



# Efficient production and optimization of biodiesel from kapok (*Ceiba pentandra*) oil by lipase transesterification process: Addressing positive environmental impact

S. Pooja<sup>a</sup>, B. Anbarasan<sup>b</sup>, V. Ponnusami<sup>a, \*\*</sup>, A. Arumugam<sup>a, \*</sup>

<sup>a</sup> Bioprocess Intensification Laboratory, Centre for Bioenergy, School of Chemical & Biotechnology, SASTRA Deemed University, Thirumalaisamudram, Tamil Nadu, Thanjavur, India, 613401

<sup>b</sup> Department of Mechanical Engineering, PSNA College of Engineering and Technology, Dindugul, 624622, India

## ARTICLE INFO

### Article history:

Received 10 March 2020

Received in revised form

9 November 2020

Accepted 11 November 2020

Available online 13 November 2020

### Keywords:

*Ceiba pentandra* oil

Biodiesel

Enzymatic transesterification

Emission characteristics

Engine performance

Thermophysical parameter

## ABSTRACT

*Ceiba pentandra*, non-edible oil (acid value of 21 mg KOH/g estimated using ASTM D664 methodology) is employed as a source for producing biodiesel using lipase immobilized on mesoporous material as a catalyst. Optimum conditions for maximum yield (96.4%) were the temperature of 33 °C, methanol to oil molar ratio of about 13.3:1 with a water content of 14.5%. From the reusability studies, it can be observed that greater than 85% conversion could be obtained up to 10 cycles, thereby proving the significant efficacy of the catalyst. Density, flash point, cloud point, calorific value, and cetane number of the produced biodiesels were 885 kg/m<sup>3</sup>, 152 °C, −3 °C, 38.44 kJ/kg and 57.2, respectively meeting the ASTM standards specified for biodiesel. The performance and emissions characteristics of 20% biodiesel (CIB20) and petroleum diesel were studied in a VCR under varying speeds in a full load condition. Blended biofuel showed a 13% lower mean brake power (BP) and 25% higher mean specific fuel consumption (SFC) compared to diesel fuel. Though NO<sub>x</sub> emission of the blended diesel was 31% higher than that of petroleum diesel, Hydrocarbon, CO<sub>2</sub>, and CO emissions were 8.4%, 13.7%, and 5.08% lower than that of diesel fuel, respectively.

© 2020 Elsevier Ltd. All rights reserved.

## 1. Introduction

The world's dependency on non-renewable fossil fuels is increasing annually for the rapidly growing industries, causing a hike in crude oil prices every single day [1]. To limit the uncertainties existing, scientists realized that raw materials from biological sources can be a substitute to produce renewable fuels. Nowadays the production of biodiesel is gaining immense attention and various reports suggest its superiority as an alternative over conventional petrochemical-based fuels [2].

Biodiesel, irrespective of using it alone or blended with diesel offers many advantages related to the properties such as high cetane number, low viscosity, high lubricity, high flash point. Biodiesel can also be termed environmental friendly because of lower toxic gas

emissions compared to conventional fossil fuels, thereby play a very important role in reducing air pollution [3]. The biodiesel properties are dictated by feedstock, catalyst as well as the alcohol used, it can be substituted for traditional diesel fuel provided, the standard specifications are satisfied. Certain reports suggested that biodiesel reduces carbon emissions by 78%. Generally, the production of biodiesel is carried out by transesterification using oils, animal fats, and alcohols in the presence of a catalyst [4]. Soya bean, coconut, olive, palm, sunflower, canola, and castor oil are the most commonly used edible sources. Biodiesel production from non-edible sources is getting progressive because the utilization of edible sources might have consequences related to mankind [5]. Kapok oil (*Ceiba pentandra*), a non-edible feedstock considered a better source because of abundant availability and high oil yield among other non-edible feedstock. Cultivated fruit and seeds yield about 1280 kg/ha/y of oil, which is higher for biodiesel production [6,7]. Kapok (*Ceiba pentandra*) belongs to the order Malvales of the family Malvaceae, found in the southern parts of Asia like India, Indonesia, Central America, Mexico, South America, and Central Africa. A tree may produce fruits around 500 to 4000, each fruit possessing 200 seeds.

\* Corresponding author.

\*\* Corresponding author.

E-mail addresses: [vponnu@chem.sastra.edu](mailto:vponnu@chem.sastra.edu) (V. Ponnusami), [aruchemxl@sbt.sastra.edu](mailto:aruchemxl@sbt.sastra.edu) (A. Arumugam).

Kapok oil obtained from the seeds of kapok tree which possess up to 25–28% (w/w) of each fruit [8]. Kapok oil-derived biofuel is a growing interest and is cited in lesser-known literature. Kusumo et al., 2017 used artificial neural networking to study the KOH-catalyzed transesterification of *Ceiba pentandra* oil. The parameters taken into consideration for optimization of the transesterification process are the weight of KOH, methanol: oil ratio, temperature, reaction time, and agitation time. Under the optimized conditions, they were found to be 10:1, 1%, 60 °C, 108 min, and 1100 rpm, respectively, with total methyl ester yield of 99.8% [9]. Dharma et al., 2016 carried out the transesterification process for the mixture of *Jatropha curcas* and *Ceiba pentandra* oil for the production of biodiesel. The yield was optimized using RSM, incorporating catalyst concentration, methanol: oil ratio, and rpm as parameters [10]. The optimized conditions were found to be 30% of methanol: oil ratio, 1300 rpm, and 0.5% (wt) of catalyst concentration, and the maximum yield was found to be 93.3%. The optimized oil mixture has been reported to comply with ASTM (American Society for Testing and Materials), D6751, and EN14214 standards [11]. Lieu et al., 2016 proposed that treatment of *Ceiba pentandra* oil with microwave leads to the reduction in total free fatty acids which aided the production of high-quality biodiesel in the succeeding step. The optimized condition gave a maximum yield of 94.43% with 2% of sulphuric acid compared to 20.83% without sulphuric acid [12]. Senthil Kumar et al., 2015 produced biodiesel from kapok oil using a base-catalyzed transesterification process. Pretreatment was carried out using 0.5 wt% of H<sub>2</sub>SO<sub>4</sub> to reduce the oil's free fatty acid content. Transesterification of acid-treated oil was performed using 0.5 wt% of KOH as a catalyst. The process was carried out in optimum methanol to oil molar ratio of 6:1, at 65 °C for 3 h. Engine performance and emission exhaust analysis suggested that B20 and B40 blends seem to be effective in all aspects [5]. Ong et al., 2014 extracted oil from seeds of *Jatropha curcas*, *Ceiba pentandra*, *Calophyllum inophyllum*, and produced biodiesel by two-step transesterification process. Esterification was performed using sulphuric acid, followed by NaOH catalyzed transesterification, with 9:1 Methanol: oil molar ratio at 65 °C, stirred at 1000 rpm for 1 h. Properties of the methyl ester were analyzed and performance emission analysis for B10, B20, B30, B50 results showed fewer emissions of CO, CO<sub>2</sub> along with increased NO<sub>x</sub> emissions [6]. There are several well-established technologies are available for the synthesis of biodiesel. Chemical, enzymatic catalysis, and the non-catalytic supercritical fluid method are some of the commercially applied techniques. Enzymatic transesterification started to replace chemical catalysis due to the following advantages such as (1) No requirement of pre-treatment approaches such as degumming, acid pretreatment and neutralization (2) Formation of unwanted by-products such as soaps (3) Higher methanol to oil molar ratio [13–18]. Immobilization of enzymes onto a substrate has great economical advantage owing to their ability to keep the enzyme confined to the immobilized supports [19]. In this way, the cost involved at the downstream end to purify and regenerate the costly enzymes is significantly reduced.

This is the first work to be reported in the transesterification of kapok oil using an immobilized enzyme with higher biodiesel yield. The objective of the present work aims in optimizing the transesterification parameters for the production of methyl ester from *Ceiba pentandra* seed oil using Response Surface Methodology (RSM). The effect of process parameters on the yield of biodiesel such as methanol to oil ratio, water content, and the temperature was investigated. Enzyme re-utilization was assessed under the optimized condition. Lastly, the properties of *Ceiba pentandra* biodiesel were determined according to the ASTM method. Also, the performance and emission characteristics of the kapok oil biodiesel – diesel blend (B20) was studied.

## 2. Materials and methods

### 2.1. Materials

Kapok oil was purchased from the nearby farm. The density of Kapok oil was found to be 921 kg/m<sup>3</sup>. The viscosity of the oil at 40 °C was 30.2 mm<sup>2</sup>/s. The acid value of the oil was 21 mgKOH/g. The calorific value of kapok oil was 39.2 MJ/kg. The fatty acid composition of *Ceiba pentandra* oil is linolenic acid (39.11%), oleic acid (26.69%), palmitic acid (23.2%) Margaric Acid (2.77%), Arachidic Acid (1.92%), Tricosylic Acid (1.88%), Erucic Acid (0.91%), Cerotic Acid (0.67%) acids and lipids (2.85%). Porcine Pancreatic lipase and other chemicals used in this study were procured from Sigma Aldrich. Engine test analysis was done in the VCR engine and the emissions characteristic was analyzed by using a gas analyzer (DELTA 1600). Design expert 11.0 was used to optimize biodiesel production.

### 2.2. Methodology

#### 2.2.1. Synthesis of support matrix and immobilization of lipase

Synthesis of SBA-15 (Santa Barbara Amorphous-15) can be done by hydrothermal and sol-gel routes [18]. The preparation of mesoporous SBA-15 requires a structure-directing agent, Pluronic P123 (PEO<sub>20</sub> PPO<sub>70</sub> PEO<sub>20</sub>). 5 g of P123 was dissolved in 35 mL of water leading to the formation of micelles. 155 mL of 2 N HCl was added to it. 14 mL of TEOS was added to this mixture dropwise, transferred to a Teflon bottle, and aging was done at 90 °C for 24 h. The aged sample was calcined for 5 h at 550 °C to remove the surfactant. Immobilization of lipase on mesoporous material was carried out by dispersing 500 mg of support in 20 mL of lipase (1 mg/mL of lipase dissolved in phosphate buffer solution) and kept in a shaker for 12 h and the immobilized lipase centrifuged at 6000 rpm for 10 min. Specific enzyme activity (olive oil emulsion method) was found to be 40 IU/mg [20]. Lowry's assay was performed and 40% immobilization was observed.

#### 2.2.2. Transesterification process

All performed transesterification experiments were conducted in 30 mL vials. 5 g of kapok oil was mixed with 8:1 Methanol to oil ratio, 10 vol% of hexane, the Water content of 10 vol%, and 100 mg of immobilized lipase was used as catalyst and temperature of 30 °C was maintained at 100 rpm for 10 h. After 10 h, the sample was centrifuged out at 5000 rpm to remove the residual methanol in the mixture. To optimize production, methanol to oil ratio of 3:1 to 20:1, the temperature of 25–40 °C, the water content of 2–24 vol% was varied and methyl esters yield in each case was determined by performing GC-MS. In all cases, methanol was added in a stepwise manner for every 2 h in order not to reduce the activity of the enzyme to enhance the catalyst activity. All the experiments were done in triplicates. The biodiesel produced from the kapok oil (*Ceiba pentandra*) was analyzed by Gas Chromatography-Mass Spectroscopy (CLARUS 500, PerkinElmer, USA). Capillary Column Elite-5MS (5%Phenyl 95% dimethylpolysiloxane) length 30 m and 250 μm inner diameter was used as chromatography capillary column. With the split ratio of 1:10, helium as carrier gas @flow rate 1 mL/min was used by the column. At the rate of 10 °C/min, the column was heated to 280 °C. To handle the chromatogram and mass spectra, Turbomass ver5.2.0 software was used. The mass and electron energy were found to be 40–600 amu and 70 eV respectively. The hexane, along with the methyl esters of heptadecanoic acid (300 μL) and the reaction mixture (5 μL), was used as a solvent.

#### 2.2.3. Characterization of SBA-15

The nitrogen adsorption/desorption isotherms were used to

identify the pore size distribution surface area of the specimen, the measured absorption rate of desorption  $P/P_0$  from 0.01 to 0.99, and isothermal  $N_2$  adsorption at 77 K (Quanta chrome, USA).

#### 2.2.4. Experimental design for optimization

A central composite design with methanol to oil molar ratio, temperature, and water content as factors and biodiesel yield as a response variable was used to understand the individual effects of factors on biodiesel and the interaction between the factors. All factors were varied at three levels (−1, 0, and +1) and 6 central points were used. Thus, a total of 20 experiments were performed as shown in Table 1. Design expert 11.0 was used for the statistical analysis. A quadratic model was used to predict the response variable.

#### 2.2.5. Experimental test engine and instrumentation

The experiment was performed using a 3.7 kW single-cylinder, four-stroke, direct injection, 1500 rpm, water-cooled VCR diesel engine. The engine contains cylinder Bore, stroke, and connecting rod of length 87.50 (mm), 110.00 (mm), and 234.00 (mm) respectively, with a swept volume 661.45 (cc). The orifice diameter and its coefficient of discharge were found to be 20.00 (mm) and 0.60 respectively. Dynamometer, used to measure the torque and rpm, has an arm length of 185 mm. The fuel pipe diameter was found to be 12.40 mm and pulse per revolution was found to be 360 in an ambient temperature of 27 °C. Combustion parameters such as air density, adiabatic index, and the polytropic index were found to be 1.17 ( $kg/m^3$ ), 1.41, and 1.30 respectively with a specific gas constant of 1.10. Cylinder pressure and smoothing were maintained at 4 and 2 respectively, with TDC of 30. The parameters were estimated for 10 numbers of cycles, with an automated data acquisition system.

Diesel-Ceiba pentandra oil blends were arbitrarily selected in the ratio of 100:0 and 90:10 for a standard load test. The fuel is pressurized to 210 bar at the injection point, with an injection time of 20–40 °CA. Each run had a maximum compression ratio of 18:1 for every individual runs. The engine exhaust composition was measured 20 cm from the exhaust valve using DELTA 1600-L. The gas analyzer was used to analyze the exhaust containing Carbon-dioxide ( $CO_2$ ), oxides of nitrogen ( $NO_x$ ), hydrocarbons (HC) and oxygen (O), by changing the torque from 0 to maximum of 22 Nm in six intervals. The data acquiring system recorded the data of performance and emissions of the engine were used to conclude diesel-Ceiba pentandra oil blends as fuel, compared with diesel.

**Table 1**

Experimental results based on Central Composite Design for the production of biodiesel from Ceiba pentandra.

Runs	Pt type	Block	Methanol/oil (mole)	Water content (vol%)	Temperature (°C)	Experimental FAME Yield (%)	Predicted FAME Yield (%)
1	1	1	4	2	30	33.2	31.9
2	1	1	20	2	30	50.4	50.5
3	1	1	4	22	30	50.4	50.5
4	1	1	20	22	30	69.1	68.8
5	1	1	4	2	40	49.4	48.9
6	1	1	20	2	40	50.3	50.1
7	1	1	4	22	40	69.1	68.8
8	1	1	20	22	40	50.3	50.8
9	−1	1	4	12	35	40.3	39.9
10	−1	1	20	12	35	75.2	74.1
11	−1	1	12	2	35	50.3	50.1
12	−1	1	12	22	35	84.5	84.8
13	−1	1	12	12	30	40.1	39.3
14	−1	1	12	12	40	81.2	81.1
15	0	1	12	12	35	50.3	91.5
16	0	1	12	12	35	94.3	91.5
17	0	1	12	12	35	57.0	91.5
18	0	1	12	12	35	92.5	91.5
19	0	1	12	12	35	75.2	91.5
20	0	1	12	12	35	91.4	91.5

### 3. Results and discussion

#### 3.1. Characterization of SBA-15

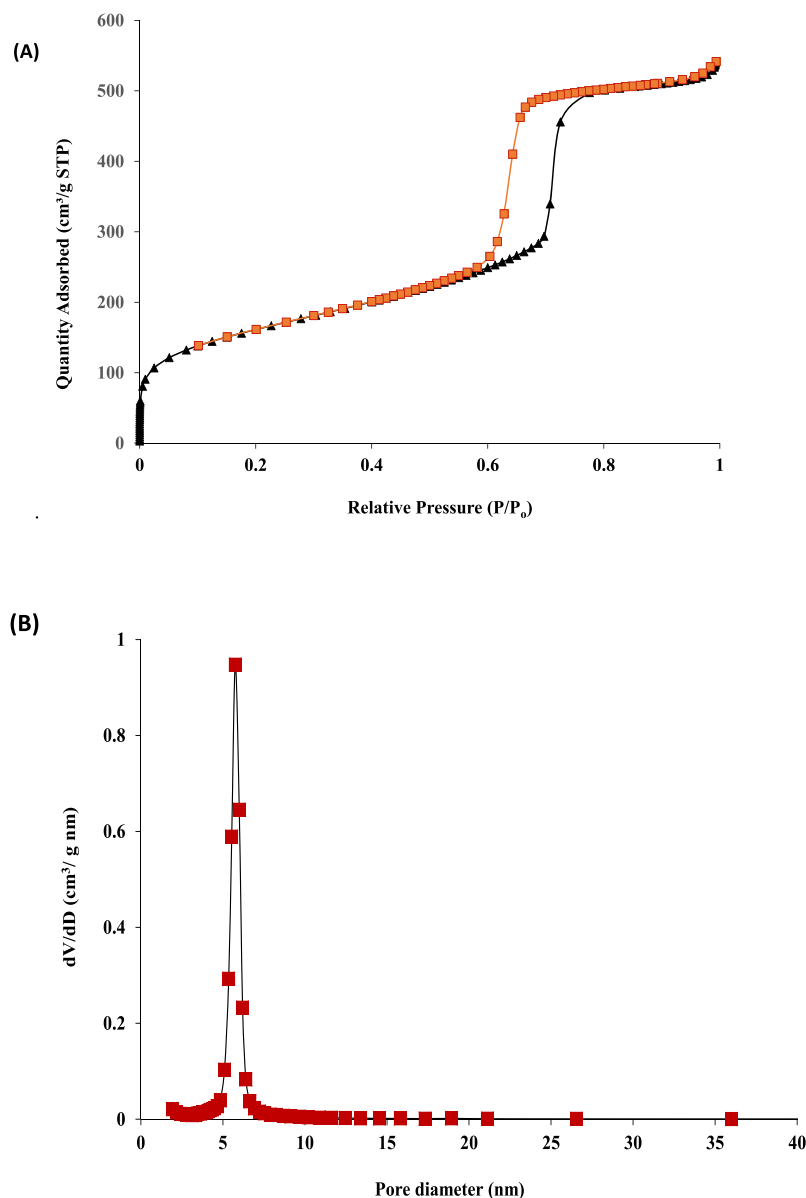
Fig. 1A shows SBA-15 exhibit IUPAC adsorption isotherm of type IV and it is evident that SBA-15 belongs to mesoporous material with a mesopore size. Adding to it, the cylindrical pores and uniform pore size are the typical characteristics of the hysteresis type of mesoporous materials. It is also evident from Fig. 1B that the SBA-15 with the uniform pore size is synthesized from the ash of backyards and concentrated in 6–12 nm. The surface area of 573.9  $m^2/g$  of the SBA-15 was calculated from the data obtained by the BET method and the volume of pores was 0.7009  $cm^3/g$  and the pore size was 5.75 nm. Porcine Pancreatic lipase (PPL) has a molecular dimension of  $4.6 \times 2.6 \times 1.1 nm^3$  and hence the PPL may enter the pores of SBA-15 (pore size 5.75 nm). The pore size of the microchannels is sufficiently large for entrapment of biomolecules inside the pores [21].

#### 3.2. Effect of methanol content

According to the transesterification stoichiometry, 3 mol of methanol was required to produce methyl esters from 1 mol of triglyceride. The yield can be improvised with the excessive addition of alcohol [22]. A higher biodiesel yield of 95.2% was observed in methanol to oil ratio of 12:1. Fig. 2A shows a 14.4% decrease in yield when the molar ratio was increased from 12:1 to 16:1, thereby supporting the fact that a decrease in yield occurs when excess methanol blocks enzyme reactivity. Similarly, Vipin et al., 2016 also suggests a 6.5% decrease in conversion when molar ratio was increased from 1:4 to 1:5 [23].

#### 3.3. Effect of temperature

Temperature is a significant parameter to be optimized as it determines the rate of the reaction and helps to get a higher yield. Here, the transesterification process was carried out at various temperatures to obtain a higher yield. From Fig. 2B, it is clear that with an increase in temperature biodiesel yield is high at 35 °C, then with an increase in temperature observed 20% decrease in yield at 40 °C which may be due to denaturation of the enzyme [24]. Following our results, Arumugam et al., 2014 also obtained higher yield at 35 °C, after which an 8% reduction in yield was observed [25].



**Fig. 1.** (A)  $N_2$  adsorption-desorption graph Nitrogen adsorption-desorption isotherms at 77 K of SBA-15 (B) Pore size distributions of SBA-15.

### 3.4. Effect of water content

Production of fatty acid methyl esters from triglycerides was determined greatly by the lipase enzyme's stability against the presence of water and/or other solvents in the transesterification reactions employing enzymes as a catalyst. Different percentages of water say 2, 8, 12, 18, and 24 (vol %), were used in the transesterification process to assess its effect on the yield. A higher biodiesel yield of 94.9% was observed with the water content of 12 vol%, after which a 13.17% decrease in yield was observed for 18 vol% water content (Fig. 2C). Reduction in the yield of biodiesel varies concerning the excess water content because lipase catalyzes hydrolysis. Experiments of Bautista et al., 2015 show an increase in transesterification rate up to 10 vol %, after which the reaction rate decreases by 20% [26].

### 3.5. Reusability

Reusability study is an important parameter in determining the stability of the immobilized lipase on the support matrix used. The choice of enzyme and support play a very important role in biodiesel yield for reusability. Separation of immobilized lipase, once the reaction finishes for every cycle, is done by centrifugation of the bottom phase at 25 °C at 5000 g. Then, a phosphate buffer solution (pH 7) was employed to wash the separated immobilized lipase. The recyclable usage of the lipase immobilized on SBA-15 has been shown in Fig. 2D. The reusability study was carried out for 10 cycles and the results were found to be in the range of 96.4%–85.2%. The reduction in the percentage yield of FAME was only 11%. Fig. 2D reflects that the gradual decrease in FAME yield was ascribed to both the losses, activity of immobilized lipase, and enzyme due to leaching [20,25]. Hence the immobilized enzyme can be successfully used in large-scale operations.

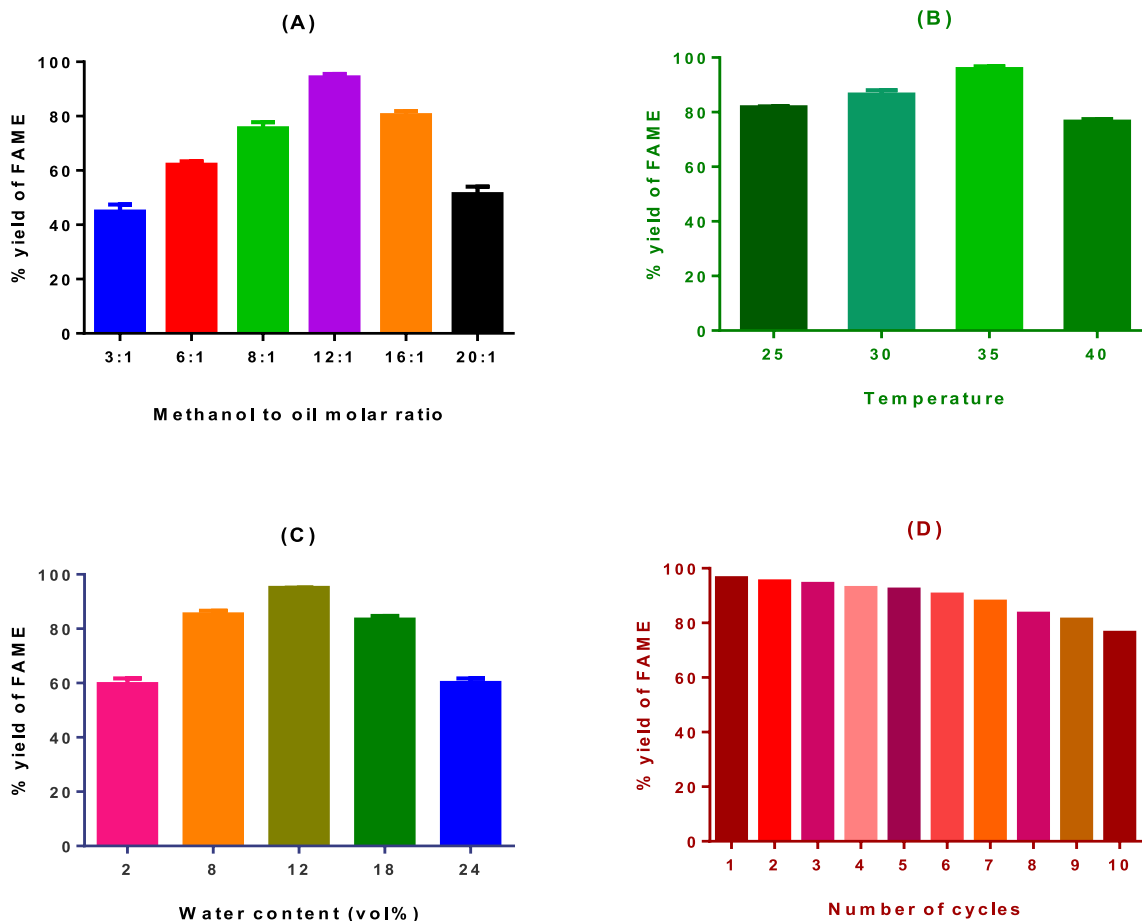


Fig. 2. (A) Effect of methanol to oil molar ratio (3:1,6:1, 8:1, 12:1, 16:1, 20:1) on methanolysis of *Ceiba pentandra* oil for temperature 30 °C and reaction time of 10 h (B) Effect of temperature on methanolysis of waste *Ceiba pentandra* oil for methanol to oil molar ratio of 12:1 and reaction time of 10 h (C) Effect of percentage water content on methanolysis of waste *Ceiba pentandra* oil for 12:1 M ratio of methanol to oil, temperature 35 °C, and reaction time of 10 h (D) Reusability studies Immobilized lipase on methanolysis of waste *Ceiba pentandra* oil at optimum conditions.

### 3.6. Optimization by response surface methodology

Individual effects of methanol to oil ratio, temperature, and water content were studied using RSM, and optimization of conditions to achieve maximum biodiesel yield was carried out. ANOVA was performed and the results are presented in Table 2. P values for all coefficients included in the quadratic model were less than 0.05, except for the two-way interaction methanol to oil molar ratio – water content.

This suggests that all terms included in the model except for methanol to oil molar ratio had a significant effect on the biodiesel yield. The value of the regression coefficient was found to be 0.99, indicating the adequacy of the model. Lack of fit shows a p-value greater than 0.05, confirming that the model adequately explains the variation in biodiesel yield. Substituting the coefficients obtained following model was obtained:

$$\begin{aligned} \% \text{ yield of FAME} = & - 424.1 - 0.39 M_{\text{Methanol/Oil}}^2 - 0.11 W_{\text{Water}}^2 \\ & - 0.32 T^2 + 0.0043 M_{\text{Methanol/Oil}} W_{\text{Water}} \\ & - 0.052 M_{\text{Methanol/Oil}} T - 0.087 W_{\text{Water}} T \\ & + 12.04 M_{\text{Methanol/Oil}} + 6.15 W_{\text{Water}} + 23.25 T \end{aligned}$$

An error analysis was done to compute the total uncertainty in the experiment using the instrumentation. The method of

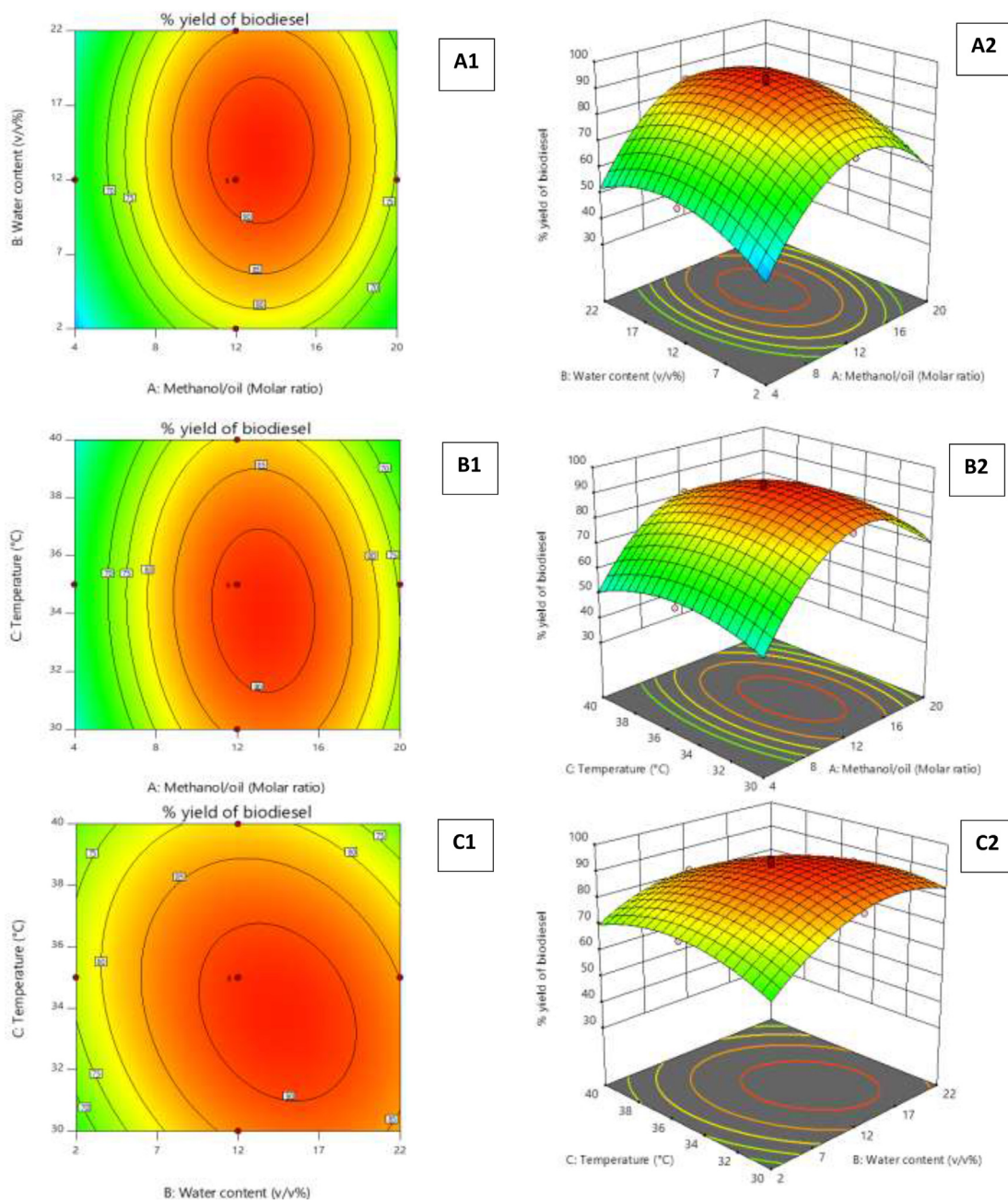
Table 2  
Analysis of variance for the biodiesel production from *Ceiba pentandra* oil.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	8416.96	935.22	333.38	0.000
Linear	3	823.42	274.47	97.84	0.000
Methanol/Oil (Molar) $M_{\text{Methanol/Oil}}$	1	566.92	566.92	202.09	0.000
Water ( $W_{\text{water}}$ )	1	195.48	195.48	69.68	0.000
Temperature (T)	1	61.02	61.02	21.75	0.001
Square	3	7403.35	2467.78	879.70	0.000
$M_{\text{Methanol/Oil}} \times M_{\text{Methanol/Oil}}$	1	1703.51	1703.51	607.26	0.000
$(W_{\text{water}}) \times (W_{\text{water}})$	1	342.77	342.77	122.19	0.000
$T \times T$	1	170.84	170.84	60.90	0.000
2-Way Interaction	3	190.19	63.40	22.60	0.000
$M_{\text{Methanol/Oil}} \times W_{\text{water}}$	1	0.96	0.96	0.34	0.572
$M_{\text{Methanol/Oil}} \times T$	1	34.57	34.57	12.32	0.006
$W_{\text{water}} \times T$	1	154.67	154.67	55.13	0.000
Error	10	28.05	2.81		
Lack-of-Fit	5	12.62	2.52	0.82	0.585
Pure Error	5	15.43	3.09		
Total	19	8445.02			

$$R^2 = 99.67\%; R^2 (\text{adj}) = 99.37\%; R^2 (\text{pred}) = 98.66\%.$$

propagation of errors was used to compute the total uncertainty of the experiment from the individual uncertainty about individual parameters [26]. The surface plot and contour plot of Fig. 3 (A1 & A2) depicts the relationship between methanol to oil ratio and water content on biodiesel yield through the doom shaped curve.





**Fig. 3.** Interaction effect of (A1&A2) Methanol to oil ratio and water content on fatty acid methyl ester (FAME) yield. (B1& B2) Methanol to oil molar ratio and temperature on fatty acid methyl ester (FAME) yield. (C1& C2) Water content and Temperature on fatty acid methyl ester (FAME) yield.

Biodiesel yield reached a maximum of up to 91% at 12:1 methanol to oil ratio and 12 vol% water content after which a decrease in yield was observed. Increasing the methanol to oil ratio beyond 12:1

shows a decrease in the yield of 75%. When the water content of 22 vol% was used biodiesel yield decreased to 85% [27].

$$\left(\frac{\Delta Y}{Y}\right) = \sqrt{\left\{ \left( \text{Uncertainty of BTE} \right)^2 + \left( \text{Uncertainty of BSFC} \right)^2 + \left( \text{Uncertainty of CO} \right)^2 \right.}$$

$$\left. + \left( \text{Uncertainty of CO}_2 \right)^2 + \left( \text{Uncertainty of NO}_x \right)^2 + \left( \text{Uncertainty of HC} \right)^2 \right\}$$

$$= \sqrt{\left\{ \left( (0.1)^2 + (1)^2 + (0.2)^2 + (0.1)^2 + (0.2)^2 + (0.1)^2 \right) \right\}}$$

= 1.05%

Fig. 3 (B1 & B2) illustrates the influence of methanol to oil ratio and temperature on biodiesel yield. Maximum of 91% conversion was obtained if methanol to oil molar ratio was kept 12:1 and temperature was maintained at 35 °C. 12:1 methanol to oil molar ratio has a significant effect on biodiesel yield and variation of temperature between 30 °C and 35 °C shows a variation in yield from 85 to 91% [28]. Fig. 3 (C1 & C2) relates the significance of temperature and water content on biodiesel yield. The maximum Biodiesel yield of 91% was obtained in 12 vol % and a temperature of 35 °C. If the temperature was increased beyond 35 °C in spite of maintaining 12 vol % water content biodiesel yield reduces up to 81% and beyond 22 vol% water content with a temperature of 35 °C yield of 85% was observed [27]. Optimal conditions of temperature, methanol to oil ratio, and water content obtained through RSM are found to be in similar lines to those of single-factor experiments, thereby strengthening the previously obtained results. Inverse matrix was used to solve the model and optimum conditions were found to be the temperature of 33 °C, methanol to oil molar ratio of about 13.3:1 with a water content of 14.5%. The model was validated and a yield of 93.1% was obtained.

Residual plots from Fig. 4 indicate the significance of the model. Normal probability plots show that predicted response from empirical correlation is found to agree with observed values. The graph of residual versus fits indicates the behavioral deviation between fits and experimental values. Plots of residuals versus order imply the random scattering pattern. The histogram shows a bell-shaped curve indicating the adequacy of the model [29].

### 3.7. Thermophysical properties of biodiesel

Different physical properties were determined for the *Ceiba pentandra* using the methyl esters obtained during the enzymatic transesterification process. Physio-chemical properties such as chemical composition, density, kinematic viscosity, moisture content, flash point, calorific value, iodine value, acid value, and fatty acid composition were determined using ASTM and AOCS methods (American Oil Chemists' Society). The GCMS results of kapok oil (*Ceiba pentandra*) methyl ester biodiesel showed the composition of 37.75% linolenic acid methyl ester, 26.21% oleic acid methyl ester, 22.43% palmitic acid methyl ester and methyl esters of other acids 4.98%. The Methyl ester produced from the transesterification process has a density of 885 kg/m<sup>3</sup> [30]. This is in accordance with the standard property of the methyl ester fuel, as indicated in ASTM D6751 standards, suggesting the density of biodiesel fuel at 15 °C must be within 860–900 kg/m<sup>3</sup> [16]. The value of kinematic viscosity of kapok oil methyl ester was found to be 4.8 mm<sup>2</sup>/s at a temperature of 40 °C [31]. This value of kinematic viscosity falls within value is well within the limit of ASTM D445 standard (1.9–6 cSt). Fatty acid content was evaluated by following the ASTM D664 method and the fuel's acid value was calculated. Diesel was found to degenerate quickly at high acid numbers [32]. The acid number is represented as the amount of KOH needed to neutralize free fatty acids in the biodiesel. The acid value of *Ceiba pentandra* was found to be 1.22 mg KOH/g of oil [6]. Flashpoint is defined as the minimum temperature at which the fuel gets ignited when there is a sufficient amount of vapors present in them. The fire point is defined as the temperature at which the fuel burns continuously

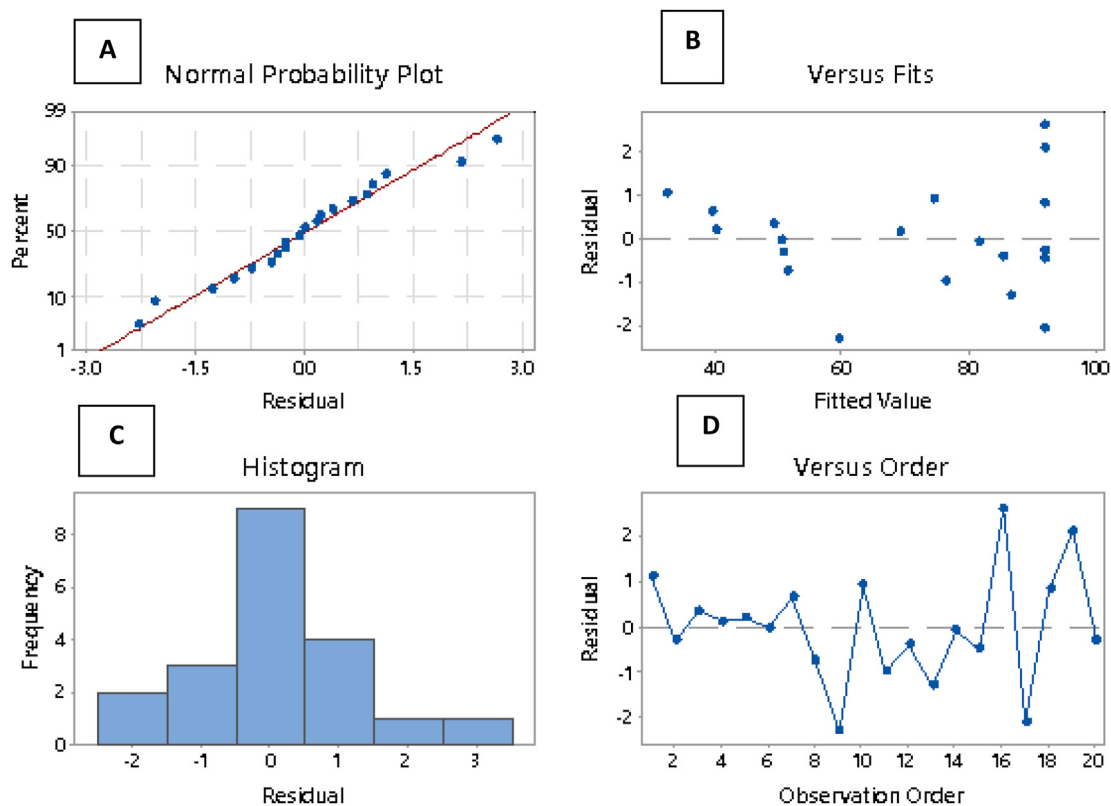


Fig. 4. Residual plots (A) Normal Probability plot (B) Residual versus Fits (C) Histogram (D) Residual versus run order.

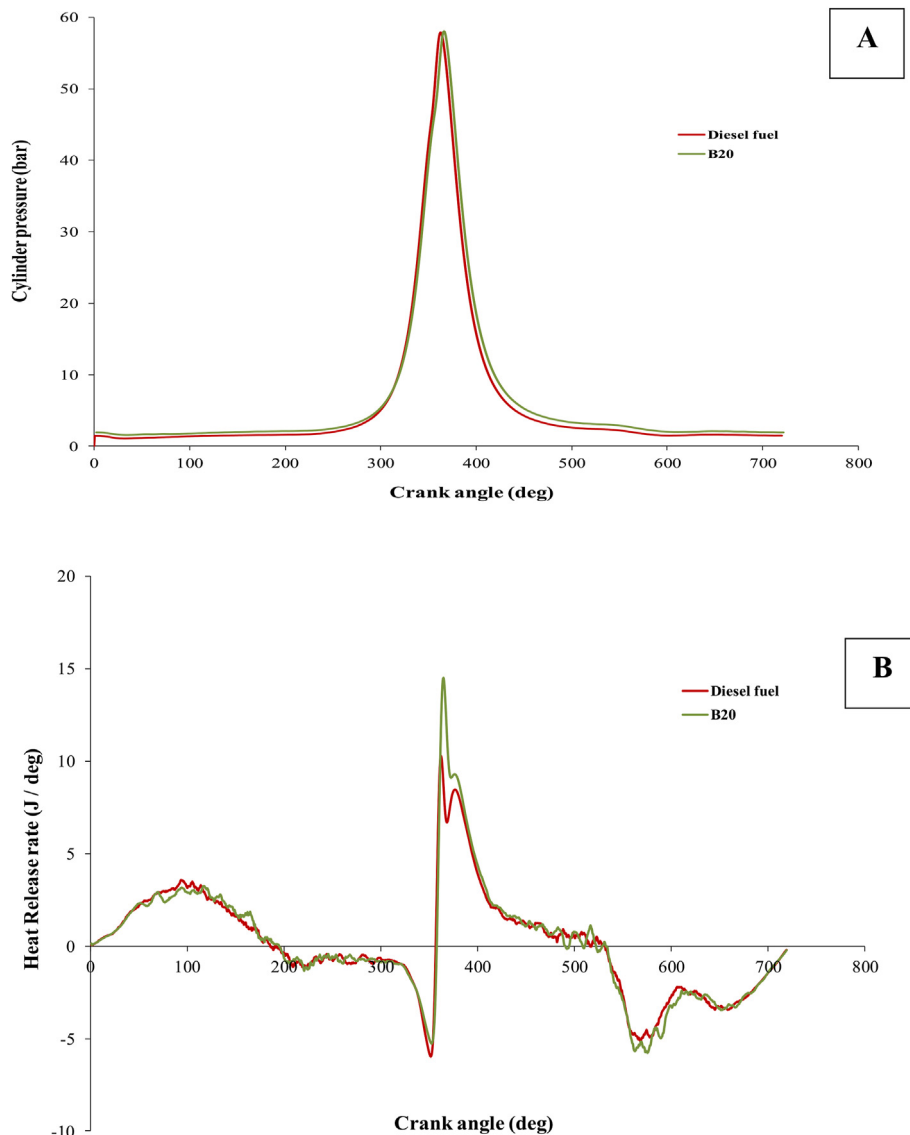


Fig. 5. In-cylinder pressure and heat release rate versus crank angle comparison for diesel and B20 at full load condition.

Table 3

Comparison of the thermophysical property of biodiesel from *Ceiba pentandra* oil with other biodiesel obtained from nonedible feedstocks are reported in the literature.

Properties of biodiesel	ASTM methods	<i>Ceiba Pentandra</i> (Present work)	<i>Hevea brasiliensis</i> [37]	<i>Jatropha curcas</i> [38]	<i>Madhuca Indica</i> [39]	<i>Azadirachta indica</i> [40]	<i>Calophyllum inophyllum</i> [24]	<i>Pongamia pinnata</i> [41]	ASTM standard values of biodiesel
Density (kg/m <sup>3</sup> ) at 15 °C	D5002-94	885	860	873	890	899	877	940	860–900
Flashpoint (°C)	D93	152	149	135	208	168	134	163	130
Cloud point (°C)	D91	-3	-	2.7	11	20	15	-10	-3 to 12
Kin.viscosity at 40 °C (cSt)	D445	4.8	4.9	4.32	5.9	5.5	3.47	4.9	1.9 to 6
Cetane number	D613	57.2	48	55.4	-	56.8	64	57	49
Acid value (mg KOH/g of oil)	D664	1.22	0.13	0.35	0.49	0.22	0.29	1.05	0.8 max
Water and sediments (%) vol	D2709	0.045	0.013	0.03	0.02	-	0.0028	0.005	0.30 max
Calorific value (kJ/kg)	DIN 51,900	38.44	43	36.5	37	-	43.28	42.13	30 min
Distillation temperature (°C)	D1160	337.4	-	-	-	-	-	-	360 max



for 5 s. This property exerts significant effects on determining fire hazards. In the present study, biodiesel's flashpoint was found to be 152 °C, much higher than the conventional diesel fuels, as determined by the ASTM D93 methodology [31]. The value is well within the limit set by ASTM D6751 standards of 130 °C. The efficacy of biodiesel during combustion is limited by its response towards lower temperatures. Filter plugging and engine starving are two methods used as a precaution to monitor the operation of biodiesel. Filter plugging is used to filter the wax formed when the engine is operating at a lower temperature. The cloud point of the biodiesel was found to be −3 °C, as estimated from ASTM D2500. The value was found to be at least 18–26 times higher than the cloud point of standard diesel values [33]. Bomb calorimeter was used to identify the calorific value of vegetable oils and their corresponding methyl

esters as 38.44 kJ/kg, using the ASTM D240 method [20]. Biodiesel produced from *Ceiba pentandra* was found to contain higher calorific values when compared with other biodiesels, but lesser than petroleum-based diesel. The lower calorific value might be due to the higher oxygen content in the fuel.

Ignition quality of the biodiesel can be estimated by evaluating the cetane number and ignition delay time, which is measured using CN after injection of fuel into the combustion chamber [34]. An increase in ignition delay time corresponds to a similar decrease in CN value. Besides, this trend contributes to gaseous and particulate exhaust emissions (PM), a phenomenon named as diesel knocking. This phenomenon arises due to incomplete combustion which also results in engine deposits. CN number was estimated for the biodiesel using ASTM D613 and was found to be 57.2. The

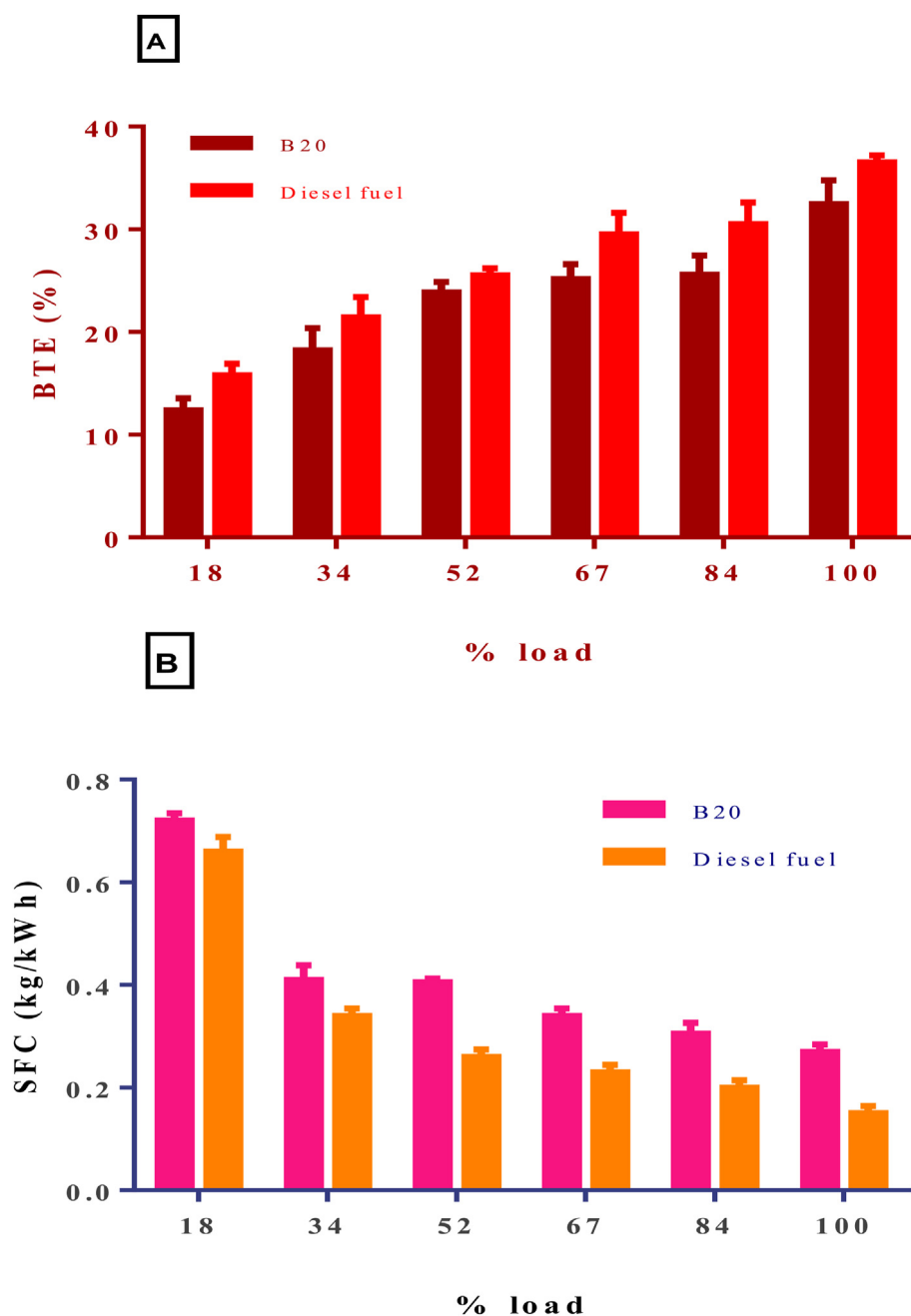


Fig. 6. Performance characteristics of *Ceiba pentandra* oil biodiesel. (BTE-Break thermal efficiency; SFC- Specific fuel consumption (A) Variation in brake thermal efficiency with load for biodiesel blend (B20) and diesel fuel. (B) Variation in specific fuel consumption with load for biodiesel blend (B20) and diesel fuel.

distillation temperature of biodiesel is 337.41 °C (ASTM-D1160) is well within the maximum allowable temperature of 360 °C [35]. The surface tension of biodiesel at 40 °C is given by 31.21 mN/m<sup>2</sup>. Evaporation and combustion are affected by fuel atomization which, in turn, depends on the surface tension and boiling point of the biodiesel. Surface tension is a physical property of the liquids, which affects the degree of atomization in fuels. The surface tension of diesel was found to be considerably lower when compared with the three biodiesels. Improved atomization with better air-fuel mixing is necessary for the complete combustion of fuels, which simultaneously increase the efficiency of the engines and reduce pollutant emissions. Fig. 5A reflects cylinder pressure with crank angle variation at full load conditions. With a 4-degree delay in crank angle, as compared to diesel, B20 attained maximum cylinder pressure at full load condition. At the above condition, the amount of energy from the burning of fuel being released for Diesel fuel and B20 has been represented in Fig. 5B. Heat release curve. There is no prominent drop in its heating value as the peak heat release rate for B20, from Fig. 5B, was in par with diesel fuel when compared to diesel. Vedharaj et al., 2013 have reported the B25 blend of the biodiesel obtained from *Ceiba pentandra* showed the same magnitude of the diesel fuel. B75 and B100 blends show a progressive decrease in the peak heat release rate. A decrease in peak heat release rate of biodiesel blends than diesel due to high viscosity and poor spray characteristics which affect the fuel atomization in the combustion process [36]. A comparison of the thermophysical

properties of various biodiesel from non-edible feedstock with *Ceiba pentandra* methyl ester is reported in Table 3.

### 3.8. Performance characteristics

#### 3.8.1. Break thermal efficiency (BTE)

The Brake output obtained from an engine for a given amount of heat applied to the engine is referred to as the Brake Thermal Efficiency (BTE). In our present study, an increase in BTE was observed concerning load and maximum BTE of 30.8% was observed in 100% load (Fig. 6A). It is also seen that diesel fuel has lower brake thermal efficiency about 13% at 100% load compared to biodiesel blend (B20) which is due to the high cetane number and the shorter ignition delay. Moreover, better atomization and lower viscosity of the fuel contribute to the complete fuel combustion thereby increasing the fuel BTE value. Similarly, Senthil Kumar et al., 2015 have reported that the increased brake thermal efficiency at load conditions up to 75% for B20 [3].

#### 3.8.2. Break specific fuel consumption (BSFC)

The efficiency of the engine can be determined by burning a specific amount of fuel in it to produce power. This parameter is defined as the brake specific fuel consumption (BSFC). The low heating value and viscosity of the biodiesel attribute to the better BSFC values for B20 blends. At 100% load, BSFC of B20 decreases by 12.5% from that of 80% load (Fig. 6B). Biodiesel BSFC was 25% higher

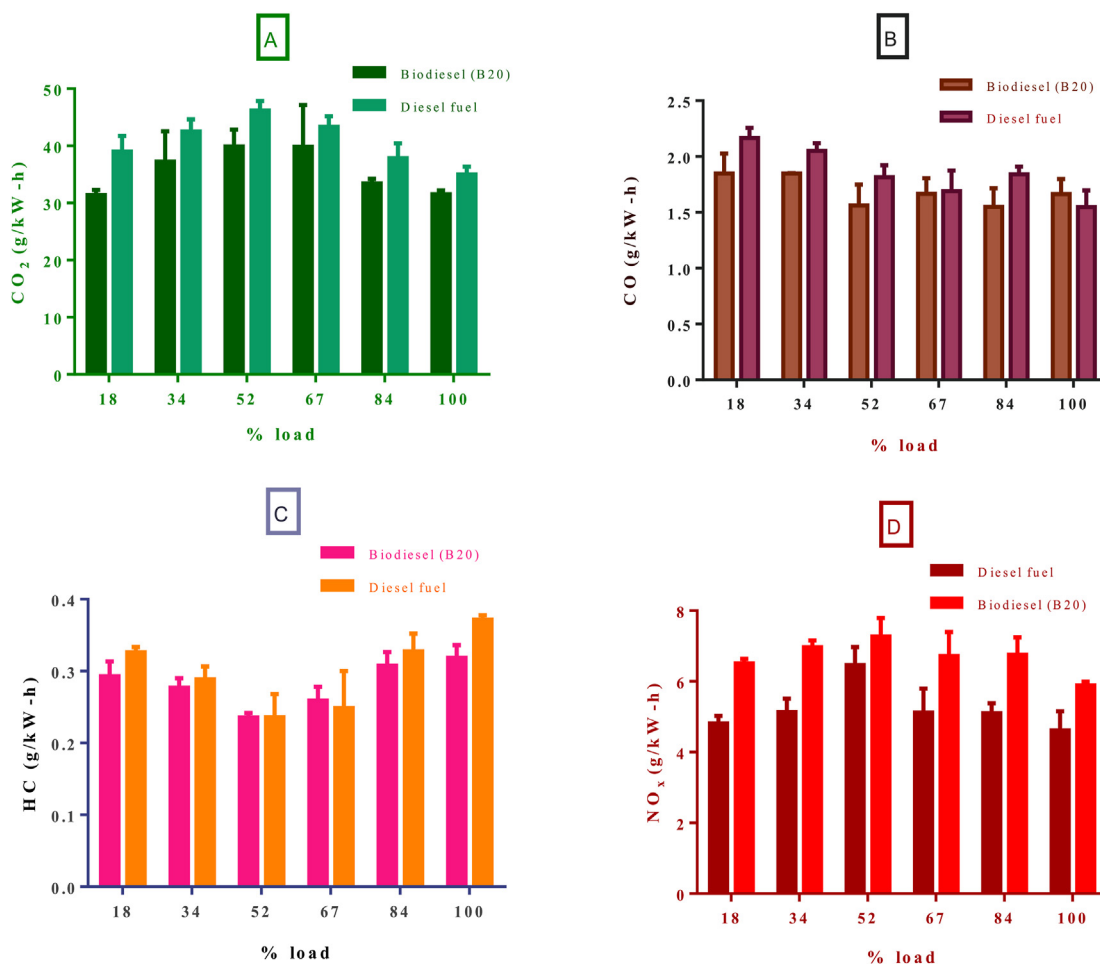


Fig. 7. Emission characteristics of *Ceiba pentandra* oil biodiesel. (A) Variation in CO<sub>2</sub> emission at various loads for diesel fuel and biodiesel blend (B20). (B) Variation in CO emission at various loads for diesel fuel and biodiesel blend (B20). (C) Variation in HC and emission at various loads for diesel fuel and biodiesel blend (B20). (D) Variation in NO<sub>x</sub> emission at various loads for diesel fuel and biodiesel blend (B20).

compared to petroleum diesel at 100% load condition. High viscosity, density, and low heat content of biodiesel blend, when compared with that of diesel attribute to the high BSFC initial load, showed maximum fuel consumption, indicating the engine efficiency. A similar result of decreased BSFC was observed in terms of increasing Brake Power [37].

### 3.9. Emission characteristics

Exhaust gas emissions have been a significant area of interest in the field of environmental sciences since the 1960s [38]. Thus, the production of alternative fuels that emits nearly lesser pollutants than conventional fuels is more important. The biodiesel obtained was used to run an engine at a blend of B20 and the exhaust emissions were trapped and analyzed.

#### 3.9.1. Carbon dioxide and carbon monoxide emission

The Kapok methyl esters have a lower amount of carbon when compared with diesel and also a higher amount of oxygen present in the biodiesel facilitates complete combustion of the biodiesel which reduces the CO<sub>2</sub> emissions [39] (Fig. 7A). suggests a 13.7% decrease in CO<sub>2</sub> emissions can be observed in comparison with Diesel for 100% load of Kapok B20. Similar to our results [10], also showed decreasing CO<sub>2</sub> emissions than that of diesel, for increasing speed. Concerning CO emissions, the B20 blend shows 5.08% decrease than diesel Fuel for 100% load (Fig. 7B), which is similar to that of a 3.5% decrease reported [5]. The decrease in CO emission is related to the complete combustion of the fuel which depends on

the oxygen requirement during combustion.

#### 3.9.2. Hydrocarbon emission

Hydrocarbon emissions occur as a result of incomplete combustion of fuel. The presence of excess oxygen in biodiesel, as well as a high cetane number of biodiesel, is responsible for decreased HC emissions [40]. In Kapok B20, at 100% load condition emission seems to reduce by 8.4% in comparison with ordinary diesel, indicating that the B20 blend emits less HC than that of diesel (Fig. 7C) [16].

#### 3.9.3. NO<sub>x</sub> emission

High temperatures at the combustion chamber of diesel engines can lead to the oxidation of the fuel-producing oxides of nitrogen [40]. The biodiesel from kapok oil (B20) shows a 31% increase in NO<sub>x</sub> emissions in comparison with that of an ordinary diesel (Fig. 7D). This could be because NO<sub>x</sub> formation is dependent on the temperature of the combustion. Indirectly, the higher amount of oxygen present in the kapok methyl esters also increases the temperature at which combustion occurs resulting in increased NO<sub>x</sub> emission [41,42]. Several researchers are reported that the emission of NO<sub>x</sub> is determined by the availability of oxygen which influences higher than diesel fuel [13,42]. Performance and emissions results for B20 blend showed that biodiesel produced from *Ceiba pentandra* can be used as an effective alternative to diesel fuel, due to its efficient engine performance and decreased toxic emissions. A comparison table of kapok oil-based biodiesel production, reported in recent literature, is given in Table 4.

**Table 4**

The comparison of biodiesel production from *Ceiba pentandra* oil using various technologies reported in the literature with the present work.

Catalysts	Methanol/oil (molar)	Temperature, °C	Time	Yield %	Thermophysical properties	Reference
Lipase	12:1	35 °C	8 h	96.4	Density:885 kg/m <sup>3</sup> kinematic viscosity: 4.8 mm <sup>2</sup> /s. The acid value: 1.22 mg of KOH/g; Cetane number: 57.2. flash point: 152 °C. The cloud point: 3 °C; Gross calorific value (kJ/kg): 38,439	Present work
9.94 wt% KOH	16.23:1	40 °C.	20.08 min at an amplitude of 32.04%.	—	—	Chuah et al., 2017 [13]
1 wt% KOH	10:1	60 °C	108 min	99.8	Density at 15 °C kg/m <sup>3</sup> : 875; Kinematic viscosity at 40 °C mm <sup>2</sup> /s: 5.4; Flash point °C: 156; Pour point °C: 8; Cloud point °C: 8; Higher heating value MJ/kg: 36.29; Sulfur content mg/kg: 0.5; Cetane number: 54; Copper strip corrosion: 1a	Kusumo et al., 2017 [9]
0.5 wt% KOH	Methanol to oil ratio of 30%	60 °C	2 h	93.3	Kinematic viscosity at 40 °C, density at 15 °C, calorific value, acid value and oxidation stability are 3.950 mm <sup>2</sup> /s, 831.2 kg/m <sup>3</sup> , 40.929 MJ/kg, 0.025 mg KOH/g and 10.01 h.	Dharma et al., 2016 [11]
0.5 wt% KOH	6:1	65 °C	3 h	—	Specific gravity 0.86; Kinematic viscosity at 40 °C in cSt: 4.2; Flash point (°C) 148; Cloud point (°C) 4; Pour point (°C): 4.4; Calorific value (MJ/kg): 39.4; Cetane number: 51	Senthil Kumar et al., 2015 [5]
1 wt% sodium methoxide	6:1	65 °C	1 h	—	—	Rashid et al., 2015 [7]
2.15 wt% KOH	1:9.85	57.1 °C	3.29 min	98.9	—	Lieu et al., 2015 [12]
1 wt% KOH	4:1	60 °C	2 h	94	Calorific value (kJ/kg): 40,064; Dynamic viscosity (mPa.s) at 40 °C: 3.8264; Flash point (°C): 202.5; Specific gravity at 15 °C 0.8852; Acid value (mg KOH/g oil): 0.16; Oxidation stability (h at 110 °C): 1.14; Cloud point (°C): 3; Pour point (°C): 5; CFPP (°C): 4; Viscosity index (VI): 216.5.	Rashid et al., 2014 [7]
1 wt% NaOH	9:1	50 °C	1 h	98.6	Kinematic viscosity: 4.61 mm <sup>2</sup> /s; Density: 876.9 kg/m <sup>3</sup> ; Flash point: 156.5 °C; Free fatty acid: 0.40 mg KOH/g.	Ong et al., 2014 [6]
10 g of KOH	—	—	—	—	Density (kg/m <sup>3</sup> ): 822; Kinematic viscosity (m <sup>2</sup> /s): 5.4 × 10 <sup>6</sup> ; Flash point (°C): 156; Pour point (°C):8; Copper strip corrosion @ 100 °C for 3 h: Not worse than no 1; Gross calorific value (kJ/kg): 36,292; Sulfur content (%): Less than 0.05; cetane index: 54.	Vedharaj et al., 2013 [13]
Supercritical Methanol	30:1	322 °C. pressure 16.7 MPa	7.9 h	95.5	Cetane index: 57; Flash point (°C): 154; Pour point (°C): 0; Acid value (mg KOH/g): 0.19; Relative density:0.88; Kinematic viscosity (mm <sup>2</sup> /s) at 40 °C: 4.3.	Ong et al., 2013 [48]
1 wt% KOH	6:1	65 °C	45 min	99.5	—	Pandian et al., 2013 [26]

#### 4. Conclusion

*Ceiba pentandra* seems to be an effective alternative source for the production of biodiesel. The production of biodiesel was carried out using enzymatic transesterification process. Maximum biodiesel yield of 96.4% was observed at 13.3:1 methanol to oil ratio, the temperature of 33 °C and water content of about 14.5%. The properties of biodiesel obtained from *Ceiba pentandra* seems to be on similar lines with that of the diesel. Performance analysis for the B20 blend shows better efficiency than that of the diesel. Emission exhaust analysis showed less CO, HC, CO<sub>2</sub> emissions than that of the diesel fuel except for the NO<sub>x</sub> emissions, which increased by 31%. A 13% decrease in CO<sub>2</sub> emission was recorded for the blend when compared to diesel. Similarly, a 5% decrease in CO emission was recorded for the biodiesel, when compared with the standard 100% diesel. Research and development on varied blends of biodiesel need to be carried out to exploit biodiesel as an alternative source of fuel in existing diesel engine. Without any key hardware alteration to the structure, *Ceiba pentandra* based biodiesel can be successfully used in the engines instead of fossil fuel-based diesel with the same performance and also improved characteristics. The performance analysis showed that a blend of *Ceiba pentandra* biodiesel with diesel fuel is a potential alternative fuel that can be used effectively in a diesel engine.

#### Credit author statement

S. Pooja: Wrote the paper & performed the enzymatic transesterification.

B. Anbarasan: Performed the analysis (Performance and emissions characteristics of produced biodiesel); Wrote the paper (Performance and emissions characteristics of produced biodiesel).

Dr. V. Ponnusami: Performed the analysis & Design of response surface analysis.

Dr. A.Arumugam: Conceived and designed the analysis, Design of enzyme technology and mesoporous material synthesis; Contributed data or analysis tools; Wrote the paper, Content and framework for the paper; Other contribution, Validation, Funding acquisition

#### Funding

We acknowledge the financial support provided by the Research board, INDIA (Grant no. ECR/2017/001038/2017–20 - SERB) in accompanying us to complete the work.

#### Authorship

We confirm that the manuscript has been read and approved by all named authors.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Acknowledgment

The authors gratefully acknowledge the financial support provided by SERB (Science & Engineering Research Board), INDIA (Grant no. ECR/2017/001038/2017–20) to carry out this research work.

#### References

- [1] V.K. Gupta, N. Atar, M.L. Yola, Z. Üstündağ, L. Uzun, A novel magnetic Fe@Au core-shell nanoparticles anchored graphene oxide recyclable nanocatalyst for the reduction of nitrophenol compounds, *Water Res.* 48 (2014) 210–217, <https://doi.org/10.1016/j.watres.2013.09.027>.
- [2] F.F. Osterstrøm, J.E. Anderson, S.A. Mueller, T. Collings, J.C. Ball, T.J. Wallington, Oxidation stability of rapeseed biodiesel/petroleum diesel blends, *Energy Fuels* 30 (2016) 344–351, <https://doi.org/10.1021/acs.energyfuels.5b01927>.
- [3] B. Mohan, W. Yang, K.L. Tay, W. Yu, Experimental study of spray characteristics of biodiesel derived from waste cooking oil, *Energy Convers. Manag.* 88 (2014) 622–632, <https://doi.org/10.1016/j.enconman.2014.09.013>.
- [4] T. Witoon, S. Bumrungsalee, P. Vathavanichkul, S. Palitsakun, M. Saisriyoot, K. Faungnawakij, Biodiesel production from transesterification of palm oil with methanol over CaO supported on bimodal meso-macroporous silica catalyst, *Bioresour. Technol.* 156 (2014) 329–334, <https://doi.org/10.1016/j.biortech.2014.01.076>.
- [5] T. Senthil Kumar, P. Senthil Kumar, K. Annamalai, Experimental study on the performance and emission measures of direct injection diesel engine with Kapok methyl ester and its blends, *Renew. Energy* 74 (2015) 903–909, <https://doi.org/10.1016/j.renene.2014.09.022>.
- [6] H.C. Ong, H.H. Masjuki, T.M.I. Mahlia, A.S. Silitonga, W.T. Chong, T. Yusaf, Engine performance and emissions using *Jatropha curcas*, *Ceiba pentandra* and *Calophyllum inophyllum* biodiesel in a CI diesel engine, *Energy* 69 (2014) 427–445, <https://doi.org/10.1016/j.energy.2014.03.035>.
- [7] U. Rashid, G. Knothe, R. Yunus, R.L. Evangelista, Kapok oil methyl esters, *Biomass Bioenergy* 66 (2014) 419–425, <https://doi.org/10.1016/j.biombioe.2014.02.019>.
- [8] N. Panneerselvam, M. Ramesh, A. Murugesan, C. Vijayakumar, D. Subramaniam, A. Kumaravel, Effect on direct injection naturally aspirated diesel engine characteristics fuelled by pine oil, *ceiba pentandra* methyl ester compared with diesel, *Transport. Res. Transport Environ.* 48 (2016) 225–234, <https://doi.org/10.1016/j.trd.2016.08.023>.
- [9] F. Kusumo, A.S. Silitonga, H.H. Masjuki, H.C. Ong, J. Siswanto, T.M.I. Mahlia, Optimization of transesterification process for *Ceiba pentandra* oil: a comparative study between kernel-based extreme learning machine and artificial neural networks, *Energy* 134 (2017) 24–34, <https://doi.org/10.1016/j.energy.2017.05.196>.
- [10] S. Dharma, M.H. Hassan, H.C. Ong, A.H. Sebayang, A.S. Silitonga, F. Kusumo, et al., Experimental study and prediction of the performance and exhaust emissions of mixed *Jatropha curcas*–*Ceiba pentandra* biodiesel blends in diesel engine using artificial neural networks, *J. Clean. Prod.* 164 (2017) 618–633, <https://doi.org/10.1016/j.jclepro.2017.06.065>.
- [11] S. Dharma, H.H. Masjuki, H.C. Ong, A.H. Sebayang, A.S. Silitonga, F. Kusumo, et al., Optimization of biodiesel production process for mixed *Jatropha curcas*–*Ceiba pentandra* biodiesel using response surface methodology, *Energy Convers. Manag.* 115 (2016) 178–190, <https://doi.org/10.1016/j.enconman.2016.02.034>.
- [12] T. Lieu, S. Yusup, M. Moniruzzaman, Kinetic study on microwave-assisted esterification of free fatty acids derived from *Ceiba pentandra* Seed Oil, *Bioresour. Technol.* 211 (2016) 248–256, <https://doi.org/10.1016/j.biortech.2016.03.105>.
- [13] S. Vedharaj, R. Vallinayagam, W.M. Yang, S.K. Chou, K.J.E. Chua, P.S. Lee, Experimental investigation of kapok (*Ceiba pentandra*) oil biodiesel as an alternate fuel for diesel engine, *Energy Convers. Manag.* 75 (2013) 773–779, <https://doi.org/10.1016/j.enconman.2013.08.042>.
- [14] L.F. Chuah, A. Bokhari, S. Yusup, J.J. Klemes, M.M. Akbar, S. Saminathan, Optimisation on pretreatment of kapok seed (*Ceiba pentandra*) oil via esterification reaction in an ultrasonic cavitation reactor, *Biomass Convers. Biorefinery* 7 (2017) 91–99, <https://doi.org/10.1007/s13399-016-0207-9>.
- [15] N. Damanik, H.C. Ong, W.T. Chong, A.S. Silitonga, Biodiesel production from *Calophyllum inophyllum*–palm mixed oil, *Energy Sources, Part A Recover Util Environ Eff* 39 (2017) 1283–1289, <https://doi.org/10.1080/15567036.2017.1324537>.
- [16] D. Soares, A.F. Pinto, A.G. Gonçalves, D.A. Mitchell, N. Krieger, Biodiesel production from soybean soapstock acid oil by hydrolysis in subcritical water followed by lipase-catalyzed esterification using a fermented solid in a packed-bed reactor, *Biochem. Eng. J.* 81 (2013) 15–23, <https://doi.org/10.1016/j.bej.2013.09.017>.
- [17] W. Xu, L. Gao, S. Wang, G. Xiao, Biodiesel production in a membrane reactor using MCM-41 supported solid acid catalyst, *Bioresour. Technol.* 159 (2014) 286–291, <https://doi.org/10.1016/j.biortech.2014.03.004>.
- [18] B. Norjannah, H.C. Ong, H.H. Masjuki, Effects of methanol and enzyme pretreatment to *Ceiba pentandra* biodiesel production, *Energy Sources, Part A Recover Util Environ Eff* 39 (2017) 1548–1555, <https://doi.org/10.1080/15567036.2017.1344747>.
- [19] C.-Y. Yu, L.-Y. Huang, I.-C. Kuan, S.-L. Lee, Optimized production of biodiesel from waste cooking oil by lipase immobilized on magnetic nanoparticles, *Int. J. Mol. Sci.* 14 (2013) 24074–24086, <https://doi.org/10.3390/ijms141224074>.
- [20] A. Arumugam, V. Ponnusami, Synthesis of SBA-15 from low cost silica precursor obtained from sugarcane leaf ash and its application as a support matrix for lipase in biodiesel production, *J. Sol. Gel Sci. Technol.* 67 (2013), <https://doi.org/10.1007/s10971-013-3070-1>.
- [21] Y. Li, G. Zhou, C. Li, D. Qin, W. Qiao, B. Chu, Adsorption and catalytic activity of

- Porcine pancreatic lipase on rod-like SBA-15 mesoporous material, *Colloids Surfaces A Physicochem Eng Asp* 341 (2009) 79–85, <https://doi.org/10.1016/j.colsurfa.2009.03.041>.
- [22] C. Fushimi, A. Umeda, Comparison of biodiesel production by a supercritical methanol method from microalgae oil using solvent extraction and hydrothermal liquefaction processes, *Energy Fuels* 30 (2016) 7916–7922, <https://doi.org/10.1021/acs.energyfuels.6b00904>.
- [23] J. Sebastian, C. Muraleedharan, A. Santhiagu, Enzyme catalyzed biodiesel production from rubber seed oil containing high free fatty acid, *Int. J. Green Energy* 14 (2017) 687–693, <https://doi.org/10.1080/15435075.2017.1318754>.
- [24] D. Madhu, R. Arora, S. Sahani, V. Singh, Y.C. Sharma, Synthesis of high-quality biodiesel using feedstock and catalyst derived from fish wastes, *J. Agric. Food Chem.* 65 (2017) 2100–2109, <https://doi.org/10.1021/acs.jafc.6b05608>.
- [25] A. Arumugam, V. Ponnusami, Biodiesel production from Calophyllum inophyllum oil using lipase producing *Rhizopus oryzae* cells immobilized within reticulated foams, *Renew. Energy* 64 (2014), <https://doi.org/10.1016/j.renene.2013.11.016>.
- [26] L.F. Bautista, G. Vicente, Á. Mendoza, S. González, V. Morales, Enzymatic production of biodiesel from nanochloropsis gaditana microalgae using immobilized lipases in mesoporous materials, *Energy Fuels* 29 (2015) 4981–4989, <https://doi.org/10.1021/ef502838h>.
- [27] E. Betiku, A.M. Akintunde, T.V. Ojumu, Banana peels as a biobase catalyst for fatty acid methyl esters production using Napoleon's plume (*Bauhinia monandra*) seed oil: a process parameters optimization study, *Energy* 103 (2016) 797–806, <https://doi.org/10.1016/j.energy.2016.02.138>.
- [28] A.H. Mohammad Fauzi, N.A.S. Amin, R. Mat, Esterification of oleic acid to biodiesel using magnetic ionic liquid: multi-objective optimization and kinetic study, *Appl. Energy* 114 (2014) 809–818, <https://doi.org/10.1016/j.apenergy.2013.10.011>.
- [29] S. Mohapatra, S. Padhy, P.K. Das Mohapatra, H.N. Thatoi, Enhanced reducing sugar production by saccharification of lignocellulosic biomass, *Pennisetum* species through cellulase from a newly isolated *Aspergillus fumigatus*, *Bioresour. Technol.* 253 (2018) 262–272, <https://doi.org/10.1016/j.biortech.2018.01.023>.
- [30] S.T. Keera, S.M. El Sabagh, A.R. Taman, Castor oil biodiesel production and optimization, *Egypt J Pet* 27 (2018) 979–984, <https://doi.org/10.1016/j.ejpe.2018.02.007>.
- [31] S.O. Kareem, E.I. Falokun, S.A. Balogun, O.A. Akinloye, S.O. Omeike, Enzymatic biodiesel production from palm oil and palm kernel oil using free lipase, *Egypt J Pet* 26 (2017) 635–642, <https://doi.org/10.1016/j.ejpe.2016.09.002>.
- [32] J.G. Veneral, D.L.R. Junior, M.A. Mazutti, F.A.P. Voll, L. Cardozo-Filho, M.L. Corazza, et al., Thermophysical properties of biodiesel and related systems: low-pressure vapor + liquid equilibrium of methyl/ethyl soybean biodiesel, *J. Chem. Thermodyn.* 64 (2013) 65–70, <https://doi.org/10.1016/j.jct.2013.05.001>.
- [33] S. Sadaf, J. Iqbal, I. Ullah, H.N. Bhatti, S. Nouren, J. Nisar, et al., Biodiesel production from waste cooking oil: an efficient technique to convert waste into biodiesel, *Sustain Cities Soc* 41 (2018) 220–226, <https://doi.org/10.1016/j.scs.2018.05.037>.
- [34] R. Selaimia, A. Beghuel, R. Oumeddour, The synthesis of biodiesel from vegetable oil, *Procedia - Soc Behav Sci* 195 (2015) 1633–1638, <https://doi.org/10.1016/j.sbspro.2015.06.221>.
- [35] G.M. Chupka, L. Fouts, J.A. Lennon, T.L. Alleman, D.A. Daniels, R.L. McCormick, Saturated monoglyceride effects on low-temperature performance of biodiesel blends, *Fuel Process. Technol.* 118 (2014) 302–309, <https://doi.org/10.1016/j.ijnaoe.2018.02.002>.
- [36] S. Vedharaj, R. Vallinayagam, W.M. Yang, C.G. Saravanan, S.K. Chou, K.J.E. Chua, et al., Reduction of harmful emissions from a diesel engine fueled by kapok methyl ester using combined coating and SNCR technology, *Energy Convers. Manag.* 79 (2014) 581–589, <https://doi.org/10.1016/j.enconman.2013.12.056>.
- [37] J.N. Nair, A.K. Kaviti, A.K. Daram, Analysis of performance and emission on compression ignition engine fuelled with blends of Neem biodiesel, *Egypt J Pet* 26 (2017) 927–931, <https://doi.org/10.1016/j.ejpe.2016.09.005>.
- [38] J.N.B. Bell, S.A. Power, N. Jarraud, M. Agrawal, C. Davies, The effects of air pollution on urban ecosystems and agriculture, *Int. J. Sustain. Dev. World Ecol.* 18 (2011) 226–235, <https://doi.org/10.1080/13504509.2011.570803>.
- [39] C. Woo, S. Kook, E.R. Hawkes, P.L. Rogers, C. Marquis, Dependency of engine combustion on blending ratio variations of lipase-catalysed coconut oil biodiesel and petroleum diesel, *Fuel* 169 (2016) 146–157, <https://doi.org/10.1016/j.fuel.2015.12.024>.
- [40] V.K. Belagur, V.R. Chitimi, Few physical, chemical and fuel related properties of calophyllum inophyllum linn (honne) oil and its blends with diesel fuel for their use in diesel engine, *Fuel* 109 (2013) 356–361, <https://doi.org/10.1016/j.fuel.2013.02.015>.
- [41] K. Nanthagopal, B. Ashok, R.T.K. Raj, Influence of fuel injection pressures on Calophyllum inophyllum methyl ester fuelled direct injection diesel engine, *Energy Convers. Manag.* 116 (2016) 165–173, <https://doi.org/10.1016/j.enconman.2016.03.002>.
- [42] M. Bhuiya, M. Rasul, M.M. Khan, N. Ashwath, Performance and Emission Characteristics of Binary Mixture of Poppy and Waste Cooking Biodiesel, vol. 110, 2017, <https://doi.org/10.1016/j.egypro.2017.03.179>.