IJISET - International Journal of Innovative Science, Engineering & Technology, Vol. 6 Issue 6, June 2019
ISSN (Online) 2348 – 7968
www.ijiset.com

Extraction of Oil from African Walnut Seed (Tetracarpidium Conophorum)

OLOKO, S.A.

Department of Agiric. & Bio-Environmental Engineering,
The Federal Polytechnic, P.M.B. 5351, Ado-Ekiti, Ekiti State, Nigeria

ABSTRACT

The extraction of oil from African walnut was carried out and the Proximate analysis was conducted on the extracted oil to determine its physic o-chemical properties such as percentage of oil yield, free fatty acid, acid value, moisture content, saponification value, peroxide value, iodine value, refractive index value and the colour of the oil yield. Results showed the oil yield was 42.15% Free fatty acid was 0.71%; Acid value was 1.59 mg/KOH/g; moisture content was1.76%; Sponification value was 189.88 mg/KHO/g; Peroxide value was 9.46 mg/KOH/g; Iodine value was 8.10 g$I_2$100/g; Refractive Index value was 1.477 and the Colour of the oil was found to be Brownish yellow. All results were obtained at 100^{o}C. The results obtained from the proximate analysis of the oil shows that the oil extracted from African Walnut seed was compares favorably with the other oils and has oil potentials both as domestic oil and industrial oil. Also, the waste product can be converted to other by-products such as bio-fuel.

Keywords: Extraction, chemical properties, African Walnut Seed, Agricultural Products.

1.0 INTRODUCTION

African walnuts is a member of juglandance family. It is one of the finest nuts of the temperature regions. African walnut is an edible simple seeded stone fruits botanically called Tetracarpidiumconophoraum and coulaeduits. Its plant is mostly cultivated for its nuts which can be cooked or consumed as snacks. The nuts comprises of families such as

EuphorbiaceaGetracarpium (onophoram) and Olacaceae (Coulaeduits), with each family having its own specific feature (Naira Health).

The Euphorbiace is mostly found in Nigeria and Cameroon, while the olacacea is found in some Central African countries such as Congo, Liberia and Gabon. In the Western part of Nigeria, the African walnut is known as Awusa or Asala while in the Southern part it is called Ukpa. In Cameroon, it is known as ngak or kaso. The African walnuts are characterized by a black thick wall that envelopes a white cream seed. Its seed kernel is edible. When eaten raw they have a bitter flavor not unlike the kolanut and are considered to be tonic and aphronisiac. We consume bits of walnuts during the course of our everyday life in cakes, cookies, snacks etc. Asides appetite quenching, the African walnut is a highly versatile nut. It serves a number of other users as medicine and health uses, furniture uses, dye etc.

A walnut is the nut of any tree of the genus Juglens family Juglandanceal, particularly the Persian or English walnut, Juglensregia. Technically, a walnut is the seed of a drupe or drupaceous nut. It is used for food after been processed while green for pickled walnuts after full ripening for its nutmeat. Nutmeat of the eastern black walnut from the juglensnigra is less commercially available as are butternut nutmeats from juglarcinerea. The walnut is nutrient dense with protein and fatty acids.

Agricultural products are classified into oil seeds (cotton, castor, sunflower etc.) and mesocarps of fruits (oil palm) (Oyedele and Oladipo, 2014). Plants bearing these agricultural products have greatly contributed to the economic development of many countries especially the development of West African countries where the products are grown for commercial purposes. Out of many types of oil that can be obtained from these products, only few are very significant in terms of world production and traded as major commodities (Ibrahim and Onwualu, 2005).

It is an evergreen tree growing to a height of 25.38m and has a dense crown that can cost deep shade. The leaves are arranged alternatively simple, 10-30cm long and 4cm broad, with an entire margin and an acuminate apex.The nut is an ellipsoidal drupe available from August to January, 3 – 4cm long with fresh surrounding the kernel, 5 – 6mm thick, smooth in texture and can be red or green. The kernel shell is extremely hard and makes germination difficult. The nuts are usually found under the mother trees. Plate 1 below shos the fresh African Walnut fruit (unshelled).

PLATE 1:fresh African walnut fruit(Unshelled)

2.0 MATERIALS AND METHODS

2.1 Sample Source

The African Walnut fruits used in the present study were procured from the local market in Ado-Ekiti in Ado-Ekiti Local Government Area of Ekiti State, Nigeria. Samples were weighed on a weighing balance. The chemical properties such as protein, carbohydrate, fat, fibre and ash were analysed at the Science Technology Laboratory of the Federal Polytechnic, Ado-Ekiti, Ekiti State.

2.2 Methodology

The African walnut " AsalaOrAwusa" were bought from palace market"ojaoba"inAdo-Ekiti. The whole walnut bought was shelled manually by removing the kernel from the seed. Then the unmatured seed was removed and later sliced into small part at the range of 0.5mm thickness for easy drying. The sliced walnut were initially sun dried to remove moisture content from the fresh seeds. After sun drying the nut were sorted out by removing decayed or defective seed to ensure the quality of oil extracted as shown in Plate 2. Then the soxhlet extraction machine was set up for oil extraction as shown in Plate 3.

PLATE 2: Shelled Walnut Fruits

2.3 Experimental Procedure

The whole portion of the shelled kernel was sun dried for six hours and then milled to powder form using electric blender to allow easy oil extraction (Adebayo, et al, 2012). Solvent extraction method was used to extract oil from milled African Walnut using soxhlet extractor (Avalier model) (Plate 3).

200g of the milled sample was wrapped with filter paper. 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°C so that the solvent is brought to the evaporation point. 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. The residue solvent was allowed to evaporate. The extracted oil was re-heated to remove the n-hexane from the oil through evaporation. The test was carried out in the Laboratory of the Department of Science Technology, the Federal Polytechnic, Ado-Ekiti.

Plate 3:Soxhlet Extractor set up

2.4 **Proximate Analysis**

In evaluating the oil potential of the extracted oil, the reflective index, density, sponification value, Acid value, Free Fatty Acid (FFA), moisture content, peroxide value. Lodine value, colour and percentage oil-yield were determined using standard method given by (AO AC, 2012).

2.4.1 Refractive Index

Refractive Index, also called index of refraction, is a measure of the blending of a ray of light when passing from one medium into another (Oyedele and Oladipo, 2014). It can also be defined as a dimensionless number that describes how light or any other radiation propagates through the medium. In this research work, refrective 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.

2.4.2 Sponification Value

The sponification value of an oil or fat is the number of potassium hydroxide required to neutralize the fatty acid resulting from the complete hydrolysis of 1g of the sample (Ayo and Aguy, 2012). In determining the sponification value of the acid, 2g of the oil fat was weighed into a flask, 25ml pipette of containing alcoholic potassium hydroxide solution was poured into the conical flask containing the oil, and it was attached to reflux condenser and was heated on boiling water both for 1 hour with occasional shaking, after which 1ml of the phenolphthalein solution was added and was titrated while hot with the standard hydrochloric acid. This procedure was repeated for each of the five samples. Then the blank (that is potassium hydroxide solution without oil) was titrated using the same procedure. The sponification value was then determined using the relation in equation (1).

$$Sponification\ vlaue = \frac{(b-a)\ 28.05}{st.of\ sample} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where: a = titrate of the sample, b = titrate of the blank and w = weight of sample.

2.4.3 Acid Value

The acid value of an oil fat is the number of potassium hydroxide required to neutralize the free acids resulting from the complete hydrolysis of 1g of the sample (Ayo and Agu, 2012). In determining the acid value, 25ml diethyl ether with 25ml ethanol was mixed and 1.0ml of 1.0% phenolphthalein solution was neutralized and was titrated with 0.1ml sodium hydroxide solution then 1 – 10g of the oil in the neutralized solvent mixture was dissolved and was titrated with 0.1ml sodium hydroxide solution.

The acid value was therefore determined using equation (2).

$$Acid\ value = \frac{Titration\ (ml) x\ 5.61}{wt\ of\ sample\ used} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

2.4.4 **Peroxide Value**

The concentration of peroxides in oil give s an indication of the extent of spoilage. The oil was treated with potassium iodine in an organic solvent. The peroxide liberated the iodine from potassium iodine. The iodine was titrated with standard thiosulphate (Ayo and Agu, 2012).

In determining the peroxide value of the acid, the test was carried out in subdued daylight and 1g of oil was weighed into a clean dry boiling tube, 1g of powdered potassium iodine and 20ml solvent mixture was added to the oil that was inside the boiling tube then, it was placed in a boiling water bat for about 60 seconds. After which the contents of water was poured into it and the washing was added to the titration flask containing 20ml potassium iodine solution. The tube to be used was washed twice with 25ml portion of water and the washings were added to the titration flask. Finally it was titrated with 0.002ml thiosulphate, using starch as indicator. The peroxides value was therefore determined using equation (3).

$$Peroxide = \frac{2(a-b)}{w} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where: a = titrate value for the sample

b = titrate value for the blank

and w = weight of sample.

2.4.5 Moisture Content

Moisture content is the quantity of water contained in a material. This is based on loss on drying at an oven temperature of 105°C. Besides water, the loss will include the other matter volatile at 105°C (Ayo and Agu, 2012).

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 5g of the oil was introduced into the dish and was spread after wish it was weighed accurately (W_2). The dish and its content W_2 as transferred into an air oven at 105°C 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 wish 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. The moisture content was determined using equation (4).

$$Moisture\ Content = \frac{(W_1 - W_2)}{W_3 - W_1}\ x\ 100 \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (4)$$

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.

2.4.6 Iodine Value

The iodine value of an oil or fat is the weight of iodine absorbed by 100 parts by weight of the sample. This is also the measure of the degree of instauration of the fatty acids in the oil or fat. The oil or fat is treated with potassium iodide and titrating the liberated iodine with standard thiosulphate solution (Ayo and Agu, 2012).

In determining the iodine value of the oil about 0.2 – 0.5g of the oil was weighed into a glass Stoppard bottle of about 250 capacities. 10ml of carbon tetrachloride was added and dissolved. 20ml if W_{ij} solution was added and stopper was inserted which has been moisture with potassium. The solution as allowed standing in the dark for 30 minutes and 15ml potassium iodide solution was added with 100ml of water. The titrate was then mixed with the standard thiosulphate solution with starch as indicator just before the end point (titration = a ml).

The iodine value was determined using equation (5).

$$Iodine\ value = \frac{(b-a)\ 1.269}{Weight\ in\ g\ of\ sample} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (5)$$

2.4.7 **Relative density**

The relative density of an oil or fat at 20°C is the ratio of the mass in air of a given volume of the oil or fat at t° to that of the same volume of water at 20°C (Ayo and Agu, 2012).

In determining the relative density of the oil, the relative density bottle was cleaned, dried and weighed. The relative density bottle was then filled with distilled water and maintained in a bath at 20°C until the water inside reaches 20°C. The outside of the bottle was wiped and weighed. The bottle was emptied and dried, then filled with the oil and the bottle was kept in a bath at t°C. Finally, the outside of the bottle was wiped and weighed again. Relative density was determined using equation (6).

$$Relative\ density\ at\ t_{20}o_C = \frac{M_2}{M_1\ (1+\infty\ (t-20o_{C\ })} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (6)$$

Where: M_2 = Mass of oil or fat

M_1 = ass of water

∞ = Coefficient of cubical expansion of glass (0.0003 for soda glass; 0.00001 for borosilicate glass at 20°C or 40°C depending on the specification for the particular oil or fat).

3.0 RESULTS AND DISCUSSIONS

3.1 Results

The results obtained from the study are presented in Tables 1 and 2. Table 1 shows the values of the oil properties observed from the extracted oil from African walnuts at the level of 100°C drying temperature. The comparison of the extracted oil with other seed oil is presented in Table 2.

Table 1: Physico-chemical Properties of Extracted Oil from African Walnuts at 100°C temperature

Physico Chemical Properties	100°c Drying Temperature
Oil Yield (%)	42.15
Free Fatty Acid (%)	0.71 ± 0.02
Acid Value (mg/KOH/g)	1.59 ± 0.01
Moisture Content (%)	1.76
Sponification Value (mg/KOH/g)	189.88 ± 0.05
Peroxide Value (mg/KOH/g)	9.46 ± 0.08
Iodine Value (gI_2 100/g)	8.10 ± 0.06
Refractive Index Value	1.477 ± 0.03
Colour	Brownish yellow

* All values are mean and standard deviation of the samples. All values are at significantly different ($p \leq 0.05$)

Table 2: Physico-chemical Comparison of African Walnuts Oil with other Common Seeds Oil.

Physicochemical Properties	Experimental African Walnuts	Some Other Common Seed Oils												
		(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(b)	(c)	(c)
		Chest nuts	Sunflower	Soya bean	Corn Oil	Melon	Cotton Seed	Avocado	African Oil bean	Cucumeropsis	Andenopus Brevifiorus	Water Melon Seed Oil	Moringal Oil	Groundnut Oil
Oil Yield (%)	42.15	40.9	30.5	21.0	4.5	NA	22.9	NA	NA	NA	NA	41.32	NA	NA
Free Fatty Acid (%)	0.71	3.01	0.4	0.5	1.5	2.38	0.7	0.37	0.79	0.35	0.40	6.40	NA	NA
Acid Value (mg/KOH/g)	1.59	5.99	0.8	1.0	3.0	4.7	1.4	0.805	1.65	1.20	0.65	2.37	9.76	1.91
Moisture Content (%)	1.76	5.80	NA	Na	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sponification Value	189.88	193.2	191.	193.	190.	197.	195.	246.7	185.	185.	192.8	183.13	155.68	168.30
(mg/KOH/g)	9.46	1.50	0	0	6	6	0	NA	1	6	NA	NA	13.80	10.80
Peroxide value (mg/KHO/g)	8.10	NA	NA	Na	NA	NA	NA	NA	1.57	2.85	NA	121.51	35.85	59.64
Iodine Value (gI 100/g)	1.477	1.449	NA	Na	NA	NA	NA	1.462	NA	NA	1.462	1.47	NA	NA
Refractive Index Value (%)	Brownish	NA	1.47	1.47	1.46	1.47	1.47	NA	1.46	1.46	NA	Light	NA	NA
Colour	yellow		5	3	7	8	0		5	2		yellow		
			NA	NA	NA	NA	NA		NA	NA				

Source: Eswoso and ODELOKUN, (1995)

Note: NA: Value not available

a: Rahib et al, 2015

b: Duduyemi et al, 2013

c: Afolayan et al, 2014

3.2 Discussions

3.2.1 Oil Yield

Table 1 shows that the oil yield from African Walnuts is significantly (P≤ 0.05) (42.15%) at 100°C temperature,. Comparing the value with other oily seeds (Table 2) showed that the oil content of African Walnut is higher than chestnuts (40.9), sunflower (30.5); soya beans (21.0); cotton seed (22.9) and water melon (41.32) as reported by Rahib et al; (2015) and Duduyemi et al; (2013). This shows that African walnuts can as well produce similar quantity like chestnuts and water melon seed oil. It also indicates that processing of African walnuts oil for industrial or edible purpose would be economical.

3.2.2 Acid Value and Free Fatty Acid

Free fatty acids can stimulate oxidative deterioration of oils by enzymatic and or chemical oxidation to form off flavor components (Rahib et al; 2015). Free fatty acid value is an indication of lipase activity (Ukhun, 1986). Fatty acid (%) has been calculated based on the molecular weight of the dominant acid. Free fatty acid indicates the possible hydrolytic degradation of the oil and the acid value is employed to ascertain the quality (condition) and edibility of the oils. Free fatty acid from African walnuts has its significantly (p≤0.05) lower value (0.71%) at drying temperature of 100°C water bath. Comparing the value with other seeds oil (Table 2), the free fatty acid from African walnuts has lower value as that of sunflower (0.4%); soya bean (0.5%) cotton seeds (0.7%); Avocado (0.37%); Cucumeropsis (0.35%) and AndenopusBreviflourus (0.4%) while free fatty acid from chestnuts (3.01%); corn oil (1.5%) and melon (2.38%) are higher than that of African walnuts. The implication of this is that African walnuts having lower value of free fatty acid (0.71%) is potentially edible. African walnuts with

low free fatty acid value will have a high smoke point since free fatty acid is also related to smoke point, so it would be suitable for stir fry cooking (Akintayo and Bayer, 2002).

3.2.3 Sponification Value

Sponification value of African Walnuts oil (Table 1) was found to be ($p \leq 0.05$) 189.88 mg/KOH/g at the drying temperature of 100°C. In Table 2, the sponification value of African Walnuts is lower than chestnuts 193.2 mg/KOH/g; Sunflower 191.0 mg/KOH/g; Soya beans 193.0 mg/KHO/g; corn oil 190.6 mg/KOH/g; melon 197.6 mg/KOH/g; Cotton seed 195.0 mg/KOH/g; 246.7 mg/KOH/g; AndenopusBrevilorus 192.8 mg/KOH/g; but higher than that of African Oil bean 185.1 mg/KOH/g; Cucumeropsis 185.1 mg/KOH/g; Cucumerpsis 185.6 mg/KOH/g; Water Melon 183.13 mg/KOH/g; Moringa Oil 155.68 mg/KOH/g; and Groundnut Oil 168.30 mg/KOH/g. A sponificiation value of 200 mg/KOH/g indicates high proportion of fatty acids of low molecular weight and chain length (Rahib et al; 2015). This shows that oil may not have a potential for use in soap making and in cosmetics industries and or in the thermal stabilization of Poly Vingl Chloride (PVC). This property makes them useful as sources of essential fatty acids required in the body (Akanni et al; 2005).

3.2.4 Peroxide Value

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 and it indicate the level of deterioration (Adebayo et al; 2012; Mohammed and Hamsa, 2008). The peroxide value of African Walnuts oil has significantly $P \leq 0.05$) value of 9.46 mg/KOH/g at 100°C. The peroxide value of African Walnuts is higher than chestnuts 1.50 mg/KOH/g; African oil bean 1.57 mg/KOH/g and cucumeropsis 2.85 mg/KOG/g but lower than Moringa oil 13.80 mg/KOH/g and Groundnut 10.80 mg/KOH/g as reported by Rahib et al; (2015) and Afolayan et al; (2014). The peroxide

value of African Walnuts (9.46 mg/KOH/g) is also within the maximum acceptable value of 10 mg/KOH/g set by the Codex Alimentarius Commission (CAC, 1999) for such oils as African Walnuts Oils.

3.2.5 Refractive Index

The recorded refractive index (RI) of African Walnuts oil at water bath temperature (100°C) was at significantly $P \leq 0.05$. The value of RI obtained from African Walnuts oil was similar to those of chest nuts 1.3449%; Sunflower 1.475; Soya bean 1.473%; Corn oil 1.467%; Melon 1.478%; Cotton seed 1.470% Avocado 1.462%; African Oil bean 1.465%; Cucumeropsis 1.462; AndenopusBreviflorus 1.462% and Water melon 1.47%. The higher values of the properties obtained for the crude oils revealed the necessity to purify the oils. The high refractive index of oil also showed that the fatty acids in the oil will contain a high number of carbon atoms (Bello and Olawore, 2012).

3.2.6 Moisture Content

The moisture content from the study has significance ($P \leq 0.05$). The possible value of 1.75% was found at 100°C drying temperature (Table 1). The moisture level of African Walnuts oil was lower than the chestnut 5.8% (Esuoso and Odetokun, 1995), Lower moisture content implies good shelf life characteristics of oil (Oguniyi, 2006).

3.2.7 Iodine Value

The iodine value is a measure of the degree of unsaturation of the fatty acids in an oil and could be used to quantify the amount of double bonds present in the oil which reflects the susceptibility of oil to oxidation (Afolayan et al. 2014). The iodine value for African Walnuts oil is the lowest (8.10 gI 100/g) (Table 1) compared to other oils in Table 2 and this reflected the presence of low percentage of unsaturated fatty acids in the African walnuts oil while Water

melon (121.51); Moringa (35.85) and Groundnut oil (59.64) has the highest iodine value and this reflected the presence of high percentage of unsaturated fatty acids as indicated in Table 2. The iodine values generally indicate that African Walnuts, Moringa and Groundnut oils have low degree of unsaturation and according to Pearson, (1981) for most edible oil, they are classified as non-drying oils(80 – 100 g/100g).

3.2.8 Relative density

The relative density of an oil or fat at 20°C is the ratio of the mass in air of a given volume of the oil or fat at t° to that of the same volume of water at 20°C (Ayo and Agu, 2012).

In determining the relative density of the oil, the relative density bottle was cleaned, dried and weighed. The relative density bottle was then filled with distilled water and maintained in a bath at 20°C until the water inside reaches 20°C. The outside of the bottle was wiped and weighed. The bottle was emptied and dried, then filled with the oil and the bottle was kept in a bath at t°C. Finally, the outside of the bottle was wiped and weighed again. Relative density was determined using equation (6).

$$Relative\ density\ at\ t_{20}o_C = \frac{M_2}{M_1\ (1+ \infty\ (t-20o_C)} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ (6)$$

Where: M_2 = Mass of oil or fat

M_1 = ass of water

∞ = Coefficient of cubical expansion of glass (0.0003 for soda glass; 0.00001 for borosilicate glass at 20°C or 40°C depending on the specification for the particular oil or fat).

4.0 CONCLUSIONS AND RECOMMENDATIONS

4.1 Conclusions

African Walnuts oil has an oil yield of 42.15% by comparing with other oil crop. Beside this other properties such as free fatty acid v alue of 0.71%; acid value of 1.50 mg/KOH/g; sponification value of 189.88 mg/KOH/g; peroxide value of 9.46 mg/KOH/g; moisture content of 1.76% and Refractive Index value of 1.447% and Iodine value of 8.10 gI_2100/g was found appropriate. The colour and odour (Brownish colour) is similar to other common oils such as groundnut oil, melon oil vegetable oil and soyabean and attractive for cooking. It is concluded from the results obtained that:

(i) The oil is edible

(ii) The oil yield indicates that it can be produced in commercial quantity.

(iii) The moisture content obtained was higher which implies good shelf life of the oil and

(iv) The odour of the oil is pleasant which implies that it will be good for cooking.

(v) The Engineering application of the research is that the waste products from the Walnut seeds can be converted into other by – products such as bio-fuel.

4.2 Recommendation

I. It was recommended that the walnut oil can be produced in large quantity.

II. It was also suggested that more research should be carried out on the pH level of the fruit.

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