresh, semi-dried and dried form in the markets and are consumed by a lot of people without knowing the nutritional benefits and products that can be obtained from the tuber such as tiger nut flour, bread, cream, oil and milk among many others (Bamishaiye and Bamishaiye, 2011). 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 and Abodunrim, 2006). Mohammed et al, 2011 also stated that tiger nut milk called 'Kunnu-aya' by northern Nigerians is a healthy drink with nourishing and energetic power. Tiger nut oil has a golden color and a nutty aroma, which makes it ideal for different uses in cosmetic and domestic applications. It is used in deep frying because of its exceptional resistant to chemical decomposition at high temperature and its low absorption than other frying oils (El-Naggar, 2016). The edible and stable oil nature of tigernut oil has made it a competitor with olive,

corn; soybean, olive and cotton seed oil and can thus serve as a substitute for these oils especially in times of scarcity (Warra, 2013). The Tigernut oil remains in a uniform liquid form at refrigeration temperature which makes it suitable for salad preparation. Ezebor *et al.,* 2005 stated that tigernut oil has a high oleic acid, low polyunsaturated fatty acid and low acidity that have made it suitable for body cream. According to Shaker *et al., 2*009, tigernut oil is regarded as high quality oil due to its extraction without external heat, its resistant to chemical decomposition at high temperatures and its usage to waterproof textile fibers. The objective of this paper is to characterize the physio-chemical properties of tigernut oil extracted mechanically and chemically

METHODOLOGY

Tiger nut was purchased from North bank market in Makurdi local Government Area of Benue State. The tubers were sorted to remove the bad/rotten ones and dried properly under sun and stored in a polyethene bag which was being exposed to air and sunlight daily to avoid decay of tubers. Tubers was milled with a milling machine and the oil was extracted using a locally made mechanical press and oil was also extracted chemically by using N-hexane at Seraph refined oil company located in Makurdi. After extracting the oil using the two methods, the oil properties such as acid value, iodine value, specific gravity, moisture content, flash point, fire point, cloud point, pour point and saponification value were analyzed in the department of agricultural and environmental engineering food processing laboratory and tabulated respectively for each extraction methods.

Chemical Method of Extraction

This Practical works was carried out at Seraph Oil mills Makurdi. The sample (tiger nut) was grounded using blender. 100g of the powdered material of the sampled tiger nut tuber (***Cyperus esculentus)*** was weighed and transferred into an extraction thimble (column). The extraction thimble (column) was covered with cotton wool at the top and was closed. N-hexane was poured into the Extraction flask and the thimble was inserted in the extractor and all extraction set-ups were connected. The temperature of the extraction process was 50°C for 1 hour. After one hour the extraction set-up was disconnected and oil was recovered from the hexane and dried using the oven. After drying, the percentage of the oil determined from 100g of the sample used was 3.5g.

Mechanical Method of Oil Extraction from Tiger Nut

The tiger nut tubers sample was toasted (fried) using gas cooker for 3 minutes and crushed using Fish oil Extracting Machine at the Department of Agricultural and Environmental Engineering, Makurdi and tied in a transparent cloth and mounted on the mechanical press. Tiger nut oil was extracted by continual press of the two portions of the grounded tiger nuts (two placed side by side) on the locally made hydraulic laboratory press. Pressure was exerted till the oil started dropping and also at the limitation of the strength of the cloth sieve material used. The dried oil was then analyzed.

Analysis of Chemo-Physical Properties of Tiger Nut Oil

This was done for all the properties following the standard methods described by AOAC (1990 and 1998)

Determination of Free Fatty Acid Value

10g of *Cyperus esculentus* oil in each case (mechanical and chemical methods) was weighed into a 25ml conical flask. 50ml of a mixture of 50% ethanol and 50% N-hexane was added containing 6ml of phenolphthalein indicator. This was titration against 0.1Mpotassium hydroxide shaking constantly until a pink color, which persisted for 15 seconds was obtained. The same procedure was repeated for the methyl ester samples of *Cyperus esculentus* oil (mechanical and chemical methods).

The acid value was determined using equation 1.

Determination of Iodine Value (Bsi Method)

0.2g of ***Cyperus esculentus*** oil in each case was weighed out and placed in a dry flask. The oil was dissolved in 20ml of carbon tetrachloride and 25ml of Wij's solution in each case. The stopper flask was swirled to mix the contents and stood in a dark cupboard for 1 hour at normal Temperature (Approx. 77 25). It was removed and 20ml of a 15% potassium iodine solution was added followed by l00 ml of distilled water. The liberated iodine was slowly titrated with 0.1M thiosulphate solution until the yellow color almost disappeared. At this stage 2ml of starch indicator was added and the blue color that appeared was discharged by further slow additions of thiosulphate. The procedure was repeated for the methyl esters of ***Cyperus esculentus.***

The iodine value was obtained by using equation 2 below.
Where,
B = blank titration, S = Sample titration, M = Molarity of sodium thiousulphate solution, 12.69 = atomic weight of iodine7'.

Determination of Specific Gravity by Density Bottle Method

The formula (equation 3) below was used to determine the specific gravity of the tigernut oil extracted using both mechanical and chemical methods

Where,
M_1 is the mass of the density bottle including the stopper
M_2 is the mass of the density bottle + stopper + sample
M_3 is the mass of density bottle including stopper + distilled water

The procedure was repeated three times and the average of specific gravity was taken. The specific gravity of *Cyperus esculentus* oil as well as their methyl esters was determined.

Determination of Moisture Content

The already washed and cleared silica dishes was dried in an oven and cooled in desiccators. Five milliliters of each of the sample was kept into the appropriate silica dish and weighed accurately. Each dish and its contents were kept in an oven at a temperature of 105°C to dry for 1 hour. Each dish was transferred into desiccators and allowed to cool and was weighed. This was repeated until a constant weight was obtained.
The moisture content was calculated using equation 4.

Flash Point:

The oil sample was poured in an aluminum container and heated over a Bunsen burner flame at the rate of 10 per minute. A thermometer was placed inside the oil and at each 5increase in temperature a small flame was passed over the oil surface until a flash appears.

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 of 5 seconds. The fire point for each sample was then read on the thermometer and recorded.

Cloud Point:

Each of the oil samples was introduce 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.

Pour Point

The oil sample was allowed to cool and the test tubes were bended at an interval to observe flow of the oil till there is no flow of oil sample. The temperature of the oil samples was then observed and recorded.

Determination of Saponification Value

2g of the oil sample in each case of *Cyperus esculentus* was weighed and transferred into a 250ml conical flask followed by the addition of 25ml of 0.5M ethanolic solution of potassium hydroxide. There flux condenser was attached and the contents in the flask were heated for 1hour with frequent swirling. While the solution was added and still hot 1 ml of phenolphthalein was added and excess of alkali was titrated with 0.5M hydrochloric acid.

The blank titration was carried out under the same conditions without the samples being examined. The same procedure was repeated for the methylester (biodiesel) samples of *Cyperus esculentus*. The saponification value was obtained by this formula.

Determination of % Impurity in Tiger Nut Oil

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

Where,

W_r is the weight of dried residue +filter paper in (g)

W_p = weight of filter paper in (g)

W_s = weight of test sample in (g)

RESULTS AND DISCUSSION

Table 1 shows the replicated lab results on quality of Tigernut oil. The mechanical method of extraction showed that the colour of the oil was Dark brown yellow, moisture content value ranged from 0.35-0.43, free fatty acid value ranged from 0.37-0.39, percentage impurity value ranged from 0.03-0.04, saponification value ranged from 207.50-212.13, extraction efficiency value ranged from 76-79, pour point value ranged from 3.00-5.00, extraction losses value ranged from 3.90-4.43, specific gravity value ranged from 0.89-0.92, flash point value ranged from 154-168, cloud point value ranged from 8.00-11.00, iodine value ranged from 91.17-92.10, fire point value ranged from 168.00-172.00, peroxide value ranged from 6.97-8.06, While for the chemical method of extraction, the colour of the oil was found to be Golden yellow, moisture content value ranged from 0.21-0.33, free fatty acid value ranged from 2.07-2.21, percentage impurity value ranged from 1.39-1.40, saponification value ranged from 201.72-217.96, extraction efficiency value ranged from 86.50-89.30, pour point value ranged from 3.00-5.00, extraction losses value ranged from 0.28-0.39, specific gravity value was 0.89, flash point value ranged from 168.00-171.00, cloud point value ranged from 7.00-9.00, iodine value ranged from 91.10-91.32, fire point value ranged from 178.00-183.00, peroxide value ranged from 0.08-0.10.

Table 2 shows the mean and standard deviation of tigernut oil extracted using mechanical and chemical methods. The oil extracted mechanically, the colour was found to be dark brown yellow, moisture content value was 0.375 (0.045), free fatty acid value was 0.381 (0.010), percentage impurity value was 0.036 (0.004), saponification value was 209.910 (2.321), extraction efficiency value was 77.500 (1.500), pour point value was 4.00 (1.000), extraction losses value was 4.250 (0.303), specific gravity value was 0.903 (0.016), flash point value was 159.00 (7.810), cloud point value was 10.00 (1.732), iodine value was 91.508 (0.515), fire point value 170.00 (2.000), peroxide value was 7.547 (.560).

The oil extracted chemically, the colour was found to be golden yellow, moisture content value was 0.265 (0.0586), free fatty acid value was 2.148 (0.067), percentage impurity value was 1.396 (0.004), saponification value was 209.330 (8.168), extraction efficiency value was 87.500 (1.562), pour point value was 4.00 (1.000), extraction losses value was 0.340 (0.056), specific gravity value was 0.892 (0.003), flash point value was 169.00

(1.732), cloud point value was 8.00 (1.000), iodine value was 91.174 (0.127), fire point value 180.00 (2.646), peroxide value was 0.86 (0.013)

ANOVA revealed as shown on table 3 that free fatty acid, percentage impurity, fire point, peroxide value, extraction efficiency and extraction losses of the tigernut oil extracted using mechanical and chemical methods were all significantly different at $p<0.05$ for both mechanical and chemical methods while moisture content, saponification, pour point, specific gravity, flash point, cloud point and the iodine value of the tigernut oil extracted mechanically and chemically were not significantly different at $p>0.05$

Table 1: Lab Result of three (3) replication on quality of oil

	EXTRACTION METHODS					
	Mechanical Method Replication			Chemical Method Replication		
OIL PROPERTIES	A	B	C	A	B	C
Moisture (%)	0.35	0.35	0.43	0.21	0.26	0.33
Fatty Acid (%)	0.39	0.38	0.37	2.16	2.21	2.07
% Impurity	0.03	0.04	0.04	1.39	1.40	1.39
Saponification (mg/g)	210.10	207.50	212.13	208.31	201.72	217.96
Extraction Efficiency (%)	76.00	79.00	77.50	86.70	86.50	89.30
Pour Point (°C)	4.00	5.00	3.00	4.00	3.00	5.00
Extraction Losses (%)	3.90	4.42	4.43	0.28	0.35	0.39
Specific Gravity (g/cm^3)	0.89	0.92	0.89	0.89	0.89	0.89
Flash Point (°C)	154.00	155.00	168.00	168.00	168.00	171.00
Cloud Point (°C)	11.00	8.00	11.00	9.00	7.00	8.00
Iondine (g/100g)	91.26	91.17	92.10	91.10	91.32	91.10
Fire Point (°C)	168.00	170.00	172.00	178.00	179.00	183.00
Peroxide (Meg)	7.63	9.95	8.06	0.08	0.08	0.10
Colour	Dark Brown Yellow			Golden yellow		

Table 2: Means and Standard Deviation

OIL PROPERTIES	EXTRACTION METHODS	
	MECHANICAL METHOD	CHEMICAL METHOD
Moisture (%)	0.375[a] (0.045)	0.265[a] (0.0586)
Fatty Acid (%)	0.381[a] (0.010)	2.148[b] (0.067)
% Impurity	0.036[a] (0.004)	1.396[b] (0.004)
Saponification (mg/g)	209.91[a] (2.321)	209.33[a] (8.168)
Extraction Efficiency (%)	77.50[a] (1.500)	87.50[b] (1.562)
Pour Point (°C)	4.00[a] (1.000)	4.00[a] (1.000)
Extraction Losses (%)	4.250[a] (0.303)	0.340[b] (0.056)
Specific Gravity (g/cm^3)	0.903[a] (0.016)	0.892[a] (0.003)
Flash Point (°C)	159.00[a] (7.810)	169.00[a] (1.732)
Cloud Point (°C)	10.00[a] (1.732)	8.00[a] (1.000)
Iondine (g/100g)	91.508[a] (0.515)	91.174[a] (0.127)
Fire Point (°C)	170[a] (2.000)	180.00[b] (2.646)
Peroxide (Meg)	7.547[a] (0.560)	0.086[b] (0.013)

Different letters along the row shows different means according to Duncan new multiple range test ($p<0.05$) and Numbers in parentices are standard deviation

Table 3: ANOVA RESULT

Oil Properties		Sum of Squares	Df	Sig.
Iodine (g/100g)	Between Groups	0.167	1	0.337
	Within Groups	0.563	4	
	Total	0.730	5	
Specific Gravity (g/cm^3)	Between Groups	0.000	1	0.282
	Within Groups	0.000	4	
	Total	0.001	5	
Free Fatty Acid (%)	Between Groups	4.683	1	0.000*
	Within Groups	0.010	4	
	Total	4.693	5	

Percentage Impurity (%)	Between Groups	2.774	1	0.000*
	Within Groups	0.000	4	
	Total	2.774	5	
Moisture Content (%)	Between Groups	0.018	1	0.062
	Within Groups	0.011	4	
	Total	0.029	5	
Flash Point (°C)	Between Groups	150.000	1	0.096
	Within Groups	128.000	4	
	Total	278.000	5	
Fire Point (°C)	Between Groups	150.000	1	0.006*
	Within Groups	22.000	4	
	Total	172.000	5	
Cloud Point(°C)	Between Groups	6.000	1	0.158
	Within Groups	8.000	4	
	Total	14.000	5	
Pour Point (°C)	Between Groups	0.000	1	1.000
	Within Groups	4.000	4	
	Total	4.000	5	
Saponification Value (mg/g)	Between Groups	0.505	1	0.912
	Within Groups	144.202	4	
	Total	144.707	5	
Peroxide Value (Meg)	Between Groups	83.500	1	0.000*
	Within Groups	0.627	4	

	Total	84.127	5	
Extraction Efficiency (%)	Between Groups	150.000	1	0.001*
	Within Groups	9.380	4	
	Total	159.380	5	
Extraction Losses (%)	Between Groups	22.932	1	0.000*
	Within Groups	0.190	4	
	Total	23.122	5	

* represents significant difference at P<0.05

Percentage Oil Composition of Tiger Nut

Yield of oil extracted chemically (solvent extraction) was recorded as 0.003875 liters per 100g of sample while the oil extracted mechanically was 0.002713 liters of oil per 100g of sample. The percentage (%) composition of oil yield for chemical method was 17.5% and that of mechanical extraction yielded 15.5% which is smaller compared to that reported as oil yield from brown and yellow tigernut (Cyperus *esculentus)* tubers reported by Warra (2013) who gave the value of oil yield to be between 26.15±3.142% and 27.50±5.721% respectively. ANOVA table shows that oil composition has significant difference at P<0.05 therefore, chemical method is preferable to mechanical method for extraction of oil from tigernut.

CONCLUSION AND RECOMMENDATIONS

The oil extracted using both the mechanical and chemical methods were dark brown yellow and golden yellow respectively. Using the mechanical extraction method, 0.012 liters of tigernut oil was yielded from 100g of tiger nut sample while the chemical extraction method yielded 0.02 liters from 100g of tiger nut sample and this represents extraction efficiency of 15.5% and 17.5% for both mechanical and chemical extraction methods respectively. The moisture content of 0.375% and 0.265%, free fatty acid of 0.381 and 2.148, percentage impurity of 0.036 and 1.396, Sponification value of 209.910 and 209.330, extraction efficiency of 77.500 and 87.5, pour point of 4.00, extraction losses of 4.250 and 0.340,

specific gravity of 0.903 and 0.892, flash point of 159.00 and 169.00, cloud point of 10.00 and 8.00, iodine value of 91.508 and 91.174, fire point value of 170.00 and 180.00, peroxide value of 7.547 and 0.86 respectively.

ANOVA results revealed that Free fatty acid, percentage impurity, peroxide value, extraction efficiency, extraction losses and fire point of tiger nut oil extracted using both the mechanical and chemical methods were all significantly different at $P<0.05$ while iodine value, specific gravity, moisture content, flash point, saponification value and cloud point of the oil extracted mechanically and chemically were not significantly different at p0.05. The Chemo-physical properties of tiger nut oil characterized showed that tigernut oil is a good source of biodiesel.

REFERENCE

Adejuyitan J.A. (2011). Tigernut processing: Its Food uses and Health Benefits. American J. Food Tech. 6 (3): 197-201. DOI: 10.3923/ajft.2011.197.201

AOAC. (1990). Official Methods of Analysis, 14th edition Vol. 67. Association of Official Analytical Chemist, Arlington, VA, pp: 1-45.

AOAC. (1998). Official Methods of Analysis of the Association of Official Analytical Chemists, 16th Edn. Gaithersburg, USA.

Bamishaiye EI, Bamishaiye OM (2011). Tiger nut: as a plant, its derivatives and benefits. African J. Food,Agric, Nutritn Dev.11 (5): 5157 5170.

Belewu MA and OA Abodunrin, 2006; Preparation of Kunnu from unexploited rich food source: Tiger nut (Cyperus esculentus). World Journal of Dairy and Food Sciences, 1: 19-21.

Ezebor F, Igwe CC, Owolabi FAT and S.O Okoh (2005) Comparison of the physico-chemical characteristics, oxidative and hydrolytic stabilities of oil and fat of *cyperus esculentus* L. and butyrospermum parkii (shea nut) from Middle-Belt States of Nigeria. Nigerian Food Journal 2005; 23: 33-39.

Mohammed, N. O.; Bamishaiye, O. M.; Usman, L. A.; Nafiu, M. O.; Oloyede, O.B.(2011). Physico-chemical properties and fatty acid

composition of *Cyperus esculentus*(Tigernut) tuber oil. Bioresearch Bulletin. www.bioindicapress.com/brb

Shaker MA, Ahmed MG, Amany MB and LN Shereen (2009) Chufa Tubers (Cyperus esculentus L.): As a New Source of Food. World Appl. Sci. J., 2009; 2: 151-156.

Warra, A.A, (2013). Quality Characteristics of oil from Two Varieties of Cyperus esculentus L. Tubers. Journal of Botanical Sciences, Sci. Agric. 2(2), 2013: 42-45, E-ISSN: 2310-953X/P-ISSN: 2311-0228,

El-Naggar, E. (2016) A Physicochemical Characteristics of Tiger Nut Tuber (Cyperu esculentus Lam) Oil, Middle East Journal of Applied Sciences ISSN 2077-4613. Volume: 06, Issue: 04, 2016 Pages: 1003-1011

Reference to this paper should be made as follows: Awulu, J.O. Omale, P.A. and Omadachi, J.O. (2018), Characterization of Tiger Nut Oil Extracted using Mechanical and Chemical Methods. *J. of Sciences and Multidisciplinary Research,* Vol. 10, No. 2, Pp. 13-25