Optimization of biodiesel production and engine performance from high free fatty acid *Calophyllum inophyllum* oil in CI diesel engine

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**A R T I C L E   I N F O**

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**A B S T R A C T**

In the present study, crude *Calophyllum inophyllum* oil (CCIO) has been evaluated as a potential feedstock for biodiesel production. *C. inophyllum* oil has high acid value which is 59.30 mg KOH/g. Therefore, the degumming, esterification, neutralization and transesterification process are carried out to reduce the acid value to 0.34 mg KOH/g. The optimum yield was obtained at 9:1 methanol to oil ratio with 1 wt.% NaOH catalyst at 50 °C for 2 h. On the other hand, the *C. inophyllum* biodiesel properties fulfilled the specification of ASTM D6751 and EN 14214 biodiesel standards. After that, the *C. inophyllum* biodiesel blends were tested to evaluate the engine performance and emission characteristic. The performance and emission of 10% *C. inophyllum* biodiesel blends (CIB10) give a satisfactory result in diesel engines as the brake thermal increase 2.30% and fuel consumption decrease 3.06% compared to diesel. Besides, CIB10 reduces CO and smoke opacity compared to diesel. In short, *C. inophyllum* biodiesel can become an alternative fuel in the future.

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1. Introduction

The fossil fuel resources are dwindling day by day. Biodiesel is one of the possible solutions to overcome oil shortage and environmental issue [1]. Biodiesel have low volatility due to high molecular weight of the triglyceride molecule and have a narrow range of viscosity changes with temperature [2]. Biodiesel is a clean burning alternative fuel was produced from renewable resources like virgin and used vegetable oils, both edible and non-edible [3,4]. Biodiesel is obtained from the chemical transformation of oils (triglycerides) by a transesterification process and many oleaginous vegetable species [5]. However, biodiesel produced from edible oils raised the concerns of feedstock competing with food supply in the long-term [6]. Therefore, non-edible oils resources are gaining worldwide attention as it is easily available in many parts of the world especially wastelands that are not suitable for food crops [7]. Besides, the use of biodiesel in diesel engines results in substantial reduction of harmful emission such as unburned hydrocarbons, carbon monoxide and particulate matters [8,9].

There are many ways and procedures to biodiesel fuel from vegetable oil such as pyrolysis, dilution, microemulsion and transesterification [10]. The transesterification process is the most viable and economical process [11]. Transesterification process is using an alcohol (methanol, ethanol or propanol) with the presence of alkali catalyst (sodium hydroxide or potassium hydroxide) to break the molecule of the vegetable oil into methyl or ethyl esters chemically with glycerol as a byproduct [12]. Biodiesel produced via transesterification process has proven to be a viable alternative fuel with similar characteristics to diesel fuel [13]. Besides, some researchers are using two-step esterification and transesterification processes in producing biodiesel from crude oils with high free fatty acid (FFA) [14]. Dhar et al. [15] reported that high FFA of neem oil (20.3%) was converted to methyl ester using two-step esterification and transesterification process. As a result, the important properties of the biodiesel were within the ASTM biodiesel specification. Jena et al. [16] investigated that acid pretreatment and base transesterification reaction process

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**Abbreviations:** ASTM, American society for testing and materials; Bsfc, brake specific fuel consumption; BTE, brake thermal efficiency; CCIO, crude *Calophyllum inophyllum* oil; CI, compression ignition; CIB, *Calophyllum inophyllum* biodiesel blends; CIB10, *Calophyllum inophyllum* biodiesel blending 10%; CIB20, *Calophyllum inophyllum* biodiesel blending 20%; CIB30, *Calophyllum inophyllum* biodiesel blending 30%; CIB50, *Calophyllum inophyllum* biodiesel blending 50%; CIME, Calophyllum *inophyllum* methyl ester; CO, carbon monoxide; DOE, design of experiment; EN, European standard; EGT, exhaust gas temperature; FFA, free fatty acid; FAME, fatty acid methyl ester; H2SO4 sulfuric acid; H3PO4 phosphoric acid; HSU, Hartridge smoke units; NaOH, sodium hydroxide; NaHCO3, sodium bicarbonate; NOx nitrogen oxide; ppm, parts per million.

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is suitable for biodiesel production from mixture of mahua and simarouba oils with high FFA. The yield of biodiesel and ester conversion was around 98% and 90% respectively.

In this study, non-edible “Calophyllum inophyllum” oil was investigated as a potential feedstock for biodiesel production. *C. inophyllum* is a multipurpose tree belonging to the family Clusiaceae, commonly known as mangosteen family [17]. This plant has multiple origins including East Africa, India, South East Asia, Australia, and the South Pacific [18]. Fig. 1 shows the distribution map of *C. inophyllum* around the world. It grows in areas with an annual rain of 1000–5000 mm at altitudes from 0 to 200 m. *C. inophyllum* is a low-branching and slow-growing tree which grows best in sandy and well drained soils. Its sizes typically ranges between 8 and 20 m (25–65 ft) tall at maturity, sometimes reaching up to 35 m (115 ft) [19]. In each fruit, it consists one large brown seed with 2–4 cm (0.8–1.6 in.) in diameter [20]. *C. inophyllum* tree can be planted at a density of 400 tree/ha with an average oil yield of 11.7 kg/tree or 4680 kg/ha [11]. Traditionally, its oil has been used as a medicine, soap, lamp oil, hair grease and cosmetic in different parts of the world [21]. Fig. 2 shows *C. inophyllum* tree, fruit, seeds and kernels.

The objective of this study is to produce biodiesel from crude *C. inophyllum* oil using H2SO4 as acid catalyst and NaOH as alkaline catalyst. After that, the optimization of *C. inophyllum* methyl ester production was studied and its properties as biofuel were analyzed based on ASTM D6751 and EN 14214 biodiesel standards. On top of that, a *C. inophyllum* biodiesel diesel blend properties were tested and the performance and emission characteristic were conducted in a direct injection diesel engine. It is notable that this study suggests a novel alternative biodiesel feedstock as energy source in compression ignition engine.

## 2. Methodology

### 2.1. Extraction of *C. inophyllum* seed oil

*C. inophyllum* seeds were collected from Kebumen, Center Java, Indonesia. After that, the seeds were dried under sunlight until the color of the seeds turn into red-brown. The ideal conditions to preserve the kernel are 26–27 ℃ and 60–70% humidity. Hydraulic machine is used to extract the oil from *C. inophyllum* kernel seed. The remaining seed cake after extraction has high commercial value and can be used either for agricultural or industrial applications. The *C. inophyllum* oil extracted was found to contain many impurities and chemical compound. Therefore, it is important to undergo a degumming process before converting crude *C. inophyllum* oil to biodiesel. The flow chart shown in Fig. 3 is the methodology applied for *C. inophyllum* biodiesel production process.

### 2.2. Degumming process

Gum contains phosphate, protein, carbohydrate, water residue and resin. In order to improve the oxidization stability of the final product, the oil is separated from the gums through the degumming process. In this process, the crude oil was heated at a temperature of 60 ℃ and stirring speed of 1000 rpm. Then, 0.5 vol.% of phosphoric acid (H3PO4, 20% concentration) was added to the preheated crude oil. The process was continued with stirring and the temperature maintained at 60 ℃ for 30 min. After that, this mixture was separated by density separation process using a separating funnel for at least 4 h in which the phosphate compounds resided at the bottom. These gums were separated from the oil and washed several times with distilled water at 40 ℃. After washing, water was evaporated with vacuum pump for 30 min to avoid the oxidization of oil.

### 2.3. Esterification process

The degummed *C. inophyllum* oil has high content of FFA and viscosity. The maximum limit of FFA amount is 2 wt.% and below. Therefore, a pretreatment process using acid catalyzed esterification is required for the crude oil with high FFA content before the transesterification process. Thus, sulfuric acid (H2SO4) is used to convert and reduce the FFA content to below 2 wt.% in the oil. Therefore, two step processes of acid catalyzed esterification...
process and base catalyzed transesterification process are used to produce the biodiesel from C. inophyllum oil with high FFA. On top of that, crude C. inophyllum oil was transferred into a preheated reactor at a temperature of 60 °C. The methanol (9:1 methanol to oil ratio) and H₂SO₄ catalyst (1 vol.%) were measured and mixed together before added into the reactor. The mixture was stirred constantly using an overhead stirrer with a constant speed of 1150 rpm for 3 h. Throughout this process, the temperature was kept constant at 60 °C. After esterification process, the sample oil was transferred into a separation funnel for 4 h to remove the water and extra methanol. The upper layer is esterified oil while the water and extra methanol were at the lower layer. After that,
the esterified oil was washed by using distilled water at 40 °C. Finally, the esterified oil was evaporated with a rotary evaporator at 65 °C for 30 min to remove residual methanol and water.

2.4. Neutralization

Neutralization process is to purify the gum and organic particles present in the esterified oils. It is observed that after esterification process, acid value of the oil is still high which is about 5–10 mg KOH/g. Thus, neutralization is carried out to reduce the FFA content. In the course of preliminary tests, the esterified oil was poured into double jacketed equipped with a motor stirrer, reflux condenser and thermometer respectively. Then, 0.23 wt.% sodium bicarbonate (NaHCO₃) is diluted to 50 ml methanol while stirring and added to the esterified oil. In this process, the reaction was maintained at 50 °C for 30 min. The esterified oil was removed from the reactor and entered into a separation funnel and left for 30 min to remove extra methanol at lower layer.

2.5. Transesterification

After esterification process, the FFA content is reduced below 2%. Then, the esterified C. inophyllum oil was measured and placed into a jacketed reactor. The esterified C. inophyllum oil was pre-heated to the temperature of 50 °C by using a heating circulator. Then 1 wt.% of alkali catalyst (NaOH) and 9:1 of methanol to oil ratio are mixed until all the NaOH has been dissolved. After that, the prepared mixtures of methanol and NaOH were added into the pre-heated crude oil. The mixture was stirred constantly at 1150 rpm by an overhead stirrer during the transesterification process for 2 h. In this process, the temperature was maintained at 50 °C.

2.6. Purification and drying

After phase separation of FAME and glycerin, the FAME was purified and washed gently with distilled water at 40 °C in order to remove impurities. The mixture was allowed to settle under gravity for 2–3 h in a separating funnel. The lower layer consists of impurities were drained out. Finally, the product was evaporated with rotary evaporator at 65 °C for 30 min to remove residual methanol and water.

2.7. Optimization of biodiesel production

The design of experiment (DOE) is used to determine the optimization condition of transesterification process. This method was conducted to determine the optimum condition for methanol to oil molar ratio, catalyst concentration, reaction temperature and time as well as agitation intensity speed.

2.8. Biodiesel diesel blends

In this study, C. inophyllum biodiesel was blended with diesel at four different ratios which are 10%, 20%, 30% and 50% of biodiesel at volume basic. The biodiesel blends are used to examine the effect of blending on physicochemical properties and further study in engine performance and emission characteristic.

2.9. Experimental setup and procedure for engine test

The engine speed varies from 1500 to 2400 rpm with 100 rpm interval range. A SAJ SE-20 Eddy current dynamometer and Auto-ETS 1 controller system was used to determine the engine performance and speed. The experimental setup and specifications of the engine, dynamometer and gas analyzer used were shown in Fig. 4 and Table 1. A BOSCH BEA 150 gas analyzer is used to analyze the exhaust emissions from the engine such as carbon monoxide (CO), oxides of nitrogen (NOₓ) and smoke opacity. Each experiment was repeated for three times and the mean value was calculated for each sample. The errors and uncertainties of instruments and operating condition are listed in Table 2. The experiment was conducted in Heat Engine Laboratory, Department of Mechanical Engineering, Faculty of Engineering, University of Malaya.

3. Results and discussion

3.1. Properties of crude C. inophyllum oil and biodiesel

The crude C. inophyllum oil is dark green color. In this study, the fatty acid composition of crude C. inophyllum oil and physicochemical properties are analyzed and shown in Table 3. The crude C. inophyllum oil contains higher amount of unsaturated fatty acids (oleic and linoleic) than saturated fatty acids (palmitic and stearic). The comparison of fatty acid composition crude C. inophyllum oil with other non-edible feedstock is shown in Fig. 5. Crude C. eleganis C. inophyllum oil contains gum such as phosphate, protein, carbohydrate, water residue and resin. Therefore, degumming process is required to separate oil from the gums in order to improve the oxidation stability of the oil. It is shown that density, viscosity, acid value and FFA value decreased after degumming process. Apart from that, transmission has increased and absorbance has decreased which attributed to the removal of gum from the oil. Table 4 shows the physicochemical properties results of produced C. inophyllum methyl ester (CIME) and compared with ASTM D6751 and EN 14214 biodiesel standards. All specified properties from CIME are in acceptable ranges according to ASTM D6751 and EN 14214 standards. The kinematic viscosity of CIME is 3.45 mm²/s which was lower than Polanga biodiesel of 3.99 mm²/s reported by Sahoo et al. [22]. The density of CIME obtained was 877.6 kg/m³. The density of fuel affects the exhaust emissions in which high density fuel will cause an increase in particular matter and NOₓ emission [23]. Generally, biodiesel fuel has slightly higher density than diesel fuel. The obtained calorific value for CIME was 41.442 MJ/kg and this value is slightly higher than polanga biodiesel which is recorded to be 41.397 MJ/kg [22]. The cold filter plugging point, cloud point and pour point for CIME are 0.0 °C, 2.0 °C and 2.0 °C respectively. It is observed that the cold filter plugging point, cloud point and pour point of CIME satisfies the biodiesel standards. Apart from that, the physicochemical properties of CIME blends with diesel fuel are summarized in Table 5. The kinematic viscosity of C. inophyllum blends (CIB) increases with the rising of biodiesel blending ratio. The blending ratio values are 2.93 mm²/s (CIB10), 3.22 mm²/s (CIB20), 3.29 mm²/s (CIB30) and 3.35 mm²/s (CIB50) which satisfies the biodiesel diesel blends (ASTM D7467) standards. The calorific value of CIB ranges from 40.172 MJ/kg to 42.542 MJ/kg. This was also reported in Sahoo and Das [14] which the viscosity and calorific value of polanga blends (PB50, PB60 and PB80) were within the range of 3.42–3.72 mm²/s and 41.397–42.542 MJ/kg respectively. Besides, flash points measured met the safety requirements for fuel handling and storage. The observed flash point for CIB10, CIB20, CIB30 and CIB50 were 77.5 °C, 79.5 °C, 82.5 °C and 83.5 °C respectively. Generally, the flash point of biodiesel diesel blends is higher than the diesel which is safe for transport and storage purpose.

3.2. Effect of biodiesel properties on combustion characteristics

Typically, non-edible oil has high percentage of double carbon chain and has a greater degree of unsaturated fatty acid than saturated fatty acid [24]. As a result, this structural fatty acid composition will influence the physicochemical properties of biodiesel
such as viscosity, density and calorific value [23,25]. Kinematic viscosity will affect the operation of fuel injection of engine particularly at low temperatures when viscosity increases and affects the fluidity of the fuel [26]. Moreover, high viscosity of biodiesel will cause the insufficient fuel atomization which lead to formation of engine deposits and soot. Besides, density also will influence the efficiency of the fuel atomization for combustion systems [27,28]. The viscosity and density of CIME are 3.45 mm²/s and 877.6 kg/m³.
and those values were close to diesel fuel (2.95 mm$^2$/s and 840.0 kg/m$^3$) which give advantages to engine combustion and performance. Besides, the cetane number is a measure of the ignition quality of diesel fuel during combustion ignition [28]. It may be used in combustion and is possible reason to complete combustion [29,30]. On other hand, it provides information about the ignition delay time of a diesel fuel upon injection into the combustion chamber and high cetane implies short ignition delay [31]. Generally, the cetane number of biodiesel is higher than diesel fuels [25].

The cetane number for CIME (59.5) and CIB10 (52.6), CIB20 (53.7), CIB30 (55.5) and CIB50 (56.5) were higher than diesel fuel (49.7). Fuels with low cetane number tend to cause the knocking and incomplete combustion which shows increased gaseous and particulate exhaust emissions. Moreover, combustion of fuel containing sulfur causes sulfur oxides emissions. Most of the vegetable oils and animal fat-based biodiesels have very low levels of sulfur content including C. inophyllum biodiesel [10,32].

### 3.3. Optimization of C. inophyllum biodiesel

#### 3.3.1. Effect of methanol to oil molar ratio and temperature

In Stoichiometry, the transesterification reaction requires 3 mol of alcohol with a mole of triglyceride to produce 3 mol of fatty esters and 1 mol of glycerol [33]. However, it is necessary to use excess alcohol from the reaction mixture in order to shift the reaction to the right. The surface contours of ester yield versus molar ratio and temperature are plotted in Fig. 6. The methanol to oil molar ratio varied within the range of 3:1–15:1 for C. inophyllum oil and temperature was varied from 40 to 60 °C. A comparison among these plots shows that the maximum yield of ester is achieved at 9:1 and 50 °C for methanol to oil molar ratio and temperature respectively. The maximum methyl ester yield was found to be 98.92% and the increase in methanol ratio after 9:1 M ratio did not affect the methyl ester yield. Besides, it is showed that temperature reaction above 50 °C will cause a decrease in methyl ester yield.

### Table 4

<table>
<thead>
<tr>
<th>Property</th>
<th>Units</th>
<th>ASTM D6751</th>
<th>EN 14214</th>
<th>CIME</th>
<th>Polanga biodiesel$^a$</th>
<th>Test method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity at 40 °C</td>
<td>mm$^2$/s</td>
<td>1.9–6.0</td>
<td>3.5–5.0</td>
<td>3.45</td>
<td>3.99</td>
<td>ASTM D445</td>
</tr>
<tr>
<td>Density at 15 °C</td>
<td>kg/m$^3$</td>
<td>880</td>
<td>860–900</td>
<td>877.6</td>
<td>869</td>
<td>ASTM D127</td>
</tr>
<tr>
<td>Acid value</td>
<td>mg KOH/g</td>
<td>Max. 0.50</td>
<td>Max. 0.5</td>
<td>0.34</td>
<td>–</td>
<td>ASTM D664</td>
</tr>
<tr>
<td>Flash point</td>
<td>°C</td>
<td>Min. 130</td>
<td>Min. 120</td>
<td>165.5</td>
<td>–</td>
<td>ASTM D93</td>
</tr>
<tr>
<td>Pour point</td>
<td>°C</td>
<td>–15 to 16</td>
<td>–</td>
<td>2.0</td>
<td>4.3</td>
<td>ASTM D2500</td>
</tr>
<tr>
<td>Cloud point</td>
<td>°C</td>
<td>–3 to 12</td>
<td>–</td>
<td>2.0</td>
<td>13.2</td>
<td>ASTM D2500</td>
</tr>
<tr>
<td>Cold filter plugging point</td>
<td>°C</td>
<td>19</td>
<td>Max. +5</td>
<td>0.9</td>
<td>–</td>
<td>ASTM D6371</td>
</tr>
<tr>
<td>Calorific value</td>
<td>MJ/kg</td>
<td>–</td>
<td>35</td>
<td>41.44</td>
<td>41.397</td>
<td>ASTM D240</td>
</tr>
<tr>
<td>Oxidation stability</td>
<td>hour</td>
<td>Min. 3</td>
<td>Min. 6</td>
<td>14.27</td>
<td>–</td>
<td>EN 14112</td>
</tr>
<tr>
<td>Cetane number</td>
<td>–</td>
<td>Min. 47</td>
<td>Min. 51</td>
<td>59.5</td>
<td>–</td>
<td>ASTM D6890</td>
</tr>
<tr>
<td>Iodine value</td>
<td>g/l/100 g</td>
<td>–</td>
<td>Max. 120</td>
<td>106.5</td>
<td>–</td>
<td>EN 14111</td>
</tr>
<tr>
<td>Condreson carbon residue</td>
<td>wt.%</td>
<td>Max. 0.05</td>
<td>Max. 0.3</td>
<td>0.03</td>
<td>–</td>
<td>ASTM D4530</td>
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<tr>
<td>Copper strip corrosion</td>
<td>–</td>
<td>Max. 3</td>
<td>Min. 1</td>
<td>1a</td>
<td>–</td>
<td>ASTM D130</td>
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<tr>
<td>Water content</td>
<td>vol.%</td>
<td>Max. 0.05</td>
<td>Max. 500</td>
<td>0.015</td>
<td>–</td>
<td>EN ISO 12937</td>
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<tr>
<td>Sulfur content</td>
<td>wt.%</td>
<td>Max. 0.05</td>
<td>Max. 100</td>
<td>6.23</td>
<td>–</td>
<td>ASTM D6667</td>
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<tr>
<td>Sulfated ash</td>
<td>wt.%</td>
<td>Max. 0.02</td>
<td>Max. 0.02</td>
<td>0.001</td>
<td>–</td>
<td>ASTM D874</td>
</tr>
<tr>
<td>FAME content</td>
<td>wt.%</td>
<td>–</td>
<td>Min. 96.5</td>
<td>98.7</td>
<td>–</td>
<td>EN 14103</td>
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<tr>
<td>Methanol content</td>
<td>wt.%</td>
<td>Max. 0.20</td>
<td>Max. 0.20</td>
<td>0.01</td>
<td>–</td>
<td>EN 14110</td>
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<tr>
<td>Total contamination</td>
<td>mg/kg</td>
<td>–</td>
<td>Max. 24</td>
<td>8.5</td>
<td>–</td>
<td>EN 12662</td>
</tr>
<tr>
<td>Phosphorus content</td>
<td>ppm</td>
<td>10</td>
<td>4</td>
<td>3</td>
<td>–</td>
<td>EN 14107</td>
</tr>
<tr>
<td>Group I metals (Na + K)</td>
<td>mg/kg</td>
<td>Max. 5</td>
<td>Max. 5</td>
<td>2</td>
<td>–</td>
<td>EN 14538</td>
</tr>
<tr>
<td>Group II metals (Ca + Mg)</td>
<td>mg/kg</td>
<td>Max. 5</td>
<td>Max. 5</td>
<td>&lt;1</td>
<td>–</td>
<td>ASTM D5291</td>
</tr>
<tr>
<td>Carbon</td>
<td>wt.%</td>
<td>77</td>
<td>–</td>
<td>72</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>wt.%</td>
<td>12</td>
<td>–</td>
<td>12.2</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Oxygen</td>
<td>wt.%</td>
<td>11.00</td>
<td>–</td>
<td>11.80</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

$^a$ mg/kg.
$^b$ Sahoo et al.
yield as well as increasing molar ratio to 12:1. This is because excessive methanol and high temperature is not favorable for the purification as synthesized biodiesel. Moreover, much energy and cost were needed to recover the large amount of unreacted methanol during separation process. Ma and Hanna [3] evaluated that higher temperature and time will burn alcohol and cause a reduction in methyl ester yield.

3.3.2. Effect of catalyst concentration and methanol to oil molar ratio
Alkali alkoxides are most effective catalyst in transesterification process compared to acidic catalyst. Transmethylation occurs in the presence of both alkaline and acidic catalysts [34]. Being less corrosive to industrial equipment, alkaline catalyst is preferred in industrial processes. Fig. 7 shows the yield of biodiesel versus NaOH concentration at different methanol to oil molar ratio. The alkali catalyst concentration in the range of 0.5–1 wt.% and molar ratio 3:1–15:1 has ester yield of 94–99%. In this study, the optimum process was found at 9:1 M ratio with 1 wt.% of NaOH catalyst concentration and produced 98.01% of methyl ester yield. However, the conversion methyl ester reached 89.33% with 0.5–0.75 wt.% of NaOH catalyst and 3:1–6:1 M ratio because low amount of catalyst were not reacted with oil completely. Moreover, the ester yield decreases with the increase of NaOH catalyst concentration after 1.25 wt.% and 12:1 M ratio. Hence, increasing NaOH catalyst will cause insolubility to oil and methanol would achieve heterogenous phase for ester yield. Consequently, the increase of alkali catalyst causes the ester yield to decrease due to formation of emulsion and saponification to the gel formation.

3.3.3. Effect of reaction time and methanol to oil molar ratio
The effect of reaction time versus molar ratio to reduce viscosity of methyl ester is shown in Fig. 8. The effect of reaction time was observed using varying time parameter at 1–3 h. The viscosity of C. inophyllum achieved 3.45 mm²/s for 2 h and increase to 3.80 mm²/s after 2 h of reaction. It was also observed that increasing reaction time beyond 2 h did not have much effect on reducing the viscosity. This might be due to the effect of high molar ratio which causes water production during the transesterification and consequently lead to incomplete further reaction. Besides, it was observed that biodiesel viscosity increased with increase in reaction time from 2.5 to 3 h for 12:1 and 15:1 M ratio. Similarity, this is also stated in Rashid et al. [35] and Yusup and Khan [33] in which the reaction time of transesterification process at 1–3 h depends on physicochemical properties of oil. In this study, the obtained optimum conditions for C. inophyllum oils is 9:1 M ratio and the reaction took 2 h to complete.

### Table 5

<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>Diesel fuel</th>
<th>Biodiesel blends</th>
<th>Calophyllum inophyllum blending</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity at 40 °C</td>
<td>mm²/s</td>
<td>2.95</td>
<td>D445</td>
<td>1.9–4.1</td>
</tr>
<tr>
<td>Density at 15 °C, max.</td>
<td>kg/m³</td>
<td>840.0</td>
<td>D6890</td>
<td>858</td>
</tr>
<tr>
<td>Flash point</td>
<td>°C</td>
<td>70.5</td>
<td>D93</td>
<td>52</td>
</tr>
<tr>
<td>Cloud point</td>
<td>°C</td>
<td>–2</td>
<td>D2500</td>
<td>–</td>
</tr>
<tr>
<td>Oxidation stability</td>
<td>hours</td>
<td>26.5</td>
<td>EN14112</td>
<td>6</td>
</tr>
<tr>
<td>Acid number</td>
<td>mg KOH/g</td>
<td>0.051</td>
<td>D664</td>
<td>0.3</td>
</tr>
<tr>
<td>Cetane number</td>
<td>–</td>
<td>49.7</td>
<td>D6890</td>
<td>Min. 47–51</td>
</tr>
<tr>
<td>Calorific value</td>
<td>MJ/kg</td>
<td>45.825</td>
<td>EN14214</td>
<td>35</td>
</tr>
<tr>
<td>Conradson carbon residue</td>
<td>mass%</td>
<td>0.821</td>
<td>D524</td>
<td>0.35</td>
</tr>
<tr>
<td>Sulfate ash content, max.</td>
<td>mass%</td>
<td>0.08</td>
<td>D2709</td>
<td>0.05</td>
</tr>
<tr>
<td>Sulfur, mg/kg max.</td>
<td>mg/kg</td>
<td>14.9</td>
<td>D1298</td>
<td>10</td>
</tr>
<tr>
<td>Water and sediment, max.</td>
<td>% v/v</td>
<td>0.003</td>
<td>EN14078</td>
<td>0.05</td>
</tr>
<tr>
<td>Copper strip corrosion</td>
<td>–</td>
<td>1</td>
<td>EN2160 Class 1</td>
<td>la</td>
</tr>
</tbody>
</table>

### Fig. 6

Effect of methanol to oil molar ratio and temperature on ester yield.
3.4. Performance and emission analysis

The performance and exhaust emission were tested by varying the engine speed at full throttle (full load) for C. inophyllum biodiesel diesel blends (CIB10, CIB20, CIB30 and CIB50) and diesel fuel. The performance of the engine is explored by evaluating the parameters like Bsfc, BTE and EGT. Besides, the exhaust emissions such as CO, NO\textsubscript{x}, and smoke opacity were also investigated. Based on the experimental results, the effects density, viscosity, calorific value and blending ration on CI engine performance are analyzed in this section.

3.4.1. Brake specific fuel consumption (Bsfc)

Brake specific fuel consumptions of biodiesel diesel blends (CIB10, CIB20, CIB30 and CIB50) are shown in Fig. 9 and compared to diesel fuel. CIB10 give quite satisfactory results in Bsfc which is 302.9 g/kW h compared to diesel fuel (313.8 g/kW h) and CIB50 (480.1 g/kW h) at 1900 rpm. CIB10 shows slightly lower Bsfc than diesel fuel due to the biodiesel has a relatively higher heat of vaporization than diesel, which affects the combustion [36]. Besides, the Bsfc of CIB20, CIB30 and CIB50 higher than CIB10 and diesel fuel due to the lower calorific value of CIB20, CIB30 and CIB50. On the other hand, CIB50 has highest Bsfc of biodiesel diesel blend which is 621.4 g/kW h at 1500 rpm. As expected, the Bsfc of the biodiesel diesel blends increase compared to diesel is mainly due to the low calorific value of the biodiesel [37,38]. Some researchers have explained that the increase in Bsfc in relation with the high density and viscosity of the biodiesel [2,39]. At full load condition, the Bsfc decreases with engine speed until a minimum value at 1900 rpm, then increasing again with an increase in engine speed. This is because the heat loss from the combustion chambers walls is proportionately greater at low speeds. Thus, combustion efficiency is poorer causing the higher fuel consumption for the power produced. However, the friction power increases at a rapid rate at higher speeds will cause in a slower increase in power than in fuel consumption with a slight increase in Bsfc [40,41]. The decrease in Bsfc is a result of better physical and chemical conditions for combustion at low engine speeds. In addition, the Bsfc was decrease in proportion of CIB10 compared to diesel fuel and the CIB 10 is the optimum blending ratio for higher power and torque output with lower Bsfc. This indicates that using CIB10 increased oxygen content and higher combustion rate at the
same engine power for various engine speeds. Similar trends of BSfc decrease with lower biodiesel blends ratio were also reported by other researchers [13,42,43].

3.4.2. Brake thermal efficiency (BTE)

Brake thermal efficiency is the ratio between work output and the heat available introduced through fuel injection. The variation of brake thermal efficiencies using C. inophyllum blends and diesel fuel is shown in Fig. 10. It can be observed that the BTE values for CIB10, CIB20, CIB30 and CIB50 were 23.32%, 22.44%, 21.21% and 20.63% compared to diesel fuel which was about 22.52% respectively. The high thermal efficiency of CIB10 has lower viscosity and increases the volatility compared to CIB20, CIB30 and CIB50. This enhances the fuel atomization leading to improved air fuel mixing. Therefore, the thermal efficiency for CIB20, CIB30 and CIB50 are lower than CIB10. This implies that fuel atomization characteristics are different in blends with higher ratios of biodiesel. It is clearly shown that BTE is inversely proportional to BSfc which as BSfc decreased the BTE values increased. This study corresponds with Rahman et al. [2] which reported that CIB5 at 1500 rpm has slightly higher combustion temperature and additional oxygen content of the biodiesel facilitate better combustion compared to diesel fuel. On the other hand, Muralidharan et al. [29] also reported that the oxygen content of the blends is sustained in the diffusive combustion phase which ignition delay is an important parameter in the combustion analysis.

3.4.3. Exhaust gas temperature (EGT)

The trend result for exhaust gas temperatures versus with the speed is shown in Fig. 11. This shows that the EGT increases as the speed increases for all fuels in CI engines. This is due to the fact that fuel combustion amount in the combustion chamber within the unit time increases and consequently the heat energy produced increases as the engine speed rise. The CIB10 shows slightly lower exhaust gas temperatures compare with all other fuels. Lower EGT (510.8 °C) is an indication of good combustion of fuel in the combustion chamber for CIB10 at 2400 rpm. CIB10 has high cetane number, good formation of mixture and oxygenated fuel. However, the highest EGT for CIB20, CIB30 and CIB50 were obtained at 518.2 °C, 520.8 °C and 525.3 °C, whereas the EGT for diesel fuel is 515.0 °C at 2400 rpm. The higher EGT of CIB20, CIB30 and CIB50 could be due to lower calorific value and higher viscosity which cause the poor atomization and not burnt properly in the combustion chamber. Panwar et al. [44] and Devan and Mahalakshmi [45] also observed the poor combustion characteristic caused by high viscosity of biodiesel blends. As a result, lengthen combustion duration is the reasons for high viscosity of biodiesel diesel blends (20–40%) to have higher exhaust gas temperatures. Moreover, Kumar et al. [46] reported that biodiesel diesel blends has higher EGT and it will increase BSfc compared to diesel fuel. In addition, this is an indication of higher EGT and it could be the possible reason for lower performance for all CIB20, CIB30 and CIB50.

3.4.4. Nitrogen Oxides (NOx) emission

Fig. 12 represents NOx emission at full throttle for C. inophyllum biodiesel diesel blends and diesel fuel. The NOx emission is 173.4 ppm, 182.2 ppm, 189.4 ppm and 196.7 for CIB10, CIB20, CIB30 and CIB50 at 2400 rpm respectively. There is a slightly reduction of NOx at 1900 rpm which was 139.3 ppm (CIB10), 148.5 ppm (CIB20), 154.9 ppm (CIB30) and 162.8 ppm (CIB50). However, the lowest NOx was observed for diesel fuel of 119.7 ppm at 1500 rpm. The presence of oxygen in C. inophyllum biodiesel blends caused higher NOx formation and it is indicated that exhaust gas temperature was increased as well [47]. Besides, Keskin et al. [48] also reported that NOx emissions are slightly increased due to increasing biodiesel concentration in the fuel. This is also agreed with Ilkılıç and Aydin [49] which reported that biodiesel with high oxygen content could adequate rising in high temperature and increases the formation rate of NOx. Furthermore, Ozezeen and Canakci [30] mentioned that the oxygen content is an affected in the NOx formation due to high exhaust gas temperatures caused excess hydrocarbon oxidation. Lin et al. [50] observed that higher EGT leads to increase in NOx emission.

3.4.5. Carbon monoxide (CO) emission

Fig. 13 shows the carbon monoxide emission for C. inophyllum biodiesel diesel blends and diesel fuel. The CO emission was 0.2297%, 0.2182%, 0.2427%, 0.2508% and 0.2578% for diesel fuel, CIB10, CIB20, CIB30 and CIB50 respectively. It is shown that CO emission of CIB20, CIB30 and CIB50 were higher than diesel fuel. It is clear that CO emissions increase when the biodiesel blend ratio is added. This is due to higher viscosity of the CIB20, CIB30 and CIB50 which causes poor spray characteristics during the combustion process and cause the CO formation. The results of increasing CO emissions when the increase of biodiesel blending ratio was agreed with Kivevele et al. [51] studies. Besides, Banapurmath et al. [52] observed that CO emissions for jatropha, honge and sesame biodiesel were higher compared to diesel on a single-cylinder, 4-stroke diesel engine at a speed of 1500 rpm with 80% load. An et al. [53] reported that CO increased when increase of biodiesel blend ratio at lower engine speed is due to high viscosity of biodie-
diesel can influence combustion process and tend to cause incomplete combustion. However, CIB10 has lower CO emission than diesel fuel and other blends in this study. This is because low viscosity and density of CIB10 compared to other blends and higher cetane number compared to diesel fuel which will improve the combustion and reduced CO emissions. This result agreed with Sanjid et al. [54] which investigated that 10% of biodiesel blends have lower CO emissions compared to diesel fuel and other blends. It is caused by lower density and viscosity leads good fuel atomization and spray formation which leads to complete combustion and hence CO emissions decrease. Moreover, Rizwanul Fattah et al. [55] stated that CO emission reduced due to fuel rich zones formation as well as advances injection and complete combustion.

3.4.6. Smoke opacity

Smoke opacity is an indirect indicator of soot content in the exhaust gases. The variation of smoke opacity with engine speed for C. inophyllum biodiesel blends compared to diesel was shown in Fig. 14. It was observed that smoke opacity for CIB10, CIB20, CIB30 and CIB40 was 24.7%, 27.92%, 30.81% and 32.98% respectively at 2400 rpm. However, the highest smoke opacity was found 40.55% for diesel fuel at the same speed. The CIB has lower smoke opacity than diesel because the oxygen content present in biodiesel diesel blends. This is agreed with Xue [56] which reported that smoke emissions for biodiesel diesel blends decreased due to the enrichment of oxygen content in the fuel. Besides, Buyulkaya et al. [57] also obtained that results that smoke opacity by using biodiesel blends was reduced compared to diesel due to presence of oxygen in biodiesel. Moreover, many researchers were agreed that the rich oxygen content in biodiesel will reduce smoke opacity [21,44,58]. Another reason for smoke emissions reduction for biodiesel is because lower carbon to hydrogen ratio and absence of aromatics compounds compared with diesel fuel. The lower carbon molecule and higher oxygen content can decrease the tendency of a fuel for soot production and reduce smoke opacity [43]. It was agreed by Gumus and Kasifoglu [59] stated that biodiesel contains rich with oxygen can decrease smoke formation. On the other hand, Zhang et al. [60] observed that start of combustion (SOC) for biodiesel blends is earlier than diesel. The result indicated that earlier SOC for biodiesel blends and advanced injection timing can reduce the smoke emission. On other hand, high smoke opacity of diesel fuel was determined due to higher sulfur content compared to biodiesel [61]. It was agreed with Çelikten et al. [47] which mentioned that reduction of smoke opacity for biodiesel is principally based on the high oxygen content of biodiesel and lower aromatic and sulfur content.

4. Conclusions

The C. inophyllum biodiesel was produced in the lab scale via degummed, acid esterification, neutralization and followed by base transesterification process. The ester yield was found to be 98.92% by using 1 wt.% catalyst concentration, 9:1 M ratio methanol to oil at 50 °C for 60 min. The present results obtained show that degummed and neutralization process improved the fuel properties of the C. inophyllum biodiesel. C. inophyllum methyl esters and the blends (CIB10, CIB20, CIB30 and CIB50) comply with ASTM biodiesel standard. Besides, it has been found that CIB10 gave good improvement in the engine performance with higher BTE from the engine performance test results. On the other hand, there is an improvement in fuel economy with lower Bsfc and EGT by using CIB10 compared to diesel fuel. Furthermore, CIB10 reduced the CO and smoke opacity compared to diesel fuel but NOx emission is slightly increased. The experimental results proved that C. inophyllum biodiesel diesel blends is a potential alternative fuel which can be used effectively in diesel engine without modification. Finally, further studies on tribology and fuel economy need to be carried out before the C. inophyllum biodiesel diesel blends can be utilized widely in the future.

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