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Characterization and Transesterification of Allanblackia Flouribunda Oil Seed for synthesis of Biodiesel

Balogun, T. A.[1], Woyengibunugha Ere[2]

[1]Department of Chemical Engineering, University of Delta, Agbor; P.M.B 2090, Delta State.
[2]Department of Agricultural and Environmental Engineering, Niger Delta University, Wilberforce Island, Bayelsa State
*Corresponding Author Email: papatriplet@gmail.com

ABSTRACT

The oil was extracted from the seed of Nigerian Allanblackia floribunda using solvent extraction method. The extracted oil was characterized and used for synthesis of biodiesel through Akali-based transesterification process. Properties of the produced biodiesel were carried out. Physicochemical properties as well as fatty acid profile of the oil extracted were measured and the results obtained showed that the oil is edible, non-drying and saturated in nature and could serve as feedstock for many industrial purposes. The viscosity of oil was 30mm²/sec⁻¹, density 914kg/m³, smoke point 210ºC, freeze point 270ºC, cloud point 25ºC and refractive index of 1.45Nd_{60}. Transesterification was carried at a temperature of 60ºC for 80 minutes with 6:1 mole ratio of the methanol to the oil and 1gram of desired base as heterogeneous catalyst. Results obtained from physicochemical properties of the produced biodiesel were compared with ASTM and EN standards the values compared well. The fuel properties of the biodiesel produced not the standard range and yield of fatty acid methyl ester (FAME) obtained was 96.8[1]%. The study supports the synthesis of biodiesel from Allalnblackia Floribunda seed oil as alternative to the fossil fuel.

Key words: Akali based Transesterification, Biodiesel derived based catalyst, Allanblackia floribunda seed oil, methyl ester.

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1. INTRODUCTION

The vast majority of plants, especially the agricultural stock, contain extractable oil that maybe of some commercial value. Since the beginning of human civilization rural communities from around the globe have used various traditional methods to extract mainly edible oil materials of plant origin. Many edible vegetable oils such as palm, corn, soybean, peanut, coconut, etc, are used as table oils because of their high nutritive value (Kinita and Badmos, 2019) for instance, fats and oils are the most concentrated form of energy, providing approximately 9 kcal of energy per gram compared to only 4 kcal per gram for proteins and carbohydrates (Gomez *et al.,* 2021). This is in addition to their industrial application as raw materials for synthesis of polyols, resins, biodiesel, pharmaceuticals etc, with regard to industrial application, focus ought to be more on non-edible oils like castor and jatropha oils, while preserving the edible ones for human consumption.

Current global vegetable oil consumption-n stands at around 182 million metric tons (Kojima *et al.,* 2018). The rising demand for vegetable oils both for human consumption and industrial application has prompted and encouraged the evolution and optimization of procedures leading to efficient production of oil of high quality and purity (Gomez *et al.,* 2021). Key issues arising from the exploitation of oleaginous materials of plant origin are: how much oil do they contain; how much of this oil can be extracted or recovered (% oil yield or % oil recovery), and what is the purity and quality of the extracted oils? With the exception of palm and olive oils which are extracted from their fruits (Kojima *et al.,* 2018), most vegetable oils are extracted from oilseeds. Consequently, seed oils have continued to attract enormous attention as potential source of platform chemicals at both laboratory and industrial scale (Adepoju *et al.,* 2018a).

2. MATERIALS AND METHODS

2.1 Materials

Matured seeds of Allanblackia floribunda were obtained from the Oyigbo LGA, Rivers State Sustainable Development Agency (RSSDA). Other materials used for the study include: Ethanol (96%), Petroleum ether and n-hexane, Petroleum ether and formic acid, a measuring cylinder, ethanolic phosphomolybdic acid, 1.18 mm of standard sieve using a Disc miller, 100 mL Schott bottle, ice-cubes, 0.5 ml p-anisaldehyde, Soxhlet apparatus, weighing machine, oven, incubator, linen cloth-bags, a thermostatically controlled oven, bottle-basedgravity bottle, AOCS (American Oil Chemist Society), Abbe refractometer (model: Bellingham + Stanlay limited, 60/70), bomb calorimeter, extraction chamber, supercritical fluid extraction set-up, microwave assisted extraction set-up, ultrasound assisted extraction set-up, ACAMAG High performance HLn Layer Chromatography (HPTLC) system, a toluene-ethyl acetate (85:15, v/v) as a mobile phase for the High-performance HLn Layer Chr5omatography (HPTLC) system, a spraying device, an air fan, a HPTLC imaging device (TLC Visualizer CAMAG), a MATALAB and an SPSS (Statistical Package for Social Sciences) Analytical software.

2.2 Catalyst preparation and characterization

Residual wood ash was obtained from oven furnace of bakery, and t6he ash was separated from unwanted impurities by using 0.3mm by mesh sieve. The fine residual ash wood powder (RAWP) was sent for catalyst characterization to examine it compositions.

The wood ash powder characterization was based on surface morphology via scanning electron microscopy (SEM), model 6300F by JEOL solution for innovation, USA, operated at a dimensional resolution of 50 μm, The elemental composition of the catalyst was (XRD) (model NEX QC series, manufactured by Rikagu, USA), and was used to determine the angular scanning of electron. The XRD was allowed to perform in the range of $20^o<20<80^o$ at the speed of 2 min^{-1} with Kă and Cu radiation source, accelerated at 10mA and 40 kV. The functional group and absorption band present in the catalyst was observed by Fourier transform infrared spectroscopy (FTIR) (Model STS-1900FT, made by Scientific Technology Solutions, India) at a resolution of *, range from 650-4000. Furthermore, the Bet surface area, the basicity, the basic site density were obtained using BET isothermal adsorption and Hammett indicator method.

2.21 Biodiesel Production

The transesterification reaction of seed oil was carried out in 500 ml capacity three necked reactor flask placed on a hot plate with magnetic stirrer. The reactor was charged with a known heating time for oil pre-heating at a temperature of 140ºF, the stirrer was kept to rotate at a speed of 600 rpm. After the pre-heating has been completed, a known weight of catalyst residual ash powder was dissolved in a known volume of anhydrous methanol and was quickly transferred into preheated oil on the hot plate with magnetic stirrer and two layers was noticed. The reactor was covered by the stopper to prevent methanol escaping as the reaction proceeded.

The hot plate was then adjusted to the desired temperature for complete reaction at a particular time.

At the end of the experiment the resulting mixture was then transformed to a separating funnel for glycerol and methyl ester separation, the glycerol was allowed to separate by gravity for 24h. Two phases separated clearly, the less dense methyl ester at top and denser phase of glycerol at bottom. The glycerol was then tapped off the bottom of the separating funnel leaving behind methyl ester in the separating funnel and the catalyst in the glycerol was recycled. Since the purity level of the biodiesel has strong effects on its fuel properties , therefore the methyl ester left in the funnel was then washed with (three times the volume of the biodiesel in the separating funnel) distilled water boiled to a temperature of 50ºC to remove residual catalyst, glycerol and methanol and soap. The water being denser than the methyl ester settled at the bottom of the funnel and was tapped off slowly. The washing process was carried out up to four times until the water becomes clear. The washed methyl ester left in the separating funnel was then tapped off into pyrex flask where it was further dried over heated calcium chloride powder (usually 3 g) placed in heating mantle to absorb the untapped water. The dried methyl ester obtained was decanted into a clean pyrex flask to remove the hygroscopic calcium chloride sediment at the bottom of the heated flask. The final product was the methyl ester known as biodiesel and yield was calculated as:

$$\textit{Experimental yield \% (w/w)} = \frac{\textit{Weight of oil biodiesel produced}}{\textit{Weight of oil sample}} \times 100 \qquad (3.1)$$

2.3 Determination of physicochemical properties of biodiesel

The determination of the biodiesels properties are stated below:

(a) Acid value *(AOAC, 1990)

The acid value was determined by dissolving 5g of oil sample in a hot mixture of 25ml (95% v/v) diethyl ether and 25 ml ethanol in 250 ml flask, the hot solution was neutralized with 0.1 M potassium hydroxide solution using three drops of phenolphthalein as an indicator. The acid value was calculated.

Acid value (mg KOH/g oil) = 2 X % *FFA* (3.2)

(b) %FFA – Free Fatty Acid (AOAC, 1990)

The % FFA was obtained by the Eqn. 3.3

$$\% \text{ FFA} = \frac{\text{V X N X 282}}{\text{W}} \text{ X 100} \quad (3.3)$$

Where V = volume of KOH used during titration, N = Normality of KOH, and W = Weight of oil sample.

(c) Peroxide Value (AOAC, 1990)

Oil sample of 2g was weighed into a 250 ml Pyrex flask; 40 ml of the solvent mixture (2:1 glacial acetic/chloroform) and 2 g KI powder were added. The mixture was boiled briskly in a water bath for 1 min at a temperature of 70ºC. To the flask containing 40 ml of 5% KI, boiled mixture was added; the resulting mixture was washed twice with 50 ml of distilled water into flask. The content of the flask was then titrated with 0.004 M sodium thiosulphate ($NA_2S_2O_3$) solution by using starch as indicator. The peroxide value was calculated as indicated in Eqn 3.4.

$$\text{Peroxide Value (meq}O_2\text{/kgoil)} = \frac{\text{Volume of } NA_2S_2O_3 \text{ x Normality of } NA_2S_2O_3}{\text{Weight of the oil sample in Kg}} \quad (3.4)$$

(d) Saponificationn Value (AOAC, 1984)

Oil sample of 2g was weighed into a 250 ml Pyrex flask with 25 ml of 0.1 M ethanoic potassium hydroxide which added. The content was constantly stirred and was allowed to boil gently for 60 min with a reflux condenser placed on the flask containing the mixture to achieve uniform temperature. Two drops of phenolphthalein indicator was added to the warmed soap solution and then titrated with 0.5 M HCL to end point until the pink color of the indicator just disappeared. The saponification value was calculated

$$s.v \text{ (mg KOH/g oil)} = 28.05 \text{ X } \frac{(\text{Vol. of HCL required by blank} - \text{Vol. of 0.5N HCL})}{\text{Weight of sample in g}} \quad (3.5)$$

(e) Iodine Value (Wij's Method)

Oil sample of 0.26 g was dissolved in 10 ml of cyclohexane. 20 ml of Wij's solution was added, the stopper flask was allowed to stand for 30 min in the dark at room temperature, and

20 ml of 10% potassium iodide solution was added. The resulting mixture was then titrated with 0.1 M $NA_2S_2O_3$ using starch as indicator. Iodine value was calculated using Eqn. 3.6.

$$Iodine\ Value = \frac{[B\text{-}S]\ X\ N\ X\ 12.69}{\text{Weight of oil sample}} \quad (3.6)$$

Where N = Concentration of sodium thiosulphate used; B = Volume of sodium thiosulphate used for blanck; S = Volume of sodium thiosulphate used for determination.

(f) Moisture Content (AOAC, 1990)

Oil sample of 5 g was carefully weighed into a moisture dish of 5 cm diameter and 2 cm deep with tight-fit-slip-over cover. This was placed inside a vacuum oven operated at temperature above boiling point of water (125ºC) at working pressure of 95 mmHg for 30 min intervals by noting down the weight of dried sample until a constant weight was attained when there is no additional loss of 0.05%. The moisture content was calculated using Eqn. 3.7.

$$\%\ Moisture\ Content = \frac{Weight\ of\ the\ sample - Weight\ of\ dried\ sample}{Weight\ of\ the\ sample} \quad (3.7)$$

(g) Specific gravity (pearson, 1976)

The specific gravity of the oil sample was measured using the specific gravity bottle. The bottle was full with water and the weight was noted. After a while, the bottle was also full with oil sample and the weight was recorded also. The specific gravity was then calculated using Eqn. 3.8.

$$\text{Specific Gravity} = \frac{\text{Weight of water obtained}}{\text{Weight of oil measured}} \quad (3.8)$$

(h) Cetane Number –- (ASTM D2015)

This was calculated using Eqn. 3.9.

$$Cetane\ No = 46.3 + \frac{5458}{Saponification\ Value} - 0.225\ Iodine\ Value \quad (3.9)$$

(i) Higher Heating Value (HHV) – (ASTM D2015)

This was calculated using Eqn. 3.10.

$$HHV\ (MJ/kg) = 49.43 - [0.041(Saponification\ value) + 0.015(\text{Iodine value})] \quad (3.10)$$

$$Mean\ molecular\ saponification\ value = \frac{56}{Saponification\ value}\ X\ 1000 \quad (3.11)$$

(j) Viscosity (AOAC, 1990)

A viscometer glass tube which was held in vertical direction was used. The sample of the Sorrel oil or biodiesel was drawn into capillary tube via suction to a marked volume. The sample was then allowed to flow down into the lower bulb and the time taken to pass through the marks was noted. The kinematic viscosity was calculated using Eqn. 3.12.

Kinematic Viscosity = time taken X Viscometer factor (3.12)

(k) API

This was determined by the equation cited by Haldar *et al.*(2009) as described in Eqn. 3.13.

$$API = \frac{141.5}{Specific\ gravity\ @\ 15^{o}C} - 131.5 \quad (3.13)$$

(l) Diesel index

This was determined by the equation cited by Haldar *et al.* (2009) as described in Eqn. 3.14

$$Diesel\ Index = \frac{Cetane\ number - 10}{0.72} \quad (3.14)$$

(m) Determination of flash point

Determination of flash point was done ASTM D93 flash-point by pensky-martens closed cup tester. Sample of the biodiesel was heated in a close vessel and ignited. When the sample burns, the temperature is recorded; the pensky-martens cup tester measures the lowest temperature which application of the test flame causes the vapor above the sample to ignite. The biodiesel is placed in a cup in such quantity as to just touch the prescribed mark on the interior of the cup. The cover is then fitted onto the position on the cup and Bunsen burner is used to supply heat to the apparatus at a rate of about 5ºC per minute. During heating, the oil is constantly stirred. As the oil approaches its flashing, the injector burner is lighted and injected into the oil container after every 10 second interval until a distinct flash is observed within the container. The temperature at which the flash is then recorded, it is repeated two times and the average taken.

(n) Determination of Pour Point

Pour point was determined by continuing the immersion of the beaker containing the biodiesel into the ice block after cloud point determination. The flow of the biodiesel was checked at 5s intervals and the point when the congealed biodiesel stops flowing and formed an immovable solid was carefully observed.

2.4 Wood Ash Powder Characterization

The wood ash powder characterization was characterized based on surface morphology via scanning electron microscopy (SEM), model 6300F by JEOL solution for innovation, USA, operated at a dimensional resolution of 50µm x 179µm. The elemental composition of the catalyst was ascertained by using energy dispersive spectroscope (EDS), while X-ray diffraction analysis (XRD) (model NEX QC Series, manufactured by Rigaku, USA), was used to determine the angular scanning of electron. The XRD was allowed to perform in the range of $20^{o}<20<80^{o}$ at a speed of 2 min^{-1} with Kă and Cu radiation source, accelerated at 10 mA an 40kV. The functional group and absorption band present in the catalyst was observed by Fourier transform infrared spectroscopy (FTIR) (model STS- 1900FT, made by scientific Technology Solutions, India) at a resolution of 8, range from 650-4000. Furthermore, the BET surface area, the basicity, the site density were obtained using BET isothermal adsorption and Hammett indicator method.

i. Process flow design diagram of oil extraction to biodiesel production

The flow design diagram indicated the production of biodiesel from the seed grinding to the end of production.

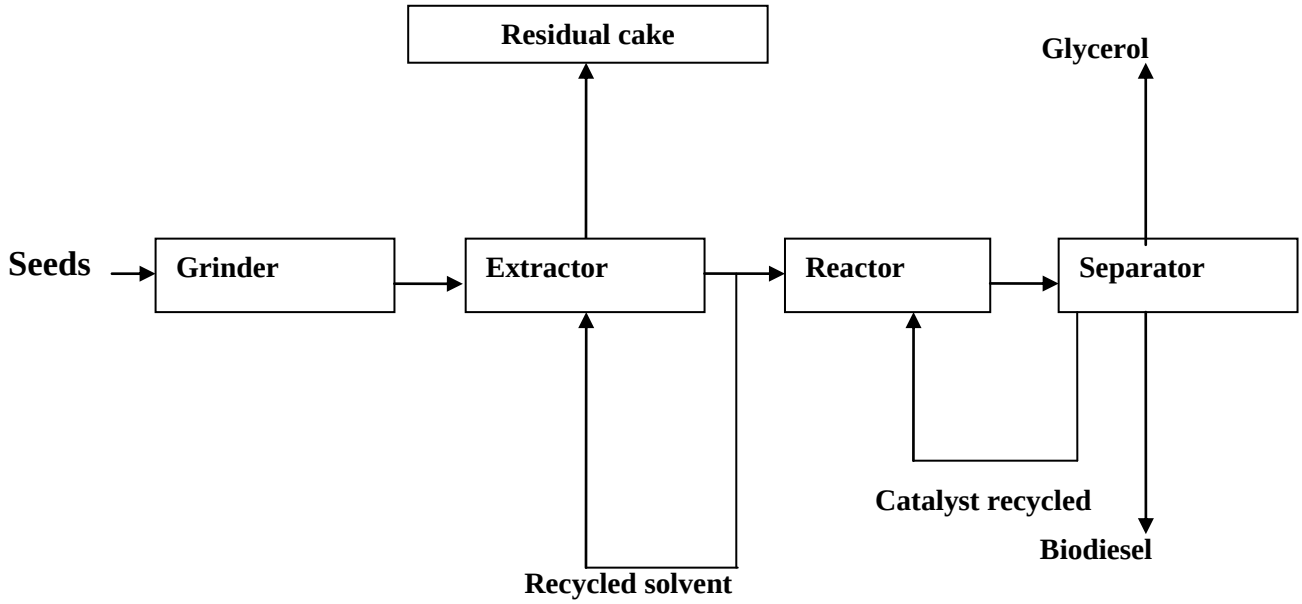


Figure 1; Flow design diagram of oil extraction to biodiesel production

Transesterification of Oil Allanblackia Floribunda Biodiesel Production

The based catalyzed reaction was carried out according to the work of (Betiku *et al.*, 2012). The transesterification reaction of Allanblackia floribunda seed oil was carried out in a 500 ml capacity spherical flat bottom flask placed on a hot plate with magnetic stirrer. The flask was charged with a known weight of the oil sample at a known heating time for oil pre-heating at a temperature of 140ºF, the stirrer was kept to rotate at a speed of 600 rpm. After the heating has been completed, a known weight of wood ash powder used as catalyst was dissolved in a known volume of anhydrous methanol and was quickly transferred into a preheated oil on the hot plate with magnetic stirrer and two layers was noticed. The flask was covered by the stopper to prevent methanol escaping as reaction proceeded. The hot plate was then adjusted to the desired temperature for complete reaction at a particular time.

At the end of the experiment the resulting mixture was then transferred to a separating funnel for glycerol and methyl ester separation, the glycerol was allowed to separate by gravity for 24 h. two phases separated clearly, the less dense methyl ester at top and denser phase of glycerol at bottom. The glycerol was then tapped off the bottom of the separating funnel leaving behind methyl ester in separating funnel. Since the purity level of the biodiesel has strong effects on its fuel properties, therefore the methyl ester left in the funnel was then washed with (three times the volume of biodiesel in the separating funnel) distilled water boiled to a temperature of 50ºc to remove residual catalyst, glycerol, methanol and soap. The water being denser than the methyl ester settled at the bottom of the funnel and was tapped off slowly. The washing process was carried out up to four times until the water becomes clear. The washed methyl ester left in the separating funnel was then tapped off into a pyrex flask where it was further dried over heated calcium chloride powder (usually 3 g) placed in heating mantle to absorb untapped water.

The dried methyl ester obtained was decanted into a clean pyrex flask to remove the hygroscopic calcium chloride sediment at the bottom of the heated flask. The final product was the *Allanblackia floribundas* methyl ester known as diesel and the yield was calculated in terms of % (w/w) as described in Eqn 3.1.

$$\textit{Experimental yield \% (w/w)} = \frac{\textit{Weight of oil biodiesel produced}}{\textit{Weight of oil sample}} \times 100$$

Table 1: Factors of their level for Box-Behnken Design

(a) Extraction

Variables	**Units**	**Symbols**	**Levels**		
			-1	**0**	**+1**
Extraction time	**Min**	**X_1**	**40**	**50**	**60**
Extraction vol.	**MI**	**X_2**	**200**	**250**	**300**
Sample weight	**G**	**X_3**	**50**	**55**	**60**

(b) *Transesterification*

Source: Box-Behnken design

Table 2: Biodiesel experimental designs

Variables	**Units**	**Symbols**	**Levels**		
			-1	0	+1
Reaction time	(min)	X_1	60	70	80
Cat. Amount	(wt.)	X_2	3	4	5
M-OH/oil molar ratio	(% v/v)	X_3	3	4	5

Source: Biodiesel experimental design

3. RESULTS AND DISCUSSION

3.1 PHYSICOCHEMICAL PROPERTIES OF ALLANBLACKIA FLORIBUNDA

The physicochemical properties of Allanblackia oil are presented in table 3 below.

The properties of the extracted oil using the three solvents, petroleum ether, ethanol and n-Hexane compared to the Chinese Tallow Tree Seed Oil (Table 4.2 and 4.3 respectively). The physicochemical properties of the Nigerian Tallow seed oil were measured and the results obtained in Table 4.2showed that the oil extracted using ethanol has iodine value of 39g/100g melting point of 42.2ºC, densit66y of 914kg/m^3, peroxide value of 8.6mEg/Kg, saponification of 185.7mg, moisture content of 0.16% and free fatty acid of 0.25% respectively. The specific gravity, refractive index, free fatty acid, acid value, peroxide value, iodine value, saponification value, smoke point, flash point, viscosity of the three solvents used for extraction of the samples were not significantly different ($p>0.05$).

Table 3: physicochemical Properties of Nigerian Allanblackia Seed Oil

Oil	Properties	Soxhlet Extractor Solvent
Allanblackia Oil	Color	
	State at ambient temperature	
	Acid value (mg koH/g)	
	Iodine value (g/100g)	
	Saponification (mgkoH/g)	
	Viscosity (mm^2/sec^{-1}) @60ºC	
	Peroxide value (meq/kg)	
	Density (kg/m^3) @ 60ºC	
	Refractive index @ 60ºC	
	Specific gravity @60ºC	
	Flash point (ºC)	
	Slip melting point (ºC)	
	Cloud point ºC	
	Smoke point ºC	
	Moisture content %	
	Free fatty acid %	

3.2 Catalyst characterization

BET Analysis of Catalyst

The result of BET analysis carried out based on data reduction acquisition employing the DA method with nitrogen as adsorbate for sample weight of 0.12g, revealed the plot of pore volume against pore diameter in Figure 4.3. It was observed that the highest pore volume was recorded at 0.321 (cc/g) at corresponding value of 2.820 nm pore diameter. It was noticed that higher pore diameter produced low volume which explained why the reaction process was faster to synthesize biodiesel. Further analysis by BJ adsorption method based on liquid density with data reduction parameter evaluation showed a plot of cumulative pore volume, surface area against the pore diameter (Figure. 4.4). The overall maximum values that described the catalyst potentiality for biodiesel production were found at surface area of 441.368 m^2/g, pore volume of 0.213 cc/g, and pore diameter of 2.136 nm. The pore size distribution data based on pore with, cumulative pore volume and surface area, dv/d, and ds/d, are as presented in APPENDIX

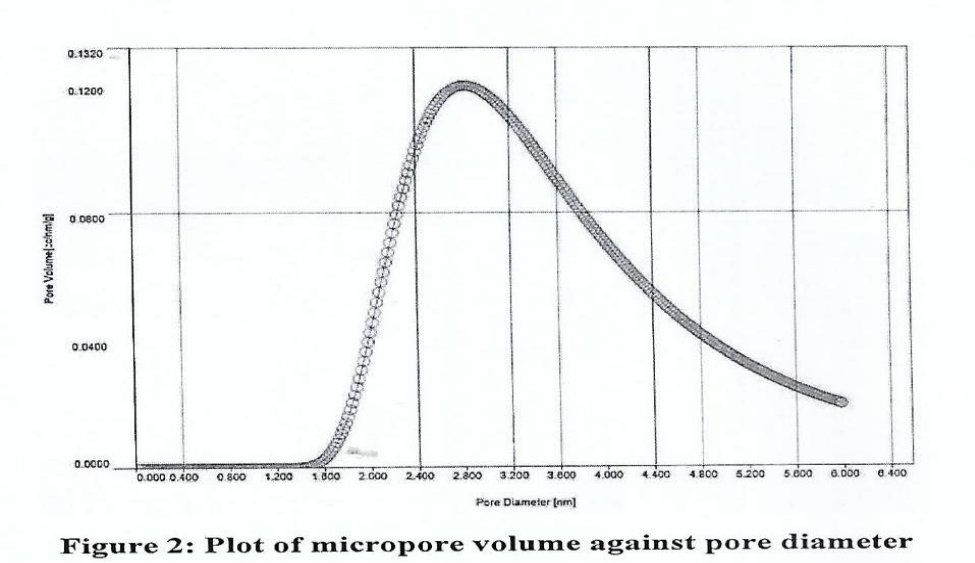


Figure 2: Plot of micropore volume against pore diameter

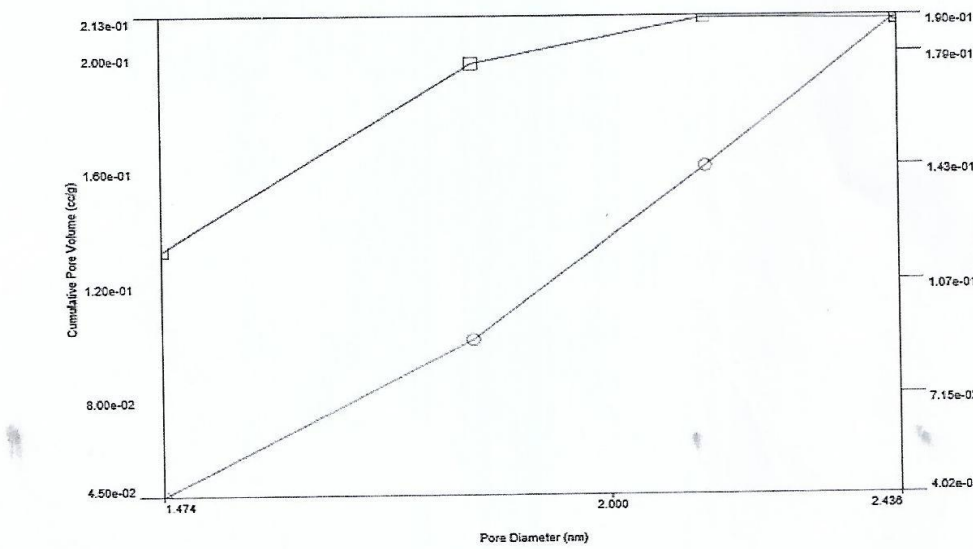


Figure 3: Plots of Cumulative Pore Volume, Surface Area Against Pore Diamet

XRS-FP ANALYSIS OF CATALYST

In order to determine the composition of the elements present in the catalyst sample, XRS-FP analysis was carried out using Gaussian method and results was as presented in Table 4.8. it was observed that the catalyst of various elements, but the major element are CaO with concentration of 42.516(wt.%), K2O with concentration of 12.163(wt.%) and SiO2 with concentration of 23.942(wt.%). Other elements are present in small qualities, but also aid in biodiesel synthesis.

The high CaO indicated that the residual ash has potential to be used as alkaline base for biodiesel production, and the presence of K2O that supported the strength of basicity of the catalyst. However, the presence of SiO2 showed that residual wood ash possesses some acidic strength but SiO2 also help the production of biodiesel due to it weak base nature. The raw results as obtain from the analysis are presented in APPENDIX.

TABLE 4: XRS-FP ANALYSIS REPORT

Component	concentration (wt.%)	mole (%)
SiO2	23.942	25.859
P2O5	2.212	1.011
MnO	1.339	1.225
Fe2O3	2.889	1.174
SO3	4.047	3.280
CaO	42.516	49.202
MgO	1.142	1.839
K2O	12.168	8.383
Al2O3	5.629	3.583
TiO2	0.910	0.740
Cl	1.611	2.950
SnO2	0.886	0.382
Others	0.709	0.372
TOTAL	**100**	**100**

SEM ANALYSIS OF CATALYST

The results of SEM ANALYSIS carried out on residual wood ash used as catalyst at magnification of 500Kand 100x are presented in figure 4.5 observed from the figures indicate a cohesive jointed-like crack shape with permeable surface. The structure showed an ice-like surface mineral particle accountable for slick flora of the element present. The soapy like look could be attributed to the presence of high concentration of SiO2 found in the catalyst. However, the whiteness, brightness and opacity look could be due to TiO2 present. The fusion adherent effect and brilliant glossy glaze found in the residual wood ash via XRS-FP Analysis report could be attributed to the presence of CaO and K2O, which make it is as a potential alkaline base catalyst for synthesis of biodiesel.

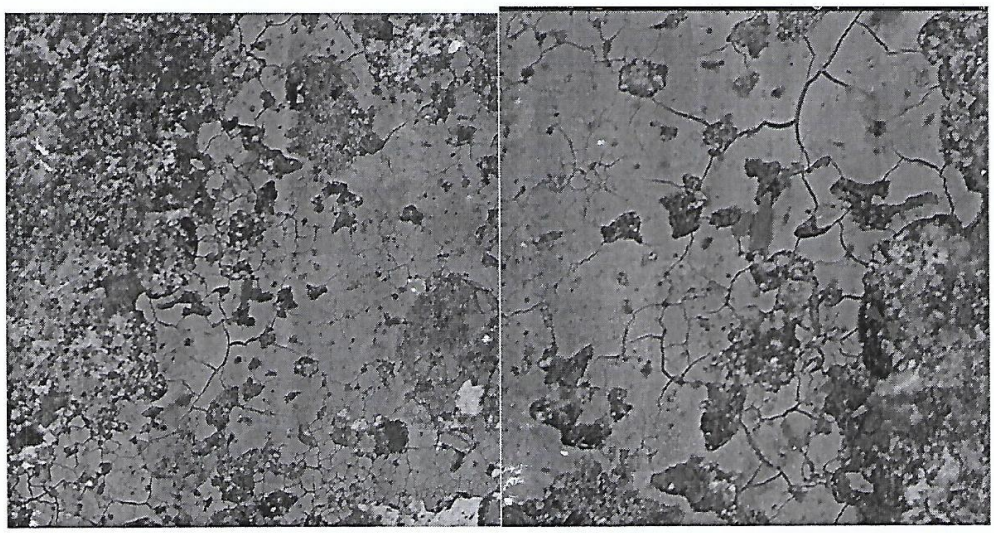


Figure. 4: SEM Analysis Images

FTIR ANALYSIS OF CATALYST

Figure 4.6 represented the results obtain for the FTIR analysis of catalyst sample used for biodiesel synthesis. Various functional elements are found at different wavelengths and angular phases. The peak noticed from 486.08 – 655.82 indicated the presence of saturated aliphatic (methylene), -(CH2)n- with rocking stretch based on the peaks valued around 1141.9 and 1404.22 indicated the presence of cyclohexane ring vibration, methylene C-H asymmetry and symmetry stretch, also present in methylene C-H stretch. Found around the 3402.54 to 3796.04 present functional groups such as alkyne C-H stretch, hydroxyl group, H-bounded, -OH stretch, polymeric –OH stretch, primary alcohol and phenol with –OH stretch.

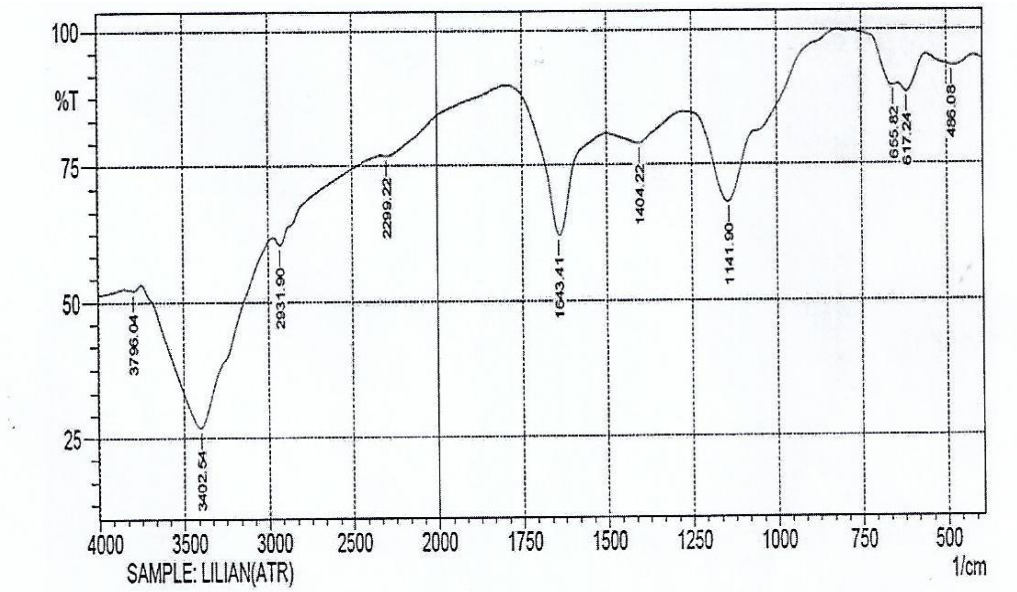


Figure. 5: FTIR analysis of Catalyst

Quantitative Analysis Report

Further analysis via quantitative analysis on the residual wood ash used as catalyst based on phase data view showed the plot of intensity against the angular phase diagram. The plot showed a zig-zag plot with graphite as the major compound containing carbon as the major element. Other compounds such as Quartz, Adamite, Gahnite, Zinicite, and Willemite are presented with figure of merit based on weight fraction via the analysis in figure 6. The presence of Quartz identified in the catalyst current which helped in oscillatory lubrication responsible for low viscous product. The presence of adamite in the catalyst helped in formation of biodiesel colour, usually adamite always yellow in colour which responsible for light yellowish biodiesel produced in this work. Other compounds also aid in biodiesel formation during production.

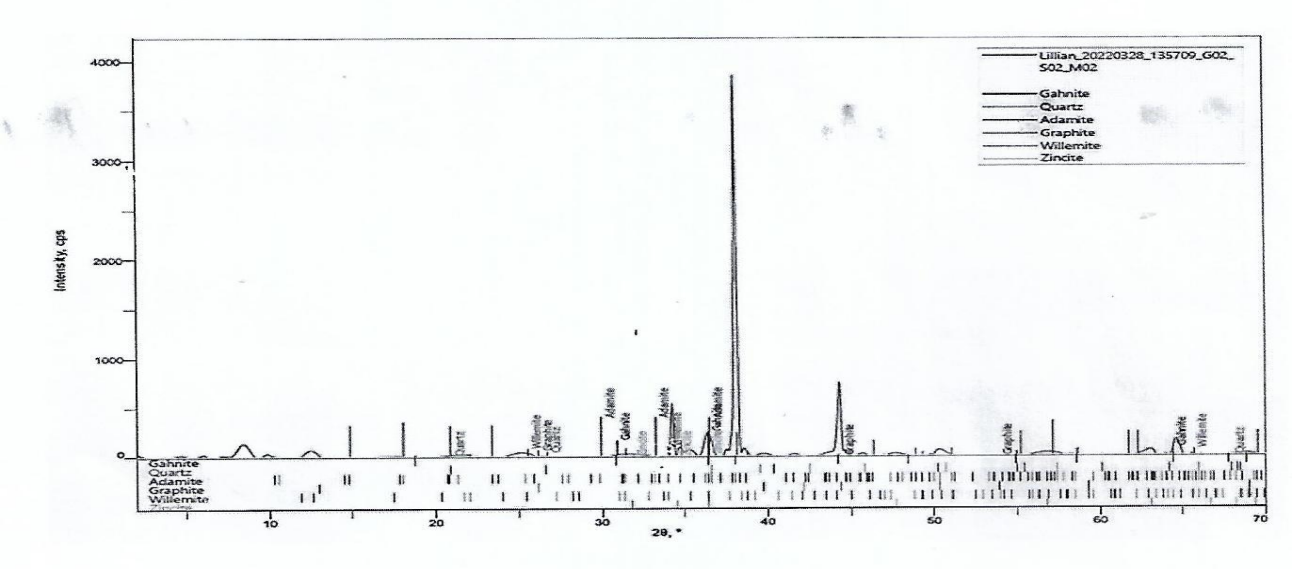


Figure. 6: Quantitative analysis plot

Physicochemical Properties of Biodiesel Produced

In order to examine the qualities of biodiesel produced from *Allanblackia floribuna's* seed oil using residual wood ash catalyst, the physicochemical properties p the diesel was examined and the results were compared with biodiesel recommended standard-ASTM D6751 and EN 14214. Observations from the table (Table 4.10) indicated that the properties of the *Allanblackia floribunda's* biodiesel produced are well within the biodiesel recommended standards. Nearly all the properties of oil have reduced in values as the conversion through two steps processes have proved to be effective ways for synthesis of biodiesel with an active catalyst from residual waste ash.

The density of the biodiesel was 860kg/m^3 which is within the standard given. Density and other gravities are important parameters for diesel fuel injection systems. The flash point for the biodiesel was 145ºc well above the 130ºc minimum ASTM recommended range and thereafter no risk of fire outbreaks in case of accidents. Sulistyu *et al.*, (2008) gave similar report.

The high saponification value of the biodiesel 190.40mg/KOH/g are normal triglycerides and very useful in production of liquid soap and shampoo. The cloud point 14ºC. This is the temperature at which crystals first start to form in the fuel. At this point the engine cannot run and is a big problem to a temperate region. The free fatty acid content in the biodiesel was 0.14%. High free fatty acid is undesirable as it is an indication of deterioration.

Table 5: physicochemical Properties of Biodiesel

Parameter	Biodiesel	ASTM D6751	EN 14214
Color @ 27ºC	Light yellowish	-	-
pH	8.2	8.0	7.8
Clarity	Clear liquid	Clear liquid	Clear liquid
Density (kg/m^3) @ 25ºC	860	870-900	860-900
Viscosity @ 40ºC/ (mm^2/s)	4.2	1.9-6.0	3.5-5.0
Moisture content (%)	<0.001	<0.03	0.02
% FFA (as oleic acid)	0.14	0.40 max	0.25 max
Acid value (mm KOH/g oil)	0.28	0.80 max	0.5 max
Iodine value (g I_2/100g oil)	68.50	-	120 max
Saponification value (mg KOH/g oil)	190.40	-	-
Peroxide value (meq O_2/kg oil)	4.86	-	12.85
HHV (MJ/kg)	40.61 `	-	-
Cetane number	59.67	57 min	51 min
API	33.03	39.95 max	-
Diesel index	68.99	50.4 min	-
Flash point ºc	145	>130	>120
Pour point ºc	-7	(-11) – (-9)	
Cloud point ºc	18	-	-

Transesterification of biodiesel synthesis and its statistical Analysis

Table 5 presented the experimental nine results of biodiesel produced from *Allanblackia* floribunda oil with the variables design. It was observed that the maximum biodiesel yield was obtained at run 6 (96.80% wt./wt.), while the minimum yield was obtained at run 1 with yield of 87.10% (wt./wt./). This proved that the conversation of *Allanblackia floribunda* oil to biodiesel via transesterification was successful, and the catalyst derived from wood ash is suitable for the synthesis. For the statistical analysis, the graph of each variable was plotted using Microsoft Excel plots. Figure. 7 (a) showed the plot of M-OH/oil molar ratio on the yield of biodiesel with a response linear equation, at a coefficient of determination of R^2 =

99.92%. This value of 85.13 in a slope of 2.1 Figure 4.8b. However, Figure. 3.8c displayed vertically

Table 6: The Results of the Experimental Biodiesel

SN yield	X_1 (min)	X_2 (wt.)	X_3 (vol/vol)	Experimental (% wt/wt.)
1	60	3	3	87.1
2	60	4	4	89.2
3	60	5	5	94.6
4	70	3	3	88.9
5	70	4	4	90.2
6	70	5	5	96.8
7	80	3	3	91.4
8	80	4	4	93.6
9	80	5	5	95.6

Perpendicular plot showing the reaction time only varies with equal proportion and so is the biodiesel yield. The high R^2 observed in the plots showed there are mutual interactions among the variables considered for synthesis of biodiesel, and the Microsoft excel proved to be good optimizer software.

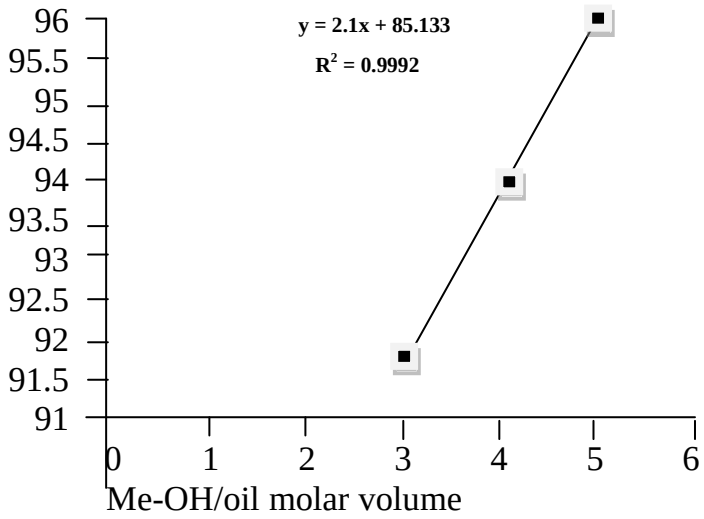


Figure. 7: Plot of M-OH/oil molar ratio on the yield of biodiesel

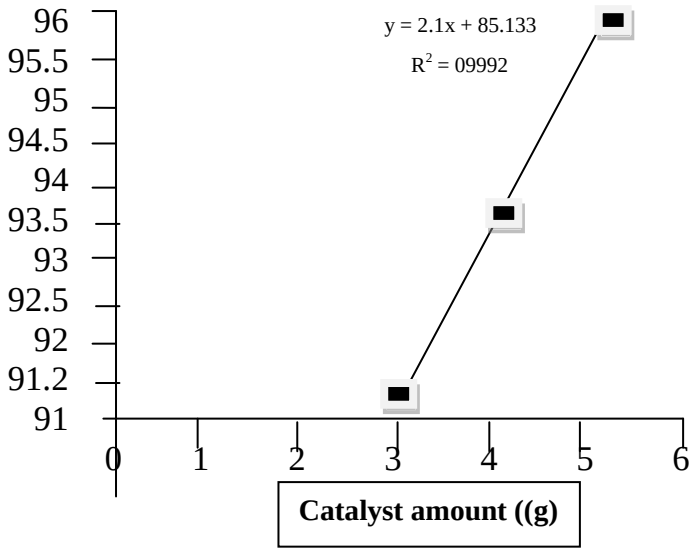


Figure. 8: Plot of Catalyst Amount on the Yield of Biodiesel

Catalyst Reusability Test

In order to examine the strength of catalyst developed from residual wood ash, the used catalyst after production was recycled, and the impurities in the catalyst was removed by washing in primary alcohol, centrifuge for 30 min at 3500 rpm, and then filtered. The residual catalyst was oven dried to constant weight and then cools at room temperature for 1 h. The obtained purified catalyst was then reused. The results obtained at different reusability test were presented in Figure 9 using Microsoft excel plot. Observation from the graph showed the catalyst strength start to drop at 8th cycle as the yield of biodiesel greatly decreased from 96.80 (% vol/vol.) to 73.10 (% vol/vol.). Hence, catalyst reusability test was stopped at 4th cycles. This observation showed that catalyst basic strength deteriorated owing to immediate products of mono/diglyceride formed during reaction, which blocked the catalyst holes, and the development of water oxygen reaction takes place at the catalyst surface also reduces the catalyst strength (Adepoju, 2021).

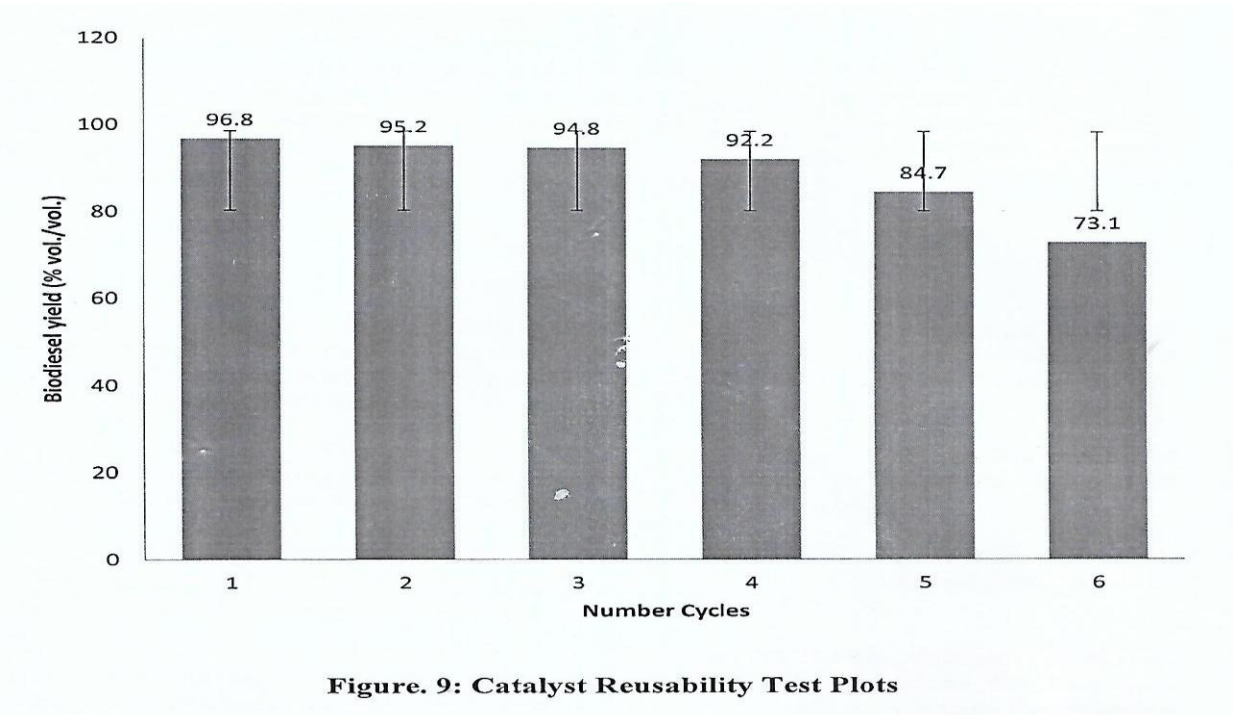


Figure. 9: Catalyst Reusability Test Plots

Conclusion

The study on process variables of oil extraction from Nigerian tallow seed (Allanblackia floribunda) with different solvents to synthesized biodiesel was investigated. Result from present study has revealed that the polarity of solvent significantly influenced the colour and the oil yield extracted from Tallow seeds. In addition, it has been found out that ethanol produced the least oil yield and quality compared with all the solvents used in this study. It can therefore be concluded that Hexane and petroleum ether are the best solvents for oil extraction at low temperature and therefore the findings from this study can be useful in choosing the suitable solvent for the oil extraction to maximize yield as well as maintain oil quality.

The highest crude oil yield from petroleum ether means that petroleum ether is more efficient in extracting non-polar tallow seeds fraction than Hexane despite being non-polar.

(i) *Allanblackia floribunda* seed were found to be rich in oil with 60.40% of fat content, which allow the possibility of industrial usage.
(ii) The wood ash characterization showed that the derived catalyst is rich in calcium, potassium and silicon dioxide.
(iii) The conversion off oil to biodiesel was successful with maximum yield of 96.8% (wt/wt).
(iv) Statistical analysis via Microsoft exce4l proved that there are mutual interactions between variable considered for transesterification.
(v) The qualities of biodiesel are within the recommended standard value, hence could serve as replacement for conventional diesel.

It is however recommended that in order to get the best result from the Allanblackia Floribunda Oil Seed to biodiesel synthesis, kinetic and thermodynamic study of biodiesel conversion should also be studied. The modeling and simulation of biodiesel should also be considered. More studies should be carried on the use of stabilizer on Allanblackia floribunda Oil Seed to reduce the viscosity so that it can be used in its straight form.

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