

## **SYNTHESIS AND CHARACTERISATION OF BIO-FUEL FROM *Parinari curatellifolia* SEED USING CLAY INCORPORATED WITH BAKING YEAST AS CATALYST**

\*<sup>1</sup>Ibrahim, M., <sup>1</sup>Gungshik, J.R., <sup>1</sup>Cletus, A.A., <sup>1</sup>Nanribet, G.S. and <sup>2</sup>Bisiriyu, M.T.

<sup>1</sup>Department of Chemistry, University of Jos, P.M.B 2084, Jos, Plateau, Nigeria

<sup>2</sup>Department of Chemistry, Federal University of Technology, P.M.B 63, Minna, Niger, Nigeria

\*Corresponding Email: [mibrahim@unijos.edu.ng](mailto:mibrahim@unijos.edu.ng)

Received: 12 May 2026

Accepted for publication: 15 June 2026

Published: 01 July 2026

### **ABSTRACT**

The growing energy crisis and the environmental hazards constituted globally by the over dependence on fossil fuel can only be resolve by sourcing viable alternatives especially from our abundant renewable vegetative reserves' rich in bio-oil with potential for biodiesel production. The synthesis of biodiesel from *P. curatellifolia* seed using n-hexane as solvent yield 36.2 % (v/w) which considered it a viable feedstock for biodiesels. The *Parinari curatellifolia* seed oil have high amount of acidic value, iodine value, saponification value and FFAs of 6.67, 82.62, 135.5 and 3.3 mgKOH/g respectively due relatively high degree of saturated bonds. The GC-MS analysis revealed high composition of major methyl ester compounds suitable for biodiesel production such as the oleic acid, stearic acid, palmitic acid, Linoleic acid and Eicosenoic acid. The favorable operating conditions for the biodieselsynthesis was 5% wt. catalyst loading, 3 hrs (reaction time), 9:1 (methanol to oil molar ratio) and 65 °C (reaction temperature) at 3000 rpm stirring rate to have a maximum of PCUME yield of 94.7 %. The important fuel properties such as Density, Flashpoint, Cloud Point, Calorific Value, Cetane Index and compared to mineral diesel and global biodiesel standards such as ASTM D6751 testing limit. The results show most of the properties were in good agreement with ASTM D6751 standard limits. Therefore, advanced technology design and methodology couple with favourable reaction conditions *Parinari curatellifolia* seeds can be considered viable biodiesel feedstock.

**Key Word:** *Parinari curatellifolia*, Bio-oil, Operating parameters, Mineral diesel, Feedstocks, Flash Point.

### **1.0 INTRODUCTION**

One basic need for socio-economic growth and development throughout the world today is the quest for clean, sustainable and environmentally friendly energy resource that can replaced fossil fuel with more efficient performance in all areas of domestic and industrial energy applications. However, Population growth, industrialization and technological advancement are factors responsible for increasing demand for energy and resulting in conflicting energy crisis (Vedharaj *et al.* 2014) that can only be resolved by instituting alternative sustainable energy solutions. The petrol fuel has been over exploited as a main source of energy coupled with its harsh ecological impact, lack of sustainable availability and rising cost charge stressed the urgent need to source a viable alternative renewable source of energy which are sustainable, economically reasonable and environmentally harmless (Canakci and Van Gerpen, 2001; Sanli, 2008; Berrios and Skelton, 2008).

Reports by several literature has identified biodiesel as the bio-fuels derived from various biomass sources using different technologies such as pyrolysis, gasification, fermentation and transesterification reaction to represent potential fuel substitute to mineral diesel (Demirbas, 2005; Chen *et al.*, 2013). Biodiesel is a mixture of monoalkylesters of long chain fatty acids resulting from renewable lipid feedstock of animal fats or plant oil through transesterification process (Kafuku and Mbarawa, 2010; Burton, 2008). Transesterification is a process whereby a number of moles of triglyceride molecule of complex free fatty acid obtained from

different plant biomass, reacts with moles of alcohol in the presence of a catalyst and at given temperature to form a methyl/ethyl ester(biodiesel) and glycerol (Demirbas, 2005; Chen *et al.*, 2013). These fuel type can be sourced from renewable, abundant, easily available and affordable feedstock that make its production cheaper, hence reduces the dependence on imported fossil fuels thereby controlling the economy and standard of living (Kivevele and Mbarawa, 2010). Although, biofuels also come with some disadvantages such as slightly lower calorific value, low cetane number, poor cold flow properties (pour and cloud point) and less oxidation stability which are associated with some feedstock properties (Encinar *et al.*, 2002; Ramadhas *et al.*, 2005).

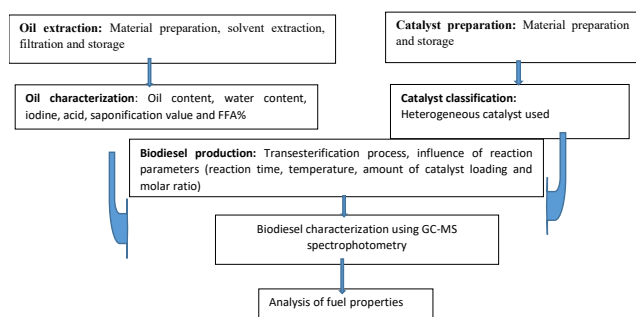
Biodiesel production depends on oil feedstock, amount of catalyst used and the technology employed but more than 70 % of its production cost depends on oil feedstock and catalyst used (Leung *et al.*, 2006; Dehkhoda *et al.*, 2013; Zhang *et al.*, 2014). According to Sanli, and Lee (2008), more than 95 % of biodiesel produced are from food grade edible oils such as sunflower oil, palm oil, soybean oil and rape seed oil using homogeneous catalysts but the extensive use of refined oils for biodiesel production results in food insecurity and price competition (Adewale *et al.*, 2015). Thus, the uses of homogeneous catalysts have also result to high production costs due to high amount of water demand during washing, number of purification steps, high wastewater management and the catalyst is not reusable after reaction. These factors contribute to rise the costs of biodiesel production and become a major obstacle towards its commercialization processes (Adewale *et al.*, 2015). Therefore, it

becomes necessary to search for non edible cheaper oil feedstocks and low-cost catalyst to optimised the biodiesel production process. Heterogeneous catalysts are mostly used to overcome the drawbacks of homogeneous catalyst and to enhance commercialization as they are easy to separate, reused, used even with low-quality oils feedstock, does not require much washing. Many researchers have produced biodiesel from non-edible oil seeds, waste oil and lignocellulosic as viable feedstock (Adewale *et al.*, 2015) and the refined feedstock is less expensive and with no conflicts with food stuffs, but associated with very high amount of FFA (greater than 2 %), high density, greater amount of saturated fatty acids, land competition and technological challenges (Tang *et al.*, 2018). Benhura *et al.* (2012) reported *Parinari curatellifolia* commonly known as “Mbula tree” as a tropical evergreen seedling tree, with pulp and seeds feed as food by arboreal animals and by man during food deficiency especially on drought season. The seeds kernel has been reported to contain high oil content > 38.5 % while some report > 60 % depending on the source geographical location (Bazongo *et al.*, 2014). However, this study seeks to synthesized biodiesel from *Parinari* seed using clay incorporated with baking yeast as catalyst and characterized the bio-fuel properties.

## 2.0 MATERIALS AND METHODS

### 2.1. Experimental Frame Work

The materials, equipment, techniques and procedures used in the preparation and analysis of both the catalyst and the biodiesel fuels produced are summarized in the flow chart, Scheme 1 below. All experimental procedures and measurements were done in triplicate and their average value were used in the determination of required fuel property.



Scheme 1: Flow Chart of the Research Frame Work

### 2.2 Materials

The apparatus and analytical equipment used in this research include: petri dishes, thermos-regulator heater with stirrer, electric digital precision weighing balance, pH meter, rotary evaporator oven, heating mantle, Soxhlet extractor and magnetic stirrer, speedo blender machine, pestle and mortal.

#### 2.2.1 Reagent/solvents

The reagents and chemicals used in this work include the following: potassium hydroxide (Iobachemie, gmbH 85 %) methanol (Merck Germany 99.5 % purity), potassium iodide solution, phenolphthalein (Merck Germany), n-hexane (99 % purity, Merck Germany) All chemicals used were of analytical grade and used without further treatment

### 2.3 Sample Collection and Preparation

Ripen *Parinari* fruits were collected from its tree plant known as “putu” in Nupe Language from Lapia LGA – Niger state, Nigeria. The tree plant (Fig. 1a), Dehulled fruit to obtain the seeds kernel (Fig.

1b) and sun-dried to remove moisture and ready for oil extraction process. The baking yeast were collected from Faringada market in Jos, Plateau state (Fig. 1c) which was already prepared and Kaolin clay (Fig. 1d) was collected from a Kaolin processing mill in Barikin Ladi LGA, Plateau state. The biodiesel feedstock sample was identified in the biological Garden University of Jos.



Figure 1(a): *P. curatellifolia* tree



Figure 1(b): *P. curatellifolia* seeds

## 2.4 Extraction and Characterization of *Parinari curatellifolia* Oil

### 2.4.1 Oil Extraction

Bio-oil was extracted from dried *P. curatellifolia* seeds kernel using solvent extraction method. The sample was first dried for 21-days under room temperature and macerated into a coarse material, in which 25 g of sample was weighed into a 250 cm<sup>3</sup> beaker and 200 cm<sup>3</sup> of n-hexane was added and allowed for 72 hrs which was then filtered and the filtrate was evaporated using Rotary evaporator. pre-treatment of the oil by filtering to remove solid dirt content using filter paper and dehydration to remove traces of water content present in the oil by using a low-pressure distillation process (at 105 °C). The produced cleaned oil was stored in a freezer for subsequent physicochemical analysis and characterization.

### 2.4.2 Characterization of *P. curatellifolia* Seed Oil

The physicochemical properties of PCSO like; acid value, oil content, saponification value, iodine value and percentage free fatty acid were determined and analyzed in accordance to American Standard Testing Method (ASTM, 1983) and American Official for Analytical Chemistry Standard method (AOAC, 1993; AOAC, 1997).

#### a) Determination of oil content (yield)

The dried *P. Curatellifolia* seed, 20 g was weighed and subjected to a mechanical pressing machine and pressed at different pressures 200, 250 and 300 kPa to extract the oil and the residue was equally weighed and recorded. The pressure was gradually increased until no more change in the residual weight was observed on the feedstock material. The triplet experiment was taken for the maximum pressure attained and the average value of residual weight was used in the calculation of percentage oil content of the seeds using equation (1) below;

$$\% \text{ of oil content} = \frac{\text{weight of dried seeds} - \text{weight of residual}}{\text{weight of dried seeds}} \times 100 \text{ --- (1)}$$

#### b) Determination of Moisture Content

Exactly 5.42 g of the sample seeds were dried in the oven for 12 hours at temperature of 105 °C to remove moisture and then allowed to cool in a desiccator for 30 min. The dried seeds were reweighed again and weighed 5.09 g. The procedures were repeated three times and the average weights were used in the calculation of the moisture content using Eqn. (2) in accordance to AOAC, (1995) procedures.

$$\frac{\% \text{ water content} = \frac{\text{weight of seed before dried} - \text{weight of dried seeds}}{\text{weight of seed before dried}} \times 100}{-} \quad (2)$$

### c) Acidic Value

Acidic value (AV) is the measure of FFA present in the oil which correspond to the weight in millimetres of KOH required to neutralize 1 gram of the organic acid present in the oil. Exactly 1 g of the oil was dissolved in a solution containing 25 cm<sup>3</sup> of diethyl ether and 13 cm<sup>3</sup> of ethanol in 250 cm<sup>3</sup> Erlenmeyer flask followed by gently warming. The mixture was titrated using 0.1 N of KOH using phenolphthalein as an indicator, until pale pink colour was observed. According to AOCS (1993) procedures and the value of AV was calculated as follows (Eqn. 3)

$$AV = \frac{\text{titre volume} \times \text{grams of NaOH in 0.1M of KOH solution}}{\text{sample weight in grams}} \quad (3)$$

### d) Free fatty Acids (%)

The FFAs are expressed as the percentage of half of value of acidic value and calculated according to the equation bellow;

$$FFA = \frac{\text{Acidic value}}{2} \times 100 \quad (4)$$

### (e) Saponification Value

A gram of the oil sample was dissolved in 25 cm<sup>3</sup> of ethanolic potassium hydroxide solution made by 0.5 M in 95 % ethanol. The solution was heated for one hour with gently stirring using magnetic stirrer. While hot, 3 drops of phenolphthalein indicator were added, the colour changed to pink and immediately titrated with 0.5 M HCl until the pink colour disappears. AOCS, (1997) procedures were adopted and the saponification value was obtained by using equation (eqn. 5) below

$$SV = \frac{(b-a) \times \text{grams KOH in 0.5M KOH solution}}{\text{sample weight in grams}} \quad (5)$$

Where a = volume of oil solution and b = volume of titre blank solution

### (f) Iodine Value

Several methods are available for iodine determination but the Hanus method (Association of analytical chemists) was used in this study for the determination of iodine value and the procedure describe by Giwa *et al.* (2016) was adopted. Exactly 10 ml of the oil was placed in 250 cm<sup>3</sup> conical flask and anhydrous chloroform was added to the flask followed by 3 cm<sup>3</sup> of Hanus solution and the flask was covered with flask stopper. Then the content was mixed and place in drawer for 30 minutes after which potassium iodide solution (1 cm<sup>3</sup> of 15 %) was added to the flask in order to wash down any iodide that might be found on the stopper. The solution was then titrated against NaCO<sub>3</sub> until it becomes light yellow. The blank determination was equally carried out under same condition (Giwa *et al.*, 2016).

## 2.5 Preparation of Catalyst for Transesterification Reaction

The baking yeast was pulverized into fine powder and thoroughly mixed with clay to homogeneity which formed the catalyst. The catalyst was introduced into the alcohol (methanol) and stirred to formed homogenous mixture of methoxide.

## 2.5.1 Transesterification Process

### (i) Experimental set up

In each reaction carried out, 25 cm<sup>3</sup> of the oil was treated with varied amount of methanol (3 to 18 cm<sup>3</sup>) and catalyst (1 % to 6 %) in 250 cm<sup>3</sup> round bottomed flask reactor. The reactor was fitted with a reflux condenser, water bath, thermal heating controller, digital thermometer, magnetic stirrer, water pump and water jar. The reflux condenser was fitted on the reactor to ensure any vapours given off were cooled back to the reactor. Heating controller and water bath maintain temperature of the reactor while water pump and water jar circulate and control the water loss. The magnetic stirrer was used to achieve a homogeneous mixture at a constant mixing speed of 3000 rpm.

### (ii) Biodiesel Production Process

The PCSO was measured in 250 cm<sup>3</sup> Erlenmeyer conical flask and preheated at 105 °C for 5 min to reduce moisture content and allowed to cool to 65 °C. Exactly 1.25 grams (5 % wt/vv of oil) of catalyst and 9 cm<sup>3</sup> of methanol were measured into the reactor and gently stirred to allow uniform mixture. Then, 25 cm<sup>3</sup> of preheated oil at 65 °C was measured and transferred into the reactor to commence the reaction. The reaction proceeds to completion in 2 hrs, allowed to cooled and two distinct layers of catalyst at bottom and the product which is the mixture of glycerol and biodiesel at the top was formed as depicted in Fig. 2(a). The top mixture was separated, measured and transferred into a separating funnel to cool overnight. After then, two more distinct layers were formed composed of glycerol at the bottom and the top layer was a mixture of *Parinari curatellifolia* methyl ester (PCUME) (biodiesel) and unreacted methanol as shown in Fig. 2(b). All the experiments were conducted in accordance with the stated procedures at different experimental conditions and the biodiesel was then separated from glycerol ready for purification and yield estimation processes.

### (iii) Product Purification and Yield Estimation

The biodiesel obtained after separation from glycerol need to be purified to eliminate impurities such as excess alcohol, traces of catalysts, glycerol and soap caused by presence of tri, di and mono-glycerides that remain during reaction. The purification is important in order to have quality biodiesel that will comply with specification and acceptable in the market. Water washing is most preferred because it is the easiest method and cheaper to apply. Before applying water washing, excess methanol was removed using batch laboratory vacuum distillation flask. The product was washed four times using warm deionized water. The washed biodiesel was dried by using anhydrous MgSO<sub>4</sub> over night to remove water content in order to obtain pure biodiesel. The weight of pure biodiesel was used to find the percentage yield using the following equations.

$$\% \text{ yield of PCUME} = \frac{\text{weight of PCUME}}{\text{weight of product}} \times 100 \quad (6)$$

## 2.6 Characterization of the Biodiesel

The fuel properties of PCUME were determined and tested according to ASTM testing methods for biodiesel and compared to ASTM D6751 standards limits and conventional automotive mineral diesel (number 2 diesels). According to Graboski *et al.* (1996), number 2 diesel is a low sulphur diesel with 30 % aromatic content. The properties determined include: fatty acid composition, density, pour point, cloud point and flash point

### (i) Determination of Free Fatty acid composition

Analysis of fatty acid composition in the produced biodiesel was done by Gas Chromatography-Mass Spectrometry GCMS-2010 Shimadzu instrument Japan, at the Yobe state university Damaturu.

### ii) Flash Point Determination

The flash point of the oil was determined experimentally by heating liquid in an instrument that contained the oil in a beaker which is known as oil flash point detector device. A small flame was introduced just above the oil surface and the temperature at which the oil ignited was recorded as the flash point of the biodiesel.

### iii) Pour Point Determination

The bio-oil sample was then cooled inside a cooling bath after the flash point determination to allow the formation of crystals, and the temperature at which the crystal was form was recorded.

### iv) Cloud Point Determination

The cloud point is defined as the highest temperature at which an oil begins to solidify. A little quantity of the bio-oil was placed in ice bath with a fixed thermometer and the temperature at which the oil began to condense was recorded as the cloud point of the bio-oil.

### vii) Calculation of Cetane Index

Cetane Index is a calculated value used to estimate ASTM cetane number in the absence of an engine to test the produced biodiesel. ASTM D976-06, (2016) was the test method adopted and Cetane Index formula was used to calculate Cetane Index (CI) value by directly estimating the ASTM cetane number. For the distillate fuels, the density at 15 °C and midpoint temperature (T50) is used as variable in the developed model (Eqn. 8). The calculated value gives a reasonably close value approximated to Cetane number. Cetane index of PCUME was calculated using empirical equation T50;

$$CI = 454.74 - 1641.416D + 774.74D^2 - 0.554T_{50} + 97.803[\log_{10}T_{50}]^2 - (7)$$

where CI is the Cetane Index number, D is the fuel density at 15 °C = 0.8915 g/ml, and T50 is the mid temperature corresponding to 50 % of distillation received = 23.42 °C.

**Table 3: Bath and Sample Temperature for Cloud and Pour Point in Testing Cabinet**

Bath Temperature Setting (°C)	Sample Temperature Range, (°C) for cloud Point	Sample Temperature Range, (°C) for pour point
0 ± 1.5	Start to 9	-24 to -42
-18 ± 1.5	9 to -6	9 to -6
-33 ± 1.5	-6 to -24	-6 to -24
-51 ± 1.5	-24 to -42	-24 to -42

Source: ASTM D2500-17 (2017) and ASTM D97-17B (2009).

### 2.7 GC-MS Characterisation of the Bio-oil, Fatty Acid Composition Analysis

The GC-MS analysis was carried out with Agilent 7890B gas chromatograph coupled with an Agilent 5977A Network mass selective detector, in a positive ion electron impact (EI) mode. Constituent identification was achieved by comparison of the NIST library data of the peaks with those reported in the literature. Percentage composition of individual constituent was computed from GC-MS peak areas. The analysis was carried out at Yobe State University, Damaturu. The number of carbon bond, retention time, molecular weight, molecular formula and amount yield from PCUME as shown in Table 7.

## 3.0 RESULTS AND DISCUSSION

### 3.1 Physio-chemical Properties of the Bio-oil (PCSO)

The results of the physicochemical properties of PCSO are presented in table four (4) and parameters such as oil content, moisture content, iodine value, acid value, saponification value and free fatty acid values were used to characterised the bio-oil.

**Table 4: Physicochemical Properties of PCSO**

Property	<i>P. curatellifolia</i> oil
Oil content (%)	36.20
Acid value (mgKOH/g of oil)	6.67
Iodine value (gI <sub>2</sub> /100g of oil)	82.62
Saponification value (mgKOH/g of oil)	135.5
FFA (mgKOH/g)	3.33

### 3.2 Percentage Yield

Four litres of biodiesel was obtained from 12 kg of dry *Parinari* seeds which represents a yield of 36.2 % (v/w) which was slightly less compared to 36.75 % and 38.5 % reported from a same seed study by Ndaba (2014) and Bazongo *et al.* (2014). This difference may be attributed to soil and geographical location, species variation and method of extraction. The *P. curatellifolia* seeds oil content has qualified it as a potential favourable feedstock for biodiesel production (Ahmad *et al.*, 2011; Atabani *et al.*, 2012).

### 3.3 Moisture Content

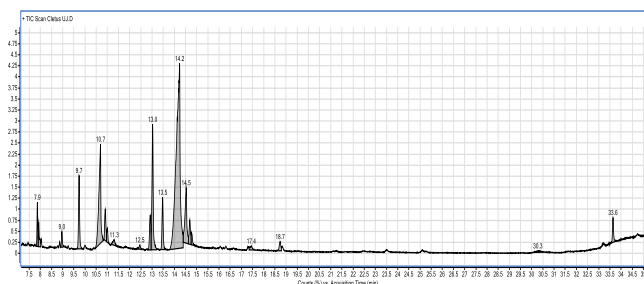
The moisture content of 6.0 % PCSO was a slightly higher by weight compared to 5.1 % reported by Oladimeji and Bello (2011) but for a bio-oil not used for biodiesel production.

### 3.4 Free Fatty Acid Value of the Oil

The acid value was used to determine the amount of FFA in the oil, which was found to be 3.3 mgKOH/g which was less compared to 5.15 and 5.9 mgKOH/g reported by Oladimeji and Bello (2011) and Bazongo *et al.* (2014) respectively from *parinari* seeds oil. According to Ghatikar (2015), the feedstock with high amount of FFA (>1 %) are more vulnerable to soap formation, reduce the catalyst efficiency, increases viscosity of the oil and hinder the separation of glycerol from the mixture. All these consequences result to reduction of biodiesel yield but heterogeneous catalyst is preferred for bio-oil with FFA greater than 1 %, to prevent more soap formation as the catalysts are reported to be more tolerant to higher FFAs and help to increase the biodiesel yield (Ramadhas *et al.*, 2005; Marchetti *et al.*, 2007; Berchmans *et al.*, 2008).

### 3.5 GC-MS Characterisation of the Seed Oil

The representative GC-MS peaks characteristics are shown in chromatogram (Fig. 9) and the major methyl ester compounds extracted from the chromatogram are presented in table 5 below



**Fig 9: GC-MS Chromatogram of the biodiesel**

**Table 5: Methyl Ester Composition of the bio-oil (PCUME)**

S/N	FAME (common name)	Molecular formula	Molecular weight	Retention time	Peak Area (%)
1	Hexadecanoic acid methyl ester (palmitic acid)	C <sub>17</sub> H <sub>34</sub> O <sub>2</sub>	270	9.7	10.8
2	9,12-Octadecadienoic acid (Z, Z) methyl ester (Linoleic acid)	C <sub>19</sub> H <sub>34</sub> O <sub>2</sub>	294	12.9	6.04
3	9-Octadecanoic acid, methyl ester, (E) – (Oleic acid)	C <sub>19</sub> H <sub>36</sub> O <sub>2</sub>	296	13	21.51
4	Methyl stearate (Stearic acid)	C <sub>19</sub> H <sub>38</sub> O <sub>2</sub>	298	13.5	8.14
5	Cis-11-Eicosanoic acid, methyl ester (cis - vaccenate)	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>	282	14.2	100
6	Eicosanoic acid, methyl ester (methyl arachidate)	C <sub>20</sub> H <sub>42</sub> O	298	7.9	4.37
7	Docosanoic acid, methyl ester (methyl behenate)	C <sub>22</sub> H <sub>44</sub>	308	10.9	5.09
8	Tetracosanoic acid, methyl ester (Methyl myristate)	C <sub>24</sub> H <sub>50</sub>	338	7.9	3.35

### 3.6 The Fuel properties of the Biodiesel

The fuel properties of the biodiesel produced were tested according to ASTM standard methods and compared to ASTM D6751 standard limits as summarized in the Table 6. The results of the fuel properties show the cloud point, pour point and acidic value to fall within the acceptable range as prescribed in ASTM D6751 while the density was slightly higher than the recommended limits. However, Ong *et al.* (2013) reported the density of biofuel to depends on the chain length of methyl ester and its saturation level which means, the higher the unsaturation of methyl esters, the higher its density. This may be responsible for the higher density of the biodiesel produced (PCUME) and reports characterised fuels oil with high density to be less compressible and causes poor atomization process resulting to incomplete combustion when burned in an engine (Canakci *et al.*, 2001; Ong *et al.*, 2013). This challenge can be address by enhancing the methodology of production.

In this study, the PCUME exhibits higher flash point temperature of about 174 °C which still fall within the range of temperatures considered safety during storage and transportation (Canakci *et al.*, 2001). The minimum allowable flash point temperature limit for biodiesel by ASTM D6751 is 130 °C for pure biodiesel but PCUME still fall between the minimum and maximum limits. Also, the calorific value of biodiesel (39.3 MJ/kg) was found to be slightly lower than that of mineral diesel (42-46 MJ/kg) as in many other reported biodiesel products (Ramadhas *et al.*, 2005; Hulwan and Joshi, 2011). The cetane number represented by cetane index obtained from the distillation process was found to be (44.4) which was considerably lesser compared to the minimum and maximum recommended range of (47–65) for biodiesel by ASTM D6751 standard limits. The relatively lower value of cetane number may be due high unsaturated level of methyl ester, number of double bond and mid temperature of distillate (T50) during distillation process of

methyl ester as reported in similar studies by Knothe *et al.* (2003) and; Knothe (2005, 2014).

**Table 6: The Fuel Properties of the Bio-diesel (PCUME)**

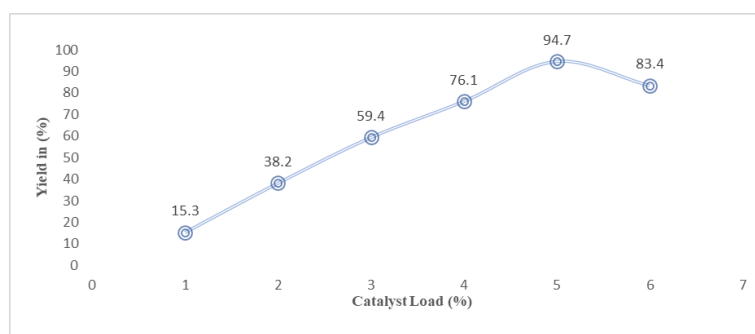
Property	Petrol diesel (ASTM D975 limits)	Biodiesel (ASTM D 6751 limits)	PCUME
Density @ 15°C (kg/m <sup>3</sup> )	850	850-880	891.5
Density @ 20°C (kg/m <sup>3</sup> )	850	850-880	888.0
Flashpoint (°C)	60-80	130 min	172
Cloud Point (°C)	-15 to -5	-3 to 12	9
Pour Point (°C)	-35 to -15	-15 to 10	-6.1
Calorific Value (MJ/kg)	42-46	–	39.3
Acid Value (mgKOH/g)	–	0.5 max	0.1
Cetane Index	–	47 – 65	44.4

### 3.7 Influence of Reaction Parameters

The optimisation of the biodiesel yield was used to evaluate the effective catalyst performance by studying the influence of reaction conditions. Demirbaş (2005) reported that, the reaction temperature, alcohol to bio-oil molar ratio, and amount of catalyst loading are the main factors influencing the conversion efficiency and rate of the transesterification process. The biodiesel yield in this study was obtained through experimental determinations by varying reaction parameters such as the methanol to bio-oil molar ratio, reaction time and percentage of catalyst loading while reaction temperature and stirring speed were kept constant at 65 °C and 3000 rpm respectively. The reaction temperature was kept at 65 °C as the temperature below the boiling point of methanol to avoid evaporation.

### 3.8 Effect of Catalyst loading

The amount of catalyst loading (% wt/v) for a given reaction has great influence in determining the product yield as excess methanol tends to shift reaction equilibrium to the product side. The effect of catalyst loading on the percentage yield of the of the bio-oil is shown in Fig. 10. Below

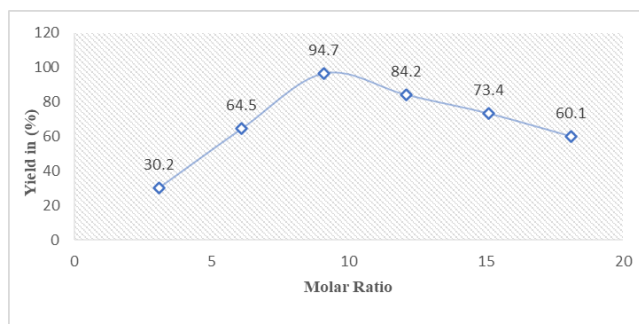
**Figure 10: Effect of Catalyst Load on the Yield**

The influence of catalyst concentration loading with respect to bio-oil yield was varied from 1 % to 6 % at intervals of 1 % and the yield was calculated using Eq. (1). The ratio of methanol to oil of 18:1 was used while reaction time was maintained at 3 hrs, the reaction temperature was kept at 65 °C and stirring rate was at 3000 rpm for all reactions. The results (Fig. 10), clearly shows a direct correlation, such that as the amount of catalyst loading increased from 1 % to 5

%, the rate of conversion also increased up to a maximum yield of 94.7 % while above 5 % catalyst, the yield was observed to drop drastically. This observation was similar to that reported by Kim *et al.* (2004) and Nurul *et al.* (2017) which was attributed to high viscosity of reaction mixture that resulted in poor diffusion of methanol to bio-oil and catalyst in the system. Therefore, catalyst loading of upto 5 % wt. was the maximum amount required to achieve the maximum yield of 94.7 % understated conditions.

### 3.9 Effect of Molar Ratio

The effect of alcohol to bio-oil molar ratio is presented in Fig. 11 below. Alcohol to bio-oil molar ratio is also an important parameter to be considered in the process of converting triglycerides to methyl esters. Generally, adopting the stoichiometric ratioing, three moles of methanol or alcohol are required to convert one mole of triglyceride during transesterification process (Lee *et al.* 2009) and in the case of heterogeneously catalysed reaction, the excess methanol helps to improve the rate of the transesterification processes (Kim *et al.*, 2004; Guo *et al.*, 2010; Issariyakul *et al.*, 2014; Lee *et al.*, 2014). In this study, methanol to bio-oil molar ratio was varied in the range of 3:1, 6:1, 9:1, 12:1, 15:1 and 18:1 and its influence in triglyceride conversion using 5 % wt. of catalyst load, 3 hrs reaction time, while reaction temperature and stirring speed were controlled at 65 °C and 3000 rpm respectively, showed the yield to increase with increase in the molar ratios. From the experiment, it was observed that the yield was increasing as molar ratio increases. Thus, Methanol to bio-oil (9:1) molar ratio gave the maximum biodiesel yield of 94.7 % and in which additional increase in molar ratio resulted in decreasing biodiesel yield as shown in Fig. 12. Report by Issariyakul and Dalai (2014) shows that polarity of the reaction mixture increases as the excess amount of alcohol increases which may causes the reverse reaction due to increase in the solubility of the glycerol and consequently reduces ester yield. In this study, the variation in the methanol to bio-oil molar ratio at (9:1) with 5 % wt. of catalyst loading, 3 hrs reaction time at 65 °C and 3000 rpm gave the maximum biodiesel yield of 94.7 %.

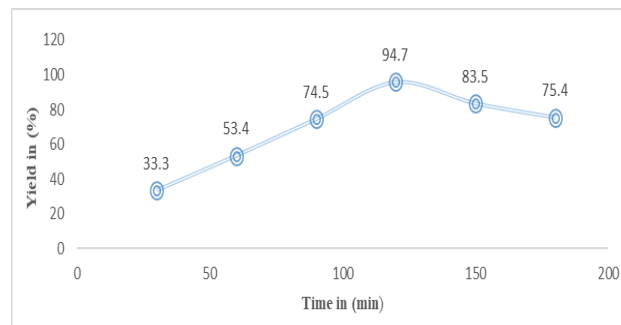


**Figure 11: Effect of Methanol to Bio-oil Molar Ratio on the Biodiesel Yield**

### 3.10 Effect of Reaction Time

The effect of reaction time on the biodiesel yield in course of transesterification reaction is presented in Fig. 12 below. The methyl esters yield during transesterification process was reported by Wei *et al.* (2009) to increase with an increase in reaction time and also, in a similar study by Li *et al.* (2018) revealed the rate of bio-oil conversion depends on how long the reaction takes place. The highest reaction time allowed for the optimum production of biodiesel using the PCSO, 5 % wt. catalyst loading and 9:1 methanol to bio-oil molar ratio was determined in this study by varying time from 30 min to 3 hrs at an interval of 30 min under the same reaction conditions. The

results obtained during conversion of triglyceride to methyl ester as presented in Fig. 13. Shows an increase of yield from 33.3 % at 30 min to 92.8 % after 120 min and after which the yield decreases. This behaviour was reported in a similar study by Leung *et al.* (2010), during transesterification reaction more reaction time will lead to a decreased in product yield caused by the backward reaction and it also promotes soap formation. This study revealed the favourable allowable reaction time for maximum yield of 92.8 % biodiesel from bio-oil to be at 120 mins under stated reaction conditions.



**Figure 13: Effect of Reaction Time on Bio-diesel yield**

## 4.0 CONCLUSION

The *P. curatellifolia* seeds has been proven as a potential oil feedstock having high oil content of upto 36.2 % to be used for suitable sustainable biodiesel production for various energy application. The bio-oil synthesized was have high amount of acidic value, saponification value and FFAs and heterogeneous catalyst for was used for the transesterification process enhance conversion process to biodiesel. solid heterogeneous catalyst was design by direct incorporation of baking yeast with clay as catalyst for transesterification of PCSO. The catalyst exhibited good catalytic property, basic strength greater than 9.8 (pH>9.8), surface area of 378 m<sup>2</sup>g<sup>-1</sup> mesoporous catalyst with pore diameter 3.2 nm and crystalline phases. The favorable operating conditions for the production of the biodiesel were 5 % wt. catalyst loading, 120 mins (reaction time), 9:1 (methanol to bio-oil molar ratio) and 65 °C (reaction temperature) at 3000 rpm stirring rate and the maximum yield of PCUME was 94.7 %. The important fuel properties were compared to mineral diesel and global biodiesel standards such as ASTM D6751 testing limit and shows appreciable agreements which considered the PCSO as a potential viable feedstock for biodiesel production in commercial scale.

## REFERENCE

- Abbaszaadeh, A., Ghobadian, B., Omidkhah, M. R. and Najafi, G. (2012). Current biodiesel production technologies: A comparative review. *Energy Conversion and Management*. 63: 138–148. <https://doi.org/10.1016/j.enconman.2012.02.027>.
- Abdullah, S. H. Y. S., Hanapi, N. H. M., Azid, A., Umar, R., Juahir, H., Khattoon, H. And Endut, A. (2017). A review of biomass-derived heterogeneous catalyst for a sustainable biodiesel production. *Renewable and Sustainable Energy Reviews*. 70: 1040–1051. <https://doi.org/10.1016/j.rser.2016.12.008>.

- Abdulrahman, M. (2016). Determination of Iodine and Saponification Values of Silver Bird Eucalyptol. *IOSR Journal of Applied Chemistry*. 9(1): 86–89. <https://doi.org/10.9790/5736-09118689>.
- Adewale, P., Dumont, M. J. and Ngadi, M. (2015). Recent trends of biodiesel production from animal fat wastes and associated production techniques. *Renewable and Sustainable Energy Reviews*. 45: 574–588. <https://doi.org/10.1016/j.rser.2015.02.039>.
- Ahmad, A. L., Yasin, N. H. M., Derek, C. J. C. and Lim, J. K. (2011). Microalgae as a sustainable energy source for biodiesel production: A review. *Renewable and Sustainable Energy Reviews*. 15(1): 584–593. <https://doi.org/10.1016/j.rser.2010.09.018>.
- Ajayi, I. A., Oderinde, R. A., Kajogbola, D. O. and Uponi, J. I. (2006). Oil content and fatty acid composition of some underutilized legumes from Nigeria. *Food Chemistry*. 99(1): 115–120. <https://doi.org/10.1016/j.foodchem.2005.06.045>.
- Akhtar, T., Tariq, M. I., Iqbal, S., Sultana, N. and Wei, K. (2017). Production and Characterization of Biodiesel from Eriobotrya Japonica Seed Oil: an Optimization Study. *International Journal of Green Energy*. ISSN: 5075. <https://doi.org/10.1080/15435075.2017.1310107>.
- Algoufi, Y. T., Kabir, G. and Hameed, B. H. (2017). Synthesis of glycerol carbonate from biodiesel by-product glycerol over calcined dolomite. *Journal of the Taiwan Institute of Chemical Engineers*. 70: 179–187. <https://doi.org/10.1016/j.jtice.2016.10.039>.
- Ali, E. N. and Isis, C. (2013). Characterization of Biodiesel Produced from Palm Oil via Base Catalyzed Transesterification. *Procedia Engineering*. 53: 7–12. <https://doi.org/10.1016/j.proeng.2013.02.002>.
- Ali, Y., Hanna, M. A. and Cuppett, S. L. (1995a). Fuel properties of tallow and soybean oil esters. *Journal of the American Oil Chemists' Society*. 72(12): 1557–1564. <https://doi.org/10.1007/BF02577854>.
- ASTM, D. 16. (2016). Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (Calculation of Kinematic Viscosity). *American Standard of Testing and Materials (ASTM) International*. 1–13. <https://doi.org/10.1520/D7042-16E03>.
- ASTM D86-15. (2015). Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure. *American Standard of Testing and Materials (ASTM) International*. 05: 01: 1–27. <https://doi.org/10.1520/D0086-15>.
- ASTM D97-17B. (2009). Standard Test Method for pour point of petroleum Products. *American Standard of Testing and Materials (ASTM) International*. 1–7. <https://doi.org/10.1520/D0097-17A>.
- ASTM D2500-17. (2009). Standard Test Method for Petroleum Fuel Products. *American Standard of Testing and Materials (ASTM) International*. 1–5. <https://doi.org/10.1520/D2500-17.2>.
- Bazongo, P., Henri, I., Bassole, N., Nielsen, S., Dicko, M. H. and Shukla, V. K. S. (2014). Studies in the Evaluation of Unconventional Oils from Burkina Faso Rich in Linoleic Acid, Oleic Acid or Other Unusual Fatty Acids. *Food Processing and Technology*. 5(2): 2–5. <https://doi.org/10.4172/2157-7110.1000303>.
- Benhura, C., Benhura, M. A. N., Muchuweti, M., Nyagura, S. F. and Gombiro, P. E. (2012). Proximate analysis of *Parinaricuratellifolia* fruit pulp of fruit from parts of Harare and a rural area in Zimbabwe. *Pakistan Journal of Nutrition*. 11(7): 541.
- Berchmans, H. J. and Hirata, S. (2008). Biodiesel production from crude *Jatropha curcas* L. seed oil with a high content of free fatty acids. *Bioresource Technology*. 99(6): 1716–1721. <https://doi.org/10.1016/j.biortech.2007.03.051>.
- Berrios, M. and Skelton, R. L. (2008). Comparison of purification methods for biodiesel. *Chemical Engineering Journal*. 144: 459–465. <https://doi.org/10.1016/j.cej.2008.07.019>.
- Boro, J., Thakur, A. J. and Deka, D. (2011). Solid oxide derived from waste shells of *Turbonilla striatula* as a renewable catalyst for biodiesel production. *Fuel Processing Technology*. 92(10): 2061–2067. <https://doi.org/10.1016/j.fuproc.2011.06.008>.
- Burton, R., and Biofuels, P. (2008). An overview of *American Standard of Testing and Materials (ASTM) D6751*: biodiesel standards and testing methods. *Alternative fuels consortium*.
- Canakci, M. (2007). The potential of restaurant waste lipids as biodiesel feedstock. *Bioresource Technology*. 98(1): 183–190. <https://doi.org/10.1016/j.biortech.2005.11.022>.
- Canakci, M. and Van Gerpen, J. (2001). Biodiesel Production From Oils and Fats With High Free Fatty Acids. *Transactions of the American Society of Automotive Engineering*. 44(6): 1429–1436.
- Canakci, M. and Sanli, H. (2008). Biodiesel production from various feedstocks and their effects on the fuel properties. *Journal of Industrial Microbiology and Biotechnology*. 35(5): 431–441.
- Dehkhoda, A. M. and Ellis, N. (2013). Biochar-based catalyst for simultaneous reactions of esterification and

- transesterification. *Catalysis Today*. 207: 86–92. <https://doi.org/10.1016/j.cattod.2012.05.034>.
- Demirbaş, A. (2003). Chemical and fuel properties of seventeen vegetable oils. *Energy Sources*. 25(7): 721–728. <https://doi.org/10.1080/00908310390212426>.
- Demirbaş, A. (2005). Biodiesel fuels from vegetable oils via catalytic and non-catalytic supercritical alcohol transesterifications and other methods: A survey. *Energy Conversion and Management*. 44(13): 2093–2109. [https://doi.org/10.1016/S0196-8904\(02\)00234-0](https://doi.org/10.1016/S0196-8904(02)00234-0).
- Encinar, J. M., González, J. F., Rodríguez, J. J. and Tejedor, A. (2002). Biodiesel fuels from vegetable oils: Transesterification of *Cynara cardunculus* L. Oils with ethanol. *Energy and Fuels*. 16(2): 443–450. <https://doi.org/10.1021/ef010174h>.
- Endalew, A. K., Kiros, Y. and Zanzi, R. (2011). Heterogeneous catalysis for biodiesel production from *Jatropha curcas* oil (JCO). *Energy*. 36(5): 2693–2700. <https://doi.org/10.1016/j.energy.2011.02.010>.
- Jegannathan, K. R., Abang, S., Poncet, D., Chan, E. S. and Ravindra, P. (2008). Production of biodiesel using immobilized lipase - A critical review. *Critical Reviews in Biotechnology*. 28(4): 253–264. <https://doi.org/10.1080/07388550802428392>.
- Jenkins, B. M., Baxter, L. L., Miles, T. R. and Miles, T. R. (1998). Combustion properties of biomass. *Fuel Processing Technology*. 54(1–3): 17–46. [https://doi.org/10.1016/S0378-3820\(97\)00059-3](https://doi.org/10.1016/S0378-3820(97)00059-3).
- Jimenez-lopez, A., Jiménez-morales, I., Santamaría-gonzález, J. and Maireles-torres, P. (2011). Chemical Biodiesel production from sunflower oil by tungsten oxide supported on zirconium doped MCM-41 silica. *Journal of Molecular Catalysis*. 335: 205–209. <https://doi.org/10.1016/j.molcata.2010.11.035>.
- Kafuku, G. and Mbarawa, M. (2010). Biodiesel production from *Croton megalocarpus* oil and its process optimization. *Fuel*. 89(9): 2556–2560. <https://doi.org/10.1016/j.fuel.2010.03.039>.
- Kouzu, M., Kasuno, T., Tajika, M., Sugimoto, Y., Yamanaka, S. and Hidaka, J. (2008). Calcium oxide as a solid base catalyst for transesterification of soybean oil and its application to biodiesel production. *Fuel*. 87(12): 2798–2806. <https://doi.org/10.1016/j.fuel.2007.10.019>.
- Kozhevnikov, I. V. (2007). Sustainable heterogeneous acid catalysis by heteropoly acids. *Journal of Molecular Catalysis A: Chemical*. 262(1–2): 86–92. <https://doi.org/10.1016/j.molcata.2006.08.072>.
- Lee, A. F., Bennett, J. A., Manayil, J. C. and Wilson, K. (2014). Heterogeneous catalysis for sustainable biodiesel production via esterification and transesterification. *Chemical Society Reviews*. 43(22): 7887–7916. <https://doi.org/10.1039/c4cs00189c>.
- Lee, D. W., Park, Y. M. and Lee, K. Y. (2009). Heterogeneous base catalysts for transesterification in biodiesel synthesis. *Catalysis Surveys from Asia*. 13(2): 63–77. <https://doi.org/10.1007/s10563-009-9068-6>.
- Leung, D. Y. C. and Guo, Y. (2006). Transesterification of neat and used frying oil: Optimization for biodiesel production. *Fuel Processing Technology*. 87(10): 883–890. <https://doi.org/10.1016/j.fuproc.2006.06.003>.
- Leung, D. Y. C., Wu, X. and Leung, M. K. H. (2010). A review on biodiesel production using catalyzed transesterification. *Applied Energy*. 87(4): 1083–1095. <https://doi.org/10.1016/j.apenergy.2009.10.006>.
- Li, C., Hu, X., Feng, W., Wu, B. and Wu, K. (2018). A supported solid base catalyst synthesized from green biomass ash for biodiesel production. *Energy Sources, Part A: Recovery, Utilization and Environmental Effects*. 40(2): 142–147. <https://doi.org/10.1080/15567036.2017.1405121>.
- Lani, N. S., Ngadi, N. and Taib, M. R. (2017). Parametric Study on the Transesterification Reaction by Using CaO/Silica Catalyst. *Chemical Engineering Transactions*, 56: 601–606.
- Obadiah, A., Swaroopa, G. A., Kumar, S. V., Jegannathan, K. R. and Ramasubbu, A. (2012). Biodiesel production from Palm oil using calcined waste animal bone as catalyst. *Bioresource Technology*. 116: 512–516. <https://doi.org/10.1016/j.biortech.2012.03.112>.
- Oladimeji, A. O. and Bello, M. O. (2011). Proximate analysis and fatty-acid profiles of mobola plum seed. *Elixir Applied Chemistry*. 41: 5942–5943.
- Ong, H. C., Silitonga, A. S., Masjuki, H. H., Mahlia, T. M. I., Chong, W. T. and Boosroh, M. H. (2013). Production and comparative fuel properties of biodiesel from non-edible oils: *Jatropha curcas*, *Sterculia foetida* and *Ceiba pentandra*. *Energy Conversion and Management*. 73: 245 – 255. <https://doi.org/10.1016/j.enconman.2013.04.011>.
- Osakwe, E. U., Ani, I. J., Akpan, U. G. and Olutoye, M. A. (2018, July). Kolanut pod husk as a biobase catalyst for fatty acid methyl ester production using *Thevetia peruviana* (Yellow oleander) seed oil. In *IOP Conference Series: Earth and Environmental Science*. 173(1): 012–028). IOP Publishing.
- Rachmat, A., Trisunaryanti, W. and Wijaya, K. (2017). Synthesis and characterization of sulfated zirconia mesopore

and its application on lauric acid esterification. *Materials for Renewable and Sustainable Energy*. 6(3): 1-9. <https://doi.org/10.1007/s40243-017-0097-1>.

Talha, N. S. and Sulaiman, S. (2016). Overview of catalysts in biodiesel production. *ARPJ Journal of Engineering and Applied Sciences*. 11(1): 439–442.

Tang, Y., Xu, J., Zhang, J. and Lu, Y. (2013). Biodiesel production from vegetable oil by using modified CaO as solid basic catalysts. *Journal of Cleaner Production*. 42: 198–203. <https://doi.org/10.1016/j.jclepro.2012.11.001>.

Vicente, G., Martínez, M. and Aracil, J. (2007). Optimisation of integrated biodiesel production. Part I. A study of the biodiesel purity and yield. *Bioresource Technology*. 98(9): 1724–1733. <https://doi.org/10.1016/j.biortech.2006.07.024>.

Viriya-empikul, N., Krasae, P., Puttasawat, B., Yoosuk, B., Chollacoop, N. And Faungnawakij, K. (2010). Waste shells of mollusk and egg as biodiesel production catalysts. *Bioresource Technology*. 101(10): 3765–3767. <https://doi.org/10.1016/j.biortech.2009.12.079>.

Wadekaar, M. P., Rode, C. V., Bendale, Y. N., Patil, K. R., Gaikwad, A. B. and Prabhune, A. A. (2006). Effect of calcination cycles on the preparation of tin oxide based traditional drug: Studies on its formation and characterization. *Journal of Pharmaceutical and Biomedical Analysis*. 41(4): 1473–1478. <https://doi.org/10.1016/j.jpba.2006.03.032>.

Wei, Z., Xu, C. and Li, B. (2009). Bioresource Technology Application of waste eggshell as low-cost solid catalyst for biodiesel production. *Bioresource Technology*. 100(11):2883–2885. <https://doi.org/10.1016/j.biortech.2008.12.039>.