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**OPTIMIZATION OF BIODIESEL PRODUCTION FROM *Parinarium macrophyllum* SEED OIL USING POTASSIUM HYDROXIDE LOADED ON CALCIUM OXIDE CATALYST**

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**ABSTRACT**

The present study was aimed at optimization of biodiesel production from *Parinarium macrophyllum* seed oil by using potassium hydroxide loaded on calcium oxide and catalyst. The standard analytical methods of chemical analysis were use. A KOH/CaO catalyst was used as a solid base catalyst for biodiesel production via transesterification of *P. macrophyllum* seed oil using the vessel. The catalytic activity of KOH/CaO is associated with the active phase (K<sub>2</sub>O) and basic properties. The catalysts were characterized by using BET, and SEM. The highest methyl ester content of 80% was obtained at 25 wt% K loading on CaO, methanol-to-oil molar ratio of 7:1, 1.6 wt% of catalyst, and at a stirrer speed of 300 rpm. Some biodiesel properties were investigated in accordance with ASTM specifications. *P. macrophyllum* oil can be a potential feedstock for biodiesel production. Since the oil is non-edible and it will not interfere with the food chain. Modified CaO with potassium hydroxide showed high catalytic activity in a vessel, which produced a methyl ester content (over 80 %) under the certain conditions such as reaction temperature, reaction time, loading, methanol-to-oil molar ratio of catalyst, and stirrer speed.

**Keywords:** Biodiesel, *P. macrophyllum* oil, catalysis, transesterification, calcium oxide

**INTRODUCTION**

The continuous increase in prices of crude oil has a direct effect on many sectors of the economy, especially transportation. Finding an alternative source of energy is one of many ways to solve this serious problem. Biodiesel is a promising substitute for fossil diesel fuel



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because its properties are quite similar to diesel (Umar *et al.*, 2021). Moreover, the continuous increase in the use of petroleum has led to an increase in air pollution and accelerated global warming due to the increase in atmospheric CO<sub>2</sub>. Biodiesel is a relatively clean fuel and contains no sulphur compounds which can lead to negligible SO<sub>x</sub> production (Park *et al.*, 2018; Buasri *et al.*, 2016). Biodiesel is a methyl ester product that is derived from triglycerides in vegetable oil that are reacted with methanol via the transesterification reaction (Otori *et al.*, 2021).

Transesterification of *P. macrophyllum* to biodiesel can be catalyzed with base or acid catalysts; however, the rate of reaction in the presence of acid catalysts is very low. Most commercial biodiesels are produced from vegetable oils using effective homogeneous base catalysts such as NaOH or KOH, which are soluble in methanol. In order to purify the products, water is used to eliminate the used homogeneous catalysts, resulting in large amounts of contaminated water. This problem can be alleviated with the use of heterogeneous catalysts, resulting in the ease of separation of the catalyst from the product, thus, making the purification step easier. The cost of biodiesel can be reduced through the use of heterogeneous catalyst because there is no need for further expensive refining operations (Cerce *et al.*, 2015; Leung *et al.*, 2015). Considerable research is being devoted to the heterogeneous catalyzed methanolysis of vegetable oils. Various types of potassium precursors have been applied by using conventional impregnation method on Al<sub>2</sub>O<sub>3</sub> support (Ebiura *et al.*, 2014; Xie and Li, 2019). It should be noted that the type of base catalyst used has a significant effect on the conversion of biodiesel because its catalytic activity is related to the basicity of the catalyst. For example, when potassium is applied on clay (Ramos *et al.*, 2019), over 90% methyl ester content of biodiesel is obtained. Verziu *et al.* (2019) studied the activity of KF, LiF, and CsF/Al<sub>2</sub>O<sub>3</sub> catalysts with different loadings from 1 to 20 wt%. They found that the different types of alkaline, which deposited on the alumina surface



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generated fluoroaluminates and aluminate species, thus the oxygen became more basic, resulting in higher methyl ester content. It has been reported that CaO-supported catalysts have higher activities in the transesterification reaction than MgAl, and demonstrated high basic properties (Hamad *et al.*, 2016).

However, few studies have been carried out on CaO support in comparison to the Al<sub>2</sub>O<sub>3</sub> system as mentioned above. Therefore, CaO potential as a support base catalyst due to its basic features and thermal stability under high calcination temperatures (Hamad *et al.*, 2016). This study focused on the catalytic performance of KOH supported on CaO in a vessel under various conditions. The influence of conditions such as reaction time, potassium loading, methanol-to-oil molar ratio, and amount of catalyst on the methyl ester (ME) content was investigated. The fuel properties of the produced biodiesel were also examined.

### MATERIALS AND METHODS

The seed of *P. macrophyllum* used in this study were collected from different locations around Bida town in Niger State, Nigeria.

#### Sample Preparation and Oil Extraction

The seeds collected were cleaned by removing foreign materials such as insects, stains, leaves, sand and dirt. After cleaning, the seeds were dried in the oven at 300 °C for 72 hours until constant were obtained. The dried seeds were then mechanically dehulled to remove the seed coat. Removal of the seed coat is important because seed coat contain little or no oil and more importantly, inclusion would make extraction less efficient. The dehulled seeds were further dried at 400 °C for another 48 hours and ground to powder using pestle and mortar. The oil was extracted from *P. macrophyllum* seed using soxhlet extractor with petroleum ether as solvent. The percentage oil yield was determined.



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Physicochemical properties of the oil extracted were carried out according to Pisitpong *et al.* (2014).

### **Refining of *P. macrophyllum* Seed Oil**

10.0 g of the seed oil was dissolved in 25 cm<sup>3</sup> propanol, after stirring for 60 min. at 300 rpm, two drops of phenolphthalein were added. The solution was titrated against 2.0 M sodium hydroxide to pink colour. The acid value (free fatty acid) was calculated from the titre value. The oil was esterified with a mixture of hydrochloric acid and methanol (3:1). The mass of methanol used was calculated as 2.25 x % FFA (free fatty acid) x mass of oil and that of hydrochloric acid as 0.02 x % FFA x mass of oil. These were mixed and added to the oil at 60 °C. The temperature was maintained at 60 °C for 55 minutes. The product was transferred into a separating funnel and left to settle. The organic layer was collected at the bottom and tested for acid value again. The process was repeated, washed and dried until the acid value was reduced to 0.58 (Otori *et al.*, 2021).

### **Catalyst Preparation**

The KOH/CaO catalyst was prepared by the impregnation method. The CaO support was impregnated with an aqueous solution of KOH at various loadings of 10 to 30 wt%. The prepared catalysts were dried in an oven at 80 °C for 18 h and calcined at 300 °C for 2 h (Rachim *et al.*, 2017).

### **Catalyst Characterization**

SEM (scanning electron microscope) was used to identify the microstructure and capture micrographs of the catalyst (KOH/CaO). Also, the specific surface area was determined by the BET (Brunauer-Emmet-Teller)



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### **Transesterification**

120 cm<sup>3</sup> of the refined oil was heated to 65 °C, a mixture of 17.5 g methanol and 0.6 g modified catalyst (KOH/CaO) were added and the temperature was maintained at 65 °C for 70 minutes. The reaction continued until it reached the desired reaction time. The reaction was stopped by instant cooling, and the catalysts were separated from the product mixture using suction flask. The biodiesel (top phase) was separated from the glycerol (bottom phase) using a separatory funnel. The procedure was repeated with 0.8, 1.0 and 1.2 g of the modified catalyst keeping other parameters constant (Umar *et al.*, 2021).

### **Determination of peroxide value**

The sample (2 g) was weighed into a conical flask. 2 g of powdered potassium iodide (KI) and 40 cm<sup>3</sup> of solvent mixture (2 volumes of glacial acetic acid + 1 volume chloroform) was subsequently added to the conical flask. The mixture was shaken vigorously and allowed to stand in the boiling water for 60 sec. and immediately poured into another flask containing 60 cm<sup>3</sup> freshly prepared 5 % potassium iodide solution. The previous conical flask was rinsed twice with 30 cm<sup>3</sup> of distilled water and poured into the flask. The mixture was titrated with 0.005 M sodium thiosulphate solution using starch indicator. The above procedure was repeated for the blank in the absence of the sample (Jaimasith and Phiyalaninmat, 2017).

The peroxide value (PV) is given by:

$$PV = \frac{2(a - b)}{\text{Weight of oil}}$$

Where a = titre value of sample;      b = titre value of blank.



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### **Determination of iodine value**

1.0 g of oil was weighed into a conical flask and 15 cm<sup>3</sup> of trichloro-methane was added to dissolve the oil. 30 cm<sup>3</sup> of wj solution was subsequently added to the conical of 250 cm<sup>3</sup> flask and covered tightly in the dark for 45 min. 30 cm<sup>3</sup> of potassium iodide solution (10 %) and 150 cm<sup>3</sup> of water were added to the flask, the solution turned red colour. This was titrated using a 0.1N solution of sodium thiosulphate until the reddish colour separates of the solution. 10 cm<sup>3</sup> of the starch indicator was added and a blue coloration appeared. The titration continued until the blue coloration disappeared. A blank titration was carried out and the Iodine value was calculated according to the recommendation of AOAC (2009).

The Iodine value (IV) is given by the expression:

$$IV = \frac{12.69 \times M \times (v_2 - v_1)}{\text{Weight of sample}}$$

$v_2$  = volume of sodium thiosulphate used for blank;

$v_1$  = volume of sodium thiosulphate used for determination

M = Molarity of sodium thiosulphate solution

12.69 = atomic weight of iodine.

### **Viscosity**

100 cm<sup>3</sup> beaker was filled to three-quarters of its capacity with biodiesel sample, heated to 40 °C and immediately placed on viscometer and a spindle of a viscometer was adjusted until it submerged into the sample. The viscometer was switched on after spinning; reading was taken from the scale (Umar *et al.*, 2021).



### Optimization Parameters

Transesterification of oil or fat to produce a high yield biodiesel was determined by optimizing alcohol/oil molar ratio, catalyst concentration, reaction temperature, and reaction time (Zhang and Meng, 2014).

### Characterization of Biodiesel

Biodiesel properties such as density, cetane number, cloud point, flash point, pour point, refractive index were determined by the standard method described by ASTM D6751.

## RESULTS AND DISCUSSION

### Result of Physicochemical Properties

**Table 1: Result of Physicochemical Properties**

S/No.	Properties	Values
1	Yield (%)	35.0
2	Moisture content (%)	4.3
3	Specific gravity	0.79
4	pH	4.3
5	Iodine value (g I <sub>2</sub> /100 g)	102
6	Saponification value (mg KOH/g)	121
7	Colour	Brown

The results of the physicochemical properties of the oil seeds of *P. macrophyllum* are presented in Table 1. Percent oil yield was 35.0% and is within the range of (25 – 55%) as reported by Mohamad *et al.* (2013) for *Gmelina arborea*. Value obtained for yield indicate that the oil seed of *P. macrophyllum* contain appreciable quantity of oil that is enough to be extracted for the commercial production of biodiesel. pH value of the oil presented in Table 1 was 4.3. This value is within the range according to Codex, (2011) for biodiesel



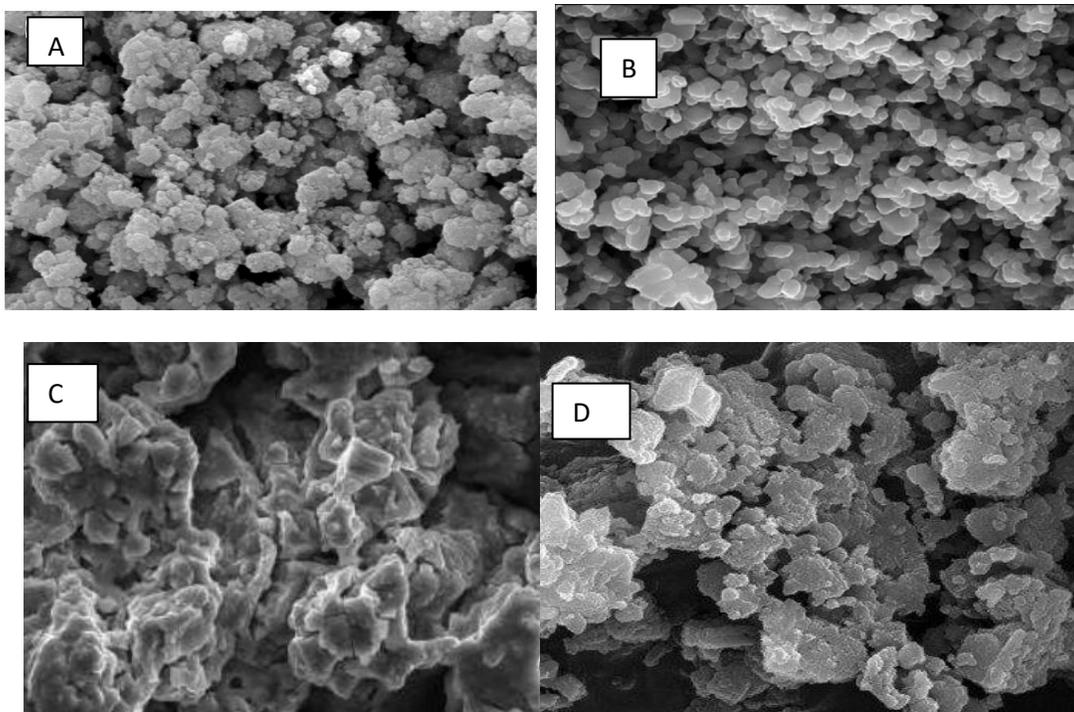
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production. The pH for this oil was less than seven and is due to the presence of fatty acid content (Lamani et al., 2021). The colour of the extracted *P. macrophyllum* oil was brown. This colour is acceptable for biodiesel according to Codex (2011). Zhang and Meng (2014) reported brown colour for *Jatropha* oil used for the production of biodiesel, Sanjay (2013) reported brown yellow colour for neem oil used for the production of biodiesel. These colours are accepted by Codex (2011) Standard for biodiesel production.

Iodine value is a measure of the degree of unsaturation and it is an identifying characteristic of oil seed making it an excellent raw material for biodiesel production. The iodine value for *P. macrophyllum* oil was 102 g/100g. Oils with iodine values less than 100I<sub>2</sub>/100g of oil are non-drying oils. Aremu *et al.* (2015) reported that oil with low iodine value would have a low number of unsaturated bonds.

**Table 2: Brunauer Emmett and Teller Analysis of the Catalyst**

Properties	Catalyst
Pore size (A)	71.52
Pore volume (cm <sup>3</sup> /g)	12.61
Specific surface area (m <sup>2</sup> /g)	12.10



**Plate I:** SEM micrographs of (a) CaO, (b) 10 wt % KOH/CaO, (c) 20 wt % KOH/CaO, (d) 30 wt % KOH/CaO

The BET surface area of CaO was  $12.10 \text{ m}^2/\text{g}$  as shown in Table 2. With an increase of potassium content from 10 to 40 wt %, the surface area decreased to  $6.9 \text{ m}^2/\text{g}$  because the crystallites of the modified CaO were larger than that of the unmodified CaO . Interestingly, despite the comparatively low surface area of the catalyst when potassium was loaded, high methyl ester content was obtained. This result corresponds with the results of Atadashi *et al.* (2012) who that lead oxides with low surface area ( $0.98 \text{ m}^2/\text{g}$ ) exhibited high methyl ester content (Babel *et al.* 2016). It could be suggested that the methyl ester content of biodiesel does not always relate to the large surface area of the catalyst.

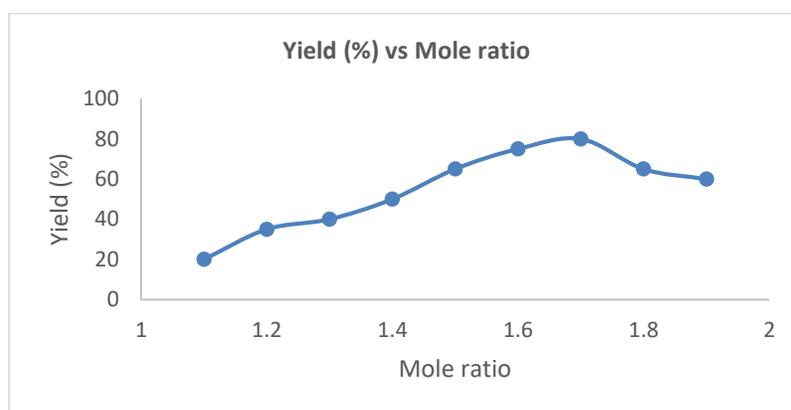
Scanning electron microscopy has been used extensively for the examination of the topology of the catalyst surfaces. Plate 1a and 1b shows the SEM pictures of CaO and



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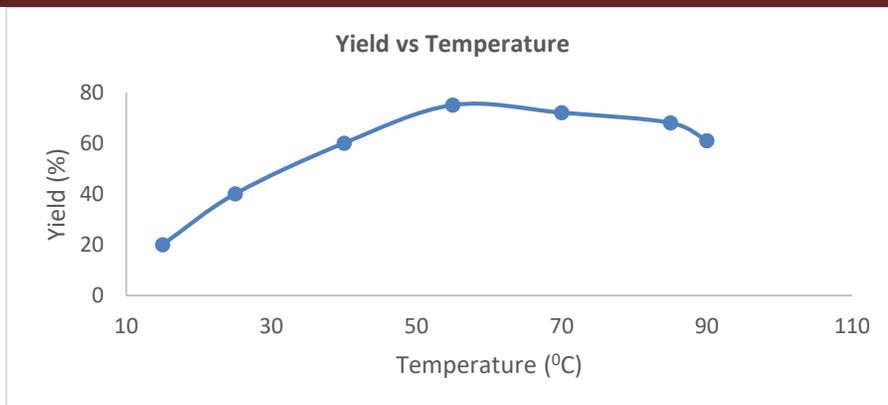
KOH/CaO catalysts. The particles of fresh CaO are agglomerated and spherical in shape with an average size of 5 to 15  $\mu\text{m}$ . The modified CaO catalysts show the agglomeration of small particles with increasing K loading (Plates 1b-d); however, the morphology of KOH/CaO is similar to the CaO sample. When K loading was increased from 10 to 20 wt%, the particle sizes were increased from 10 to 100  $\mu\text{m}$ .

### Optimized Parameters



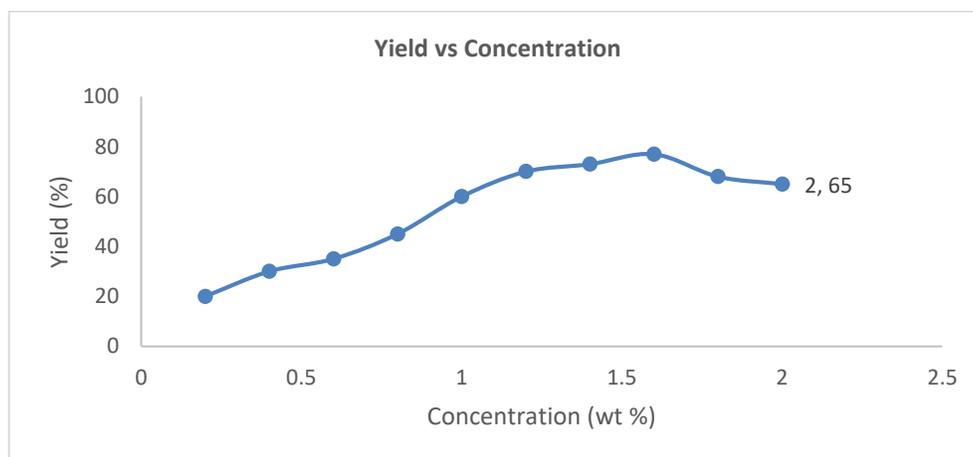
**Figure I: Methanol to Oil ratio**

Figure I shows the result of methanol to oil ratio, it can be observed that the more preferable ratio for KOH/CaO, methanol to oil ratio is 7:1 gives the maximum biodiesel yield at 80.0%.



**Figure II: Yield against Temperature (°C)**

The reaction and yield of biodiesel are strongly influenced by the reaction temperature used. The effect of varying the temperature from 15 – 90 °C was studied. From Figure II the maximum percentage yield of biodiesel obtained at 55 °C is 75.02 % from sodium hydroxide.



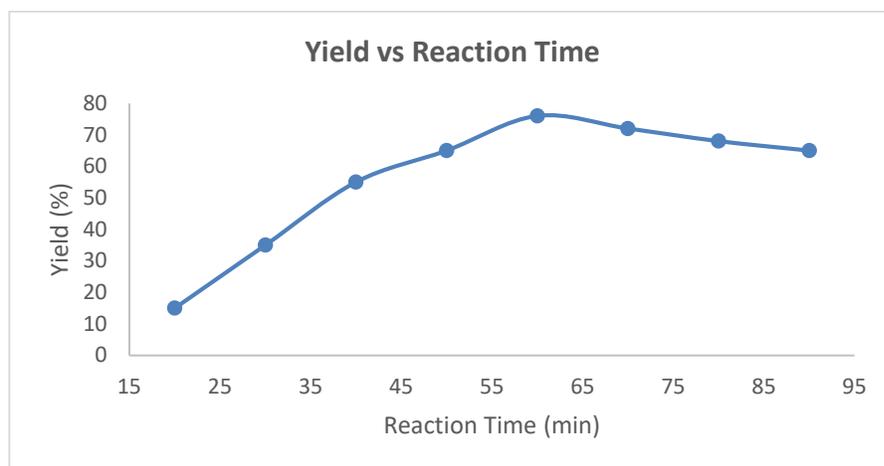
**Figure III: Catalyst Concentration (wt %)**

Concentration of the catalyst is an important parameter of transesterification reaction and a strong influence on the yield of biodiesel. Excess catalyst reacted with the oil, leading to



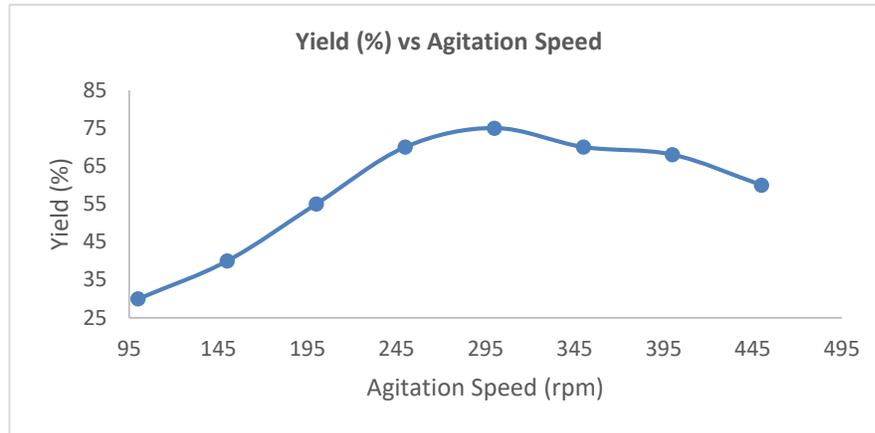
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the formation of soap, thus as the catalyst concentration increased, the separation of esters became difficult (Babajide *et al.*, 2013). The most suitable catalyst for this process proved to be potassium hydroxide. The basic catalyst, potassium hydroxide, achieved similar results but its methyl esters contents were slightly lower. The optimum concentration of potassium methoxide catalyst is 1.6 wt. % as shown in Figure III. A catalyst is used to hasten up the process and sodium hydroxide (NaOH) and potassium hydroxide (KOH) are the common catalysts used in the reaction process (Ojiego *et al.*, 2014). High concentrations of alkaline catalyst form soaps in the presence of large residues of fatty acids resulting in emulsion formation between soaps and water molecules, thus leading to low yields of methyl esters (Babajide *et al.*, 2013).



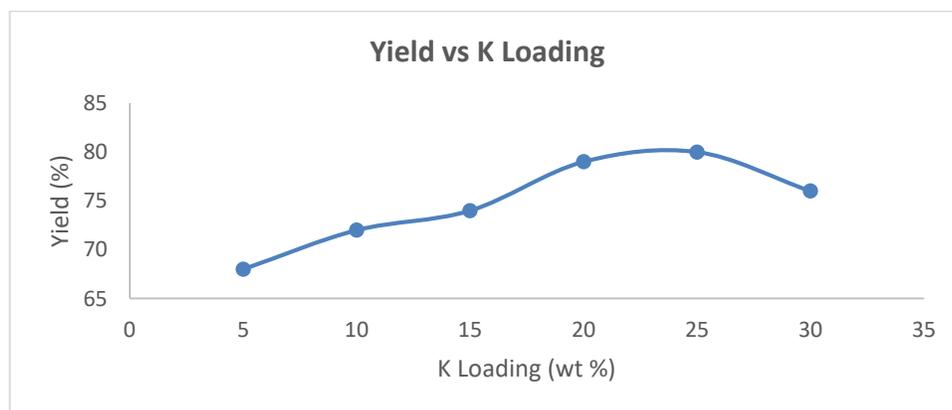
**Figure IV: Reaction Time (min)**

Methanol to oil molar ratio used was found to be the maximum yield found from this study. The reaction time is at 60 min and the result for this study is illustrated in Figure IV. Generally, the formation of biodiesel increases with temperature. However, increasing the temperature beyond the methanol boiling point may burn the methanol and influence the saponification reaction of triglycerides and reduced the biodiesel yield (Noiroj *et al.*, 2019).



**Figure V: Yield against Agitation Speed**

Most literatures indicate that during the transesterification reaction, reactants initially form a two-phase liquid system. The mixing effect has been found to play a significant role in the slow rate of the reaction. As phase separation ceases, mixing becomes insignificant. The effect of mixing on the kinetics of the transesterification process forms the basis for process scale-up and design (Kyari, 2015). In this study, the agitation speed for the produced biodiesel is at 300 rpm as shown in Figure V.



**Figure VI: Yield against K loading**



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The amount of K on the CaO support was varied from 5 to 30 wt %. The results presented in Figure VII showed that by increasing K loading on CaO from 5 to 20 wt %, the biodiesel yield increased. When K loading on CaO exceeds 25wt %, the yield is slightly decreased. As observed from the characterizations, when increasing the K loading to 25wt%, it was found that a phase of K<sub>2</sub>O was observed and the highest basic strength and basicity were obtained, causing high catalytic activity. When increasing K loading over 25 wt%, the yield decreased. Because of the excess KOH, some parts of KOH could not decompose to K<sub>2</sub>O, resulting in less basicity.

**Table 2: Fuel Properties of Biodiesel Production**

<b>Biodiesel properties</b>	<b><i>P. microphyllum</i> seed oil</b>	<b>ASTM Standard</b>
<b>Density g/cm<sup>3</sup></b>	0.83	0.75 – 0.84
<b>Cetane number</b>	46	48 – 65
<b>Cloud point (°C)</b>	-11.2	-13 – 12
<b>Flash point (°C)</b>	125	100 – 170
<b>Pour point</b>	-4.5	-1.5 – 10

Density is a key fuel property which directly affects the engine performance. It affects the mass of fuel injected into the combustion chamber and thus, the air-fuel ratio. This is because fuel injection pumps meter fuel by volume, not by mass and a denser fuel contains a greater mass in the same volume. Thus, the changes in the fuel density will influence engine power output due to a different mass of fuel injected. It is known that biodiesel density mainly depends on its esters content and the remained quantity of alcohol; hence this property is influenced primarily by the choice of vegetable oil (Encinar *et al.*, 2015)



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density is usually measured at 15 °C. The density of biodiesel is the weight of a unit volume of fluid while the specific gravity is the ratio of the density of a liquid to the density of water. The fuel injection equipment meters the fuel volumetrically and high densities translate into a high consumption of the fuel. It can be seen that the produced biodiesel has density of 0.83 g/cm<sup>3</sup> which is within

American Society for Testing and Materials (ASTM) standard for biodiesel production (0.75 g/cm<sup>3</sup> and 0.84 g/cm<sup>3</sup>) (Alnuami *et al.*, 2014).

Flash point of fuel indicates the minimum temperature at which the fuel will ignite (flash) on application of an ignition source under specified conditions. Flash point varies inversely with the fuel's volatility. Flash point minimum temperatures are required for proper safety and handling of fuels. It is noted that the biodiesel component must meet a flash point criteria, prior to blending, for the purpose of assuring that the biodiesel component does not contain methanol. The flash point of biodiesel is higher than the petro diesel, which is safe for transport purposes. High values of flash point decrease the risk of fire (Sanjay, 2013; Vuppaladadiyam *et al.*, 2013). The flash point of the produced biodiesel is 125 °C which is within the ASTM standard.

Cloud point and Pour point; the two most important criteria are the cloud and pour points. The cloud point is the temperature at which the fuel starts to form crystals, with further decrease in temperature these crystals increase in size and quantity until the fuel gels and does not move again. The pour point is the lowest temperature at which the oil specimen will flow. Both parameters are often used to specify the cold temperature usability of fuel oils. The cloud and pour point of the produced biodiesel is -11.2 °C and -4.5 respectively. However, the values were low compared to -0.93 °C and -0.67 recorded for soyabeans oil obtained for cloud and pour point of the produced biodiesel by Anup and Dev, (2007).



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The cetane number of a fuel is a measure of the ignition quality of the fuel, the higher the cetane number the better the ignition quality, which is conceptually similar to the octane number used for gasoline. Generally, a compound that has a high octane number tends to have a low cetane number and vice versa. The cetane number measures how easily ignition occurs and the smoothness of combustion. Higher is the cetane number better it is in its ignition properties. For this study, the cetane number is 46. Which is acceptable since is within the ASTM standard (Knothe *et al.*, 2013).

### CONCLUSION

In conclusion, modified CaO with potassium hydroxide showed high catalytic activity in a vessel, which produced a methyl ester content (over 80 %) under the following conditions: reaction temperature of 55 °C, 1 h of reaction time, 25 wt % K loading, methanol-to-oil molar ratio of 7:1, 1.6 wt% of catalyst, and at a stirrer speed of 300 rpm. Thermal decomposition of KOH to K<sub>2</sub>O and an increase in basic properties enhanced the catalytic activity of the modified catalysts in the transesterification reaction. The fuel properties of biodiesel from *P. macrophyllum* seed oil with 25 wt% KOH/CaO satisfied the values required by the EU standard. Consequently, KOH/CaO could be considered an attractive catalyst for biodiesel production.

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