



# Transesterification and Epoxidation of Oil Extracts from Selected Plants for Use as Bio-transformer Oil

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## Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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## ABSTRACT

Transesterification and Epoxidation of oils extracted from seeds of five different plants, namely, Jatropha, Moringa, Castor, Cotton and Shea was carried out using standard methods and characterized using American Society for Testing Material (ASTM) standard tests. The oils were tested on both low and high- medium voltage transformer and the result showed that their dielectric strength ranges from 25 – 43 KV, while the free fatty acids were found to be within the range of 0.01 – 0.008 mg KOH. These results are within the range of ASTM standards and that the oil samples have potentials to serve as bio-transformer oils.

*Keywords: Bio-transformer oil; soxhlet extractor; Jatropha; Moringa; Shea; ASTM standard.*

## 1. INTRODUCTION

The need to slow or reverse global warming and the attendant climate change is now widely accepted as a global affair. This requires reduction of greenhouse gas (GHG) emissions,

especially carbon dioxide (CO<sub>2</sub>). Using cultivated plants for energy needs instead of fossil mineral oil and coal generally reduces the net addition of CO<sub>2</sub> to the atmosphere. In addition, biofuels produce fewer particulates, hydrocarbons, nitrogen oxides and sulphur dioxides than

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mineral oils and therefore reduces the amount of pollutants that are harmful to human health. Transformer oil or insulating oil is usually a highly-refined mineral oil that is stable at high temperatures and has excellent electrical insulating properties [1]. Bio-transformer oil is bio-dielectric fluid or natural ester oil derived from biomass resources for use in electric transformers [2].

However, since biofuel production competes with food production, caution must be applied in the choice of biofuel feedstock to ensure food security. Although it has been reported that bio-based transformer oil has been produced from natural esters, the existing literature mostly reported the conversion of soya vegetable oil to transformer oil mainly in the United States [3]. Attempts have been made with sunflower oil but considered too costly, while coconut oil was also found to have possessed the necessary electrical, physical and chemical properties of bio-transformer oil [4]. In Nigeria, successful research works on bio transformer oil were carried out using soya and palm oils [5,6].

However, all of the above mentioned vegetable oils (especially soya and palm oils) are not only edible, but formed part of staple foods of most Nigerians. Besides, while the United States may have been producing excess soya seeds above their consumption needs, and, thus, uses the extra seeds for biofuels production, the same could not be applicable to Nigeria. Therefore, there could be conflicts with food sources if these vegetable oils and their likes are adopted as major feed stocks for biofuel production in Nigeria.

In contrast, the vegetable oils analyzed in this research work ranges from non-edible (*Jatropha* and Cotton seeds) to less edible (*Moringa*, Castor and Shea seeds). Surely, none of the seeds could be clearly classified as staple food item in Nigeria. Although no literature on their use as bio-transformer oil could be traced, several literatures reported successful production of biodiesel fuel from all of them, in addition to other traditional utilization of the oils [7,8,9,10,11].

Transformer oil or insulating oil is usually a highly-refined mineral oil that is stable at high temperatures and has excellent electrical insulating properties. It is used in oil-filled transformers, some types of high voltage capacitors, fluorescent lamp ballasts, and some types of high voltage switches and circuit

breakers. It forms a very significant part of the transformer insulation system and has the important function of acting as an electrical insulator. The aim of this paper is to report the viability of the five (5) selected oil producing plants in Nigeria to produce bio-transformer oil or dielectric fluid for electric transformers.

## 2. MATERIALS AND METHODS

All solvents and reagents used were of analytical grade. Distilled water was used as solvent for solution preparation and all glassware were washed, cleaned and dried.

### 2.1 Sample Collection and Preparation

*Jatropha curcas* seeds were sourced from the National Research Institute for Chemical Technology (NARICT), Zaria. The seeds were cleaned, dehulled, and dried. The dried seeds were then crushed into fine particles of 1.0 mm. [12]. *Moringa* seed pods were sourced from Kawo market, Kaduna. Both Pods and shells were removed manually and the seed kernels were grounded to 1.0 mm particle sizes [13]. Shea seed were sourced from Kurmi market, Kano. The seeds were cleaned, dehulled, dried and then crushed into fine particles of 1.0 mm [9]. Cotton seeds were sourced from Kawo market in Kaduna. The samples were properly cleaned and oven dried in the laboratory at a temperature of 130 °C, to a moisture content of 12% and grounded to 1.0 mm particle sizes. Castor seed were sourced from the National Seed Development Council, Abuja and crushed in to fine particles of 1.0 mm for the extraction process [14].

### 2.2 Oil Extraction

Oils were extracted from the powdered seeds with the aid of Soxhlet extractor using n-hexane as solvent and cold press method. The extracted oils were collected and purified for characterization.

### 2.3 De-Gumming

Extracted oils were de-gummed by washing down the fat with warm distilled water followed by removal of any water present with MgSO<sub>4</sub> [15].

### 2.4 Epoxidation and Transesterification

Measured 50 g each of extracted oil samples were first epoxidized using H<sub>2</sub>O<sub>2</sub> (30% aq) in the

presence of Oxone followed by transesterification reaction using the epoxidized product and methanol in the presence of sodium hydroxide as catalyst to synthesize the corresponding epoxy methyl ester [5]. A measured 0.20 g of finely ground anhydrous NaOH was added into 20 cm<sup>3</sup> of pure methanol in a 125 cm<sup>3</sup> Erlenmeyer flask which was swirled until the NaOH is dissolved making sodium methoxide. The methoxide solution was mixed with the oil sample at 45 – 50°C for 1.5 hours using a magnetic stirrer while heating lightly to avoid the evaporation of methanol. The mixture was then placed in to separatory funnels and was gently shaken continually till the cloudy mixture eventually separated into two layers. The funnel was left to stand for 24 hours after which the lower layer (glycerol) was drained while the top layer of epoxy methyl ester was washed with distilled water and characterized for transformer oil properties [5].

## 2.5 Characterisation of Oils

### 2.5.1 Acid value

Measured 25 cm<sup>3</sup> of ether was mixed with 25 cm<sup>3</sup> of ethanol in a 250 cm<sup>3</sup> beaker. The mixture was poured into 10 g of oil sample in a conical flask and a few drops of phenolphthalein added. The mixture was titrated with 0.1 mol dm<sup>-3</sup> NaOH to a dark pink colour end point with consistent shaking. The volume of 0.1 mol dm<sup>-3</sup> NaOH (V) was noted and used to calculate the free fatty acid as follows:

$$\text{Free fatty acid (FFA)} = Vo/Wo = 82-100$$

Where: 100 cm<sup>3</sup> of 0.1 mol dm<sup>-3</sup> NaOH= 2.83 g of oleic acid; Wo = sample weight; Acid value = FFA-2 [6].

### 2.5.2 Transformer testing

The transformer testing of the oil samples was carried out at the Kano Electricity Distribution Company (KEDCO) transformer workshop located at Dakata, Kano using a Foster Transformer (Laboratory type II) manufactured in the United Kingdom with model no: SW19 and serial no: 91ZA925. The test was carried out by placing 200 cm<sup>3</sup> of the oil samples in a constant fixed volume beaker and placed in a receptacle on the transformer. Current was then passed through oil sample and monitored till the circuit breaker goes off and flow of current stopped. The

voltage, at which the circuit breaker goes off called the breakdown voltage, is noted and recorded [6]. The process was repeated twice for each sample and average result is taken as presented in Table 1.

### 2.5.3 Specific gravity ASTM [16]

The specific gravity bottle was cleaned and dried in an oven at 60°C. The weight of the empty bottle was taken and recorded, after which the bottle was filled with the oil sample and properly covered, then, the weight was taken and recorded. The sample was removed and the bottle properly washed and filled with distilled water, after which the weight was also taken. The specific gravity was computed using the relationship below:

$$\text{Specific gravity} = \left( \frac{W_o - W}{W_o - W} \right)$$

Where, W = weight of empty bottle, Wo = weight of the bottle and oil content, W<sub>1</sub> = Weight of bottle and water content.

### 2.5.4 Viscosity (ASTM D445, 2013) [16]

Viscosity of the fluids was determined using a Cannon- Fenske 1972 – B50. 50 cm<sup>3</sup> was carefully introduced into the reservoir to avoid gas bubbles. The viscometer was placed in a water bath to allow measurements to be obtained at different temperatures between 20°C and 60°C [5].

$$\text{Viscosity (V)} = c \times t$$

Where

V = Viscosity in (mm<sup>2</sup>/s or cSt)

T = Time in seconds

C = Viscosity tube constant

### 2.5.5 Saponification value (ISO 1988) [17]

Measured 2 g of the oil sample was weighed into a conical flask to which 25 cm<sup>3</sup> of 0.1 mol dm<sup>-3</sup> ethanoic potassium hydroxide was added, stirred and allowed to boil gently for 60 min. A reflux condenser was placed on the flask containing the mixture and a few drops of phenolphthalein indicator was added to the warm solution, titrated with 0.5 mol dm<sup>-3</sup> HCl to the disappearance of the pink colour of the indicator. The same procedure was repeated for other samples and a blank. The expression for saponification value (SV) is given by:

$$SV = 56.1 N (V_o - V_i)/m$$

Where:

- $V_o$  – the volume of the solution used for the blank test,
- $V_i$  – the volume of the solution used for determination,
- $N$  – Actual normality of HCl used  $m$ – mass of the sample.

### **2.5.6 Peroxide value ASTM (2013) [16]**

Measured 1 g of potassium iodide and 20 cm<sup>3</sup> of solvent mixture (glacial acetic acid/chloroform, 2/1 by volume) was added to 1 g of the oil sample, and the mixture boiled for one minute, the hot solution was then poured into a flask containing 20 cm<sup>3</sup> of 5% potassium iodide, few drops of starch solution was then added to the mixture and the latter titrated with 0.025 N sodium thiosulphate and the peroxide value was determined as follows:

$$PV = \frac{S N 10^3}{W}$$

Where:  $S = \text{cm}^3$  of  $\text{Na}_2\text{S}_2\text{O}_3$ ,  $N$  = normality of  $\text{Na}_2\text{S}_2\text{O}_3$ ,  $W$  = Weight of oil sample (g).

### **2.5.7 Iodine value (ISO 1989) [17]**

Measured 0.4 g of the sample was weighed into a conical flask and 20 cm<sup>3</sup> of carbon tetrachloride was added to dissolve the oil. Then 25 cm<sup>3</sup> of Dam reagent was added to the flask using a safety pipette influenced chamber. A stopper was placed and the content of the flask vigorously swirled before the flask was placed in the dark for 2 h and 30 min. At the end of this period, 20 cm<sup>3</sup> of 10% aqueous potassium iodide and 125 cm<sup>3</sup> of water were added and the mixture titrated with 0.1 mol dm<sup>-3</sup> sodium-thiosulphate solution until the yellow colour almost disappeared. A few drops of 1% starch indicator was then added and the titration continue by adding thiosulphate drop-wise until blue coloration disappeared after vigorous shaking. The same procedure was used for the blank test and for other samples. The iodine value ( $IV$ ) is given by the expression:

$$IV = 12.69c (V_1 - V_2)/m$$

Where:  $c$  – concentration of sodium thiosulphate used,  $V_1$  – volume of sodium thiosulphate used for the blank,  $V_2$  – volume of sodium

thiosulphate used for determination,  $m$  – mass of the sample.

### **2.5.8 Flash point**

The test was carried out in the laboratory using the Pensky-Marten flash point tester (Closed Cup Method) and a 0 – 400°C thermometer. 5 cm<sup>3</sup> of oil samples was placed in the cup of the flash point tester and then placed in the barrel of the tester. A thermometer was inserted into the cup to monitor temperature at intervals of 10°C as the oil was heated in the cup while the fumes being released from the oil was tested with a lighted match stick to determine the temperature at which the fumes will support the flame for about 2 seconds which is the flash point [6].

### **2.5.9 Pour point and cloud point**

The test was carried out in the laboratory using an ice bath, test tubes and a negative thermometer. The test tubes containing 5 cm<sup>3</sup> each of the oil samples were stuck in the ice bath and checked periodically (at intervals of 3°C) for flow characteristics, the negative thermometer was used to check for temperature and temperature at which oil sample starts to coagulate which was determined as pour point, while the temperature at which wax first becomes visible was the cloud point [6].

### **2.5.10 Density**

The test was carried out by weighing oil samples in fixed volume density bottles that have a fixed volume and the density was derived from calculation of mass per unit volume [6].

### **2.5.11 pH**

The pH of the oil samples was determined by inserting an electronic pH meter in to 5 cm<sup>3</sup> of oil samples in a beaker for 1 minute [6].

### **2.5.12 Sulphur content**

5 cm<sup>3</sup> of the sample was placed in a ceramic crucible and put in a high frequency induction furnace and ignited to form  $\text{SO}_2$ , which was then measured by infrared detector (ISO 17025, 2013) [17].

## **3. RESULTS AND DISCUSSION**

Results of the oil characterization analysis are given in Table 1.

The values of densities of all the plant seed oils assessed varied from  $0.52 \pm 0.40 \text{ g/cm}^3$  for Moringa oil to  $0.91 \pm 0.20 \text{ g/cm}^3$  for Shea oil. Higher density means more mass of fuel per unit volume. The density of Moringa oil was lower than the ASTM standard, while that of Shea oil was higher. All other three oil samples (Jatropha, Cotton and Castor) have densities within the standard limits ( $0.55 - 0.89 \text{ g/cm}^3$ ) set by ASTM, [16], which suggest that with some refinement, they could all serve as bio-transformer oil.

Jatropha, Castor and Moringa oils with viscosities of  $16.50 \pm 0.12$ ;  $11.80 \pm 0.02$ ;  $18.10 \pm 0.12 \text{ mm}^2/\text{s}$  have promising prospects as potential transformer oils when compared with the standard value  $9.3 - 27 \text{ mm}^2/\text{s}$  (ASTM, [16]). The lower the viscosity of the oil, the easier it is to pump and atomize and achieve finer results. Viscosity is considered as one the most important property of transformer oil since it affects the operation of fuel injection equipment, particularly at low temperature when an increase in viscosity affects the fluidity of the fuel. However, Shea oil has viscosity of  $39.98 \pm 0.50$  as obtained by [9] meaning that unless severally further refined, it would be extremely difficult for

Shea oil to be used as transformer oil for high voltage transformers.

The flash points obtained from this study,  $180.60 \pm 0.50^\circ\text{C}$ ,  $231.00 \pm 2.50^\circ\text{C}$  and  $201 \pm 2.50^\circ\text{C}$  for Moringa, Shea and Cotton seed oils respectively were greater than the limits of  $140 - 155^\circ\text{C}$  set by ASTM, [16], while the flash point of  $146 \pm 2.50^\circ\text{C}$  for Jatropha seed oil is found to be within the standard limits set by ASTM, [16]. This shows that they can all be safe of ignition and fire hazard at high temperature especially during transportation and storage. Generally, oils with flash points above  $66^\circ\text{C}$  are considered safe oils [15].

Pour points obtained from this study show that among the plant seed oils assessed, Shea oil has the highest pour point of  $8.00 \pm 0.30^\circ\text{C}$ , while the lowest pour point of  $1.15 \pm 0.10^\circ\text{C}$  was recorded for Castor seed oil. Pour point is the lowest temperature at which the oil can flow. Although, the pour points of all the seed oils evaluated were found to be higher than the standard limits of  $-8 - (-6)$  set by ASTM, [16]; they are still low enough to work even in cold climes.

**Table 1. Quality parameters of extracted oils with the ASTM standard**

Property	Jatropha	Moringa	Castor	Shea	Cotton	ASTM 2013
Density at $29.5^\circ\text{C}$ ( $\text{g/cm}^3$ )	$0.72 \pm 0.50$	$0.52 \pm 0.40$	$0.74 \pm 0.30$	$0.91 \pm 0.20$	$0.85 \pm 0.4.0$	$0.55 - 0.89$
Viscosity at $27^\circ\text{C}$ ( $\text{mm}^2/\text{s}$ )	$16.50 \pm 0.12$	$11.80 \pm 0.02$	$18.10 \pm 0.12$	$39.98 \pm 0.50$	$29.40 \pm 0.12$	$9.3 - 27$
Flash point ( $^\circ\text{C}$ )	$146 \pm 2.50$	$180.6 \pm 0.50$	$132.0 \pm 1.5$	$231.0 \pm 2.50$	$201 \pm 2.50$	$140 - 155$
Acid number (mg KOH/ g oil)	$3.45 \pm 0.12$	$4.47 \pm 0.12$	$2.12 \pm 0.08$	$3.12 \pm 0.45$	$3.72 \pm 0.51$	$0.01 - 0.03$
Transformer dielectric strength (KV)	$43.00 \pm 0.02$	$40.00 \pm 0.02$	$41.01 \pm 0.01$	$25.00 \pm 0.90$	$38.03 \pm 0.01$	$25 - 40$
Pour point ( $^\circ\text{C}$ )	$4.0 \pm 0.01$	$2.0 \pm 0.15$	$1.15 \pm 0.10$	$8.0 \pm 0.30$	$6.5 \pm 0.60$	$-8 - (-6)$
pH	$5.6 \pm 0.20$	$5.7 \pm 0.13$	$6.0 \pm 0.75$	$5.50 \pm 0.10$	$5.40 \pm 0.12$	$5.5 - 8.2$
Specific gravity at $20^\circ\text{C}$	$0.83 \pm 0.12$	$0.82 \pm 0.13$	$0.85 \pm 0.15$	$0.94 \pm 0.12$	$0.84 \pm 0.04$	$0.89 - 0.91$
Saponification value (mg KOH/ g oil)	$258.21 \pm 5.30$	$232.51 \pm 0.43$	$268.31 \pm 0.20$	$261.3 \pm 6.70$	$243.51 \pm 2.15$	$150 - 244$
Iodine value (g/100 g oil)	$103.3 \pm 0.25$	$97.1 \pm 0.51$	$9.31 \pm 0.20$	$10.15 \pm 0.20$	$9.81 \pm 0.72$	$55 - 120$
Sulphur Content (wt%)	$10.5 \pm 0.30$	$8.0 \pm 0.50$	$8.12 \pm 0.50$	$11.0 \pm 0.80$	$10.50 \pm 0.20$	$7 - 15$
Free fatty acid (mg KOH)	$0.071 \pm 0.80$	$0.067 \pm 0.50$	$0.081 \pm 0.10$	$0.074 \pm 0.20$	$5.75 \pm 0.50$	$0.01 - 0.08$

The iodine values for Jatropha oil and Moringa oil ( $103.30 \pm 0.25$  g/100 g;  $97.11 \pm 0.51$  g/100 g) were found to be within the ASTM range (ASTM, [16]) which is beneficial in the sense that the lower the unsaturation of oils and fats, the greater will be its oxidation stability. While those for Castor oil:  $9.31 \pm 0.20$  g/100 g; Shea oil:  $10.15 \pm 0.20$  g/100 g and Cotton oil:  $9.81 \pm 0.72$  g/100 g analysed in this study were found to be below the standard range of 55 – 120 g/100 g set by ASTM, [16] for transformer oil.

The higher the iodine value, the greater the degree of unsaturation, thus the more fluid is the oil. Hence, in terms of iodine value, Jatropha and Moringa seed oils in this study have their values found to be within the range of transformer oil, and could therefore serve as bio-transformer oil.

Saponification values ( $232.51 \pm 0.43$  mg KOH/g and  $243.51 \pm 2.15$  mg KOH/g) of Moringa and Cotton seed oils respectively were within the range of 150 – 244 mg KOH/g set by ASTM, [16] for transformer oil. Saponification values ( $258.21 \pm 5.30$  mg KOH/g,  $268.31 \pm 0.20$  mg KOH/g and  $261.30 \pm 6.70$  mg KOH/g) of Jatropha, Castor and Shea seed oils respectively were above the standard values set by ASTM, (2013) and therefore requires further refining to reduce the values because oils with high saponification value cause exhaust emissions during burning in the engine.

At a laboratory conducted transformer testing, Jatropha oil sample was found to have the highest value of dielectric strength value of  $43.00 \pm 0.02$  KV; Castor oil, 41 kV; Moringa oil,  $40.00 \pm 0.02$  KV; Cotton oil,  $38.03 \pm 0.01$  KV and Shea oil,  $25.00 \pm 0.90$  KV. These are all within and above the standard 25 – 40 kV (ASTM [16]) but only Jatropha, Castor and Moringa oils values are within the range of the mineral oil currently being used in Nigeria which is 40 – 50 kV. The breakdown voltage improves upon refinement and purification through which the level of moisture content drastically reduces [5].

Meanwhile, the breakdown voltages of the three oil samples (Jatropha, Castor and Moringa) makes them appropriate for use in both high and low voltage transformers; while Cotton and Shea oils could be used in low-to-medium voltage transformers whose minimum dielectric breakdown voltage is within 20-39 kV.

Transformers are virtually everywhere and almost every transmission and distribution

transformer is filled with relatively large amount of oil. For instance, pole mounted transformers contain about 25 gallons, or almost 95 liters of fluid while substation transformers use between 10,000 and 20,000 gallons [3].

In Nigeria, the cost of mineral transformer oil currently stood at an average of N 800 (about USD \$2) per litre. Considering the high cost and the attendant environmental impacts of mineral oil, vis-a-vis the environmental benefits of bio-transformer oil earlier outlined, the advantage of bio-transformer oil is clearly evident.

It will noted however, that as every renewable energy source, the initial investment cost for the production of bio transformer oil could be very high considering the land needed for cultivation, the farming activities, oil production and refining technology as well as marketing of the product. Therefore, financing sources such as the clean development Mechanism (CDM) could be exploited to midwife the initial production.

#### 4. CONCLUSION

The analysed oils have great potential of being used as bio-transformer oils in both high and low voltage transformers especially when they are further refined. The research work and its outcome have shown the need for further exploitation of renewable energy resources in Nigeria to serve as alternative to fossil fuels and save the environment from continuous degradation.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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