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Optimization of Biodiesel Production from Mixed Ceiba Pentandra and Rice Bran Oil Assisted by Ultrasound

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Abstract

The current research aims to examine the feasibility of production of biodiesel with non-edible mixed oils, Ceiba Pentandra Oil (CPO) and Rice Bran Oil (RBO).Several blends of CPO and RBO, ranging from 10:90 to 50:50% w/w were put under evaluation. The transesterification process variables of CP50RB50 as the suitable blend using exposure surface methodology, they were enhanced (RSM). The proportion of methanol to gasoline, the time of reaction and the concentration of the catalyst were both the key process parameters tested.An response surface transesterification process conditions such as KOH catalyst concentration are preferable: 0.83 percentage wt, methanol to oil ratio: 55.36%, reacted for 18.58 min18.58 min, with methyl ester yield of 98.7 %. The result indicates that the CP50RB50 methyl ester properties satisfy the biodiesel requirements as laid in standards, ASTM D6751 and EN 14214.

Keywords: Biodiesel, Ceibapentandra, rice bran, mix oil, optimization, RSM

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1 Introduction

With a greater understanding of the environment, biodiesel is considered as one of the alternativesto fossil fuel due to its adaptability with petro diesel. As a biodegradable nontoxic fuel that contains no sulphur (unlike its petro diesel counterpart), biodiesel so several diesel engines with smaller or no considerable modifications will be used at all [1-5]. Among the various raw materials which can be used to produce biodiesels are animal fats, Vegetable oils that become edible and not edible, and even harmful oils including cooking. Due to the growing concern with the issue of food shortage all across the world, microalgae and low-grade non-edible vegetable oils have been thoroughly researched as a substitute to the common edible oils, namely sunflower, soy bean and palm oils [6-9].

Ceibapentandra is a plant that grows in humid, tropical regions. With the ability to produce anywhere between Ceibapentandra is also known as a plant that has a short harvesting duration of 4 to 5. 1000 to 4000 seed pods at a time, where each pod contains about 250 seeds months[10]. The seed of *C. pentandra*normally contains 25-28% oil; where saturated fatty acid is around 17.15%, while the unsaturated fatty acid amounts to approximately 76.32%[11]. Another type of non-edible rice bran oil, that is used in this investigation, was (RBO). Low free fatty acid (FFA) content of RBO; containing approximately 16 to 32wt% of oil and is obtained from the outer layers of the rice grain. Due to its low free fatty acid (FFA) content, RBO has become one of the desirable candidates to be used as raw material to produce biodiesel[12].

In addition to reducing the dependency on a particular type of raw material, blending different types of raw materials can also be viewed as an effort to further improve the properties of biodiesel. It is shown in a study conducted by Dharma et al. (2016) that now the oxidation stability of biodiesel can be improved by blending *Jatropha curcas* and *Ceibapentandra* together [13]. Milano et al (2018), revealed that a blended mixture consist of 70% waste cooking oil and 30% *Calophyluminophylum* has favorable cold flow properties, better physicochemical properties, and higher oxidation. It is found that traits such as density and viscosity of biodiesel produced from the oil mixtures are somewhere between those of the biodiesels obtained from individually processed oils [14].

Ultrasound methods have been widely used by researchers for biodiesel synthesis in the last decade [15,16]. During an ultrasound-assisted reaction, higher biodiesel yield can be usually produced at a faster speed using less amount of catalyst by consuming significantly less energy due to the cavitation phenomenon of the ultrasonication [16-19].

Response Surface Methodology (RSM) is among the methods of optimization that in order to minimise biodiesel costs, streamlining the conditions of the parameters can be used. RSM has been shown to be an efficient method optimize the process parameters for biodiesel production

[13]. Here, the experiments were designed by considering several critical factors related to the production of biodiesel. Among the parameters considered were methanol to the proportion of petroleum, catalystconcentration used, and reaction time [20,21].

The objective of this study is to create biodiesel from Ceibapentandra blended oil (CPO) and rice bran oil (RBO).In this present study, CPO and RBO were mixed using various mixing ratios and the properties of each individual mixture are then consequently evaluated. The Response Surface Methodology (RSM) was used in the effort to improve the critical parameters used in the transesterification process of the optimal CPO-RBO blend. The physicochemical properties (kinematic viscosity, density, and (acid value) of biodiesel was evaluated in line with the specifications of ASTM D6751.

2 Experimental Methodology

2.1 Methodology

Rice bran oil (RBO) was procured from ScienfieldSdn. Bhd., Selangor, Malaysia. Potassium hydroxide and methanol were purchased from Chemolab, Malaysia. *Ceibapentandra* oil was obtained from Cilacap, Centre Java, Indonesia. All chemicals for the experiments were 99% pure and of analytical chemistry grade. 250 ml borosilicate glass beakers and covered with aluminum foil lid were used as the biodiesel production reactor. The ultrasonic device Qsonica (Q500-20) was operated at 500 Watts and at the frequency of 20 kHz, attached to a 1-inch diameter probe.The amplitude of the probe was set to be at 40% during the reaction, with pulse set at 5 seconds on and 2 seconds off. The setup of the experiment involving the ultrasound system is presented in Fig. 1.

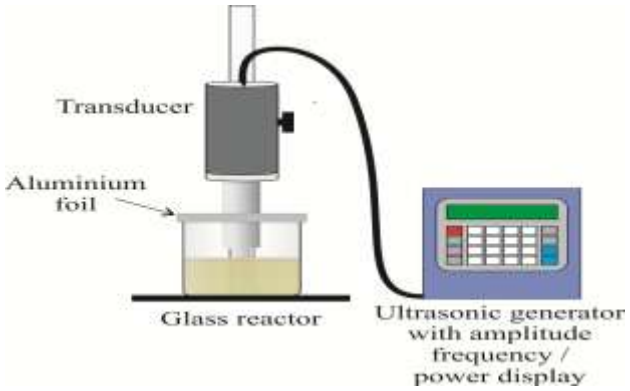


Fig. 1. Biodiesel production using ultrasound equipment system.

2.2 CPO and RBO Crude Oils Blending Procedure

The *Ceibapentandra* oil (CPO) and rice bran oil (RBO) were prepared in different blend ratios (10:90, 20:80, 30:70, 40:60 and 50:50% RBO:CPO w/ w). A mechanical stirrer was used in order to stir each mixture for 30 minutes. Several properties, namely kinematic 40 °C viscosity, acid value, and 15 °C density were determined using ASTM D6751 standard. This is done prior to the selection of the best RBO-CPO blend to be used in further study.

2.3 Esterification Process

In the effort to lower the free fatty acid (FFA) content, acid-catalyzed esterification procedure was conducted by adding 1 vol.% of sulfuric acid into 30g CPO-RBO oil mixture; while keeping the ten percent ratio of methanol to oil . The ultrasound amplitude during the maximum immersion process was retained at seven percent. The reaction time varied accordingly during the esterification process at 10, 15, 20, and 25 minutes. After each esterification process, the esterified oil was carefully forced into a totally seperate funnel to settle for 6 hours under gravity.

2.4 Transesterification Process

Initially, 30 around on a 250 ml glass beaker, g of esterified CP50RB50 was poured. Potassium hydroxide (0.5, 0.75, and 1.00), was dissolved in methanol (30, 45, and 60) and load into 30 g esterified oil and reacted at 10, 15, and 20 minutes, respectively. The amplitude of the ultrasound was maintained at 40% during the transesterification process. The sample was poured into at the end of the method of transesterification, a separation funnel to let it settle for 6 hours.After discharging the excess methanol and catalysts, distilled water was used to wash the product with the intention the extraction of impurities. The remaining methanol and water[22] were subsequently expelled from the liquid via the use of rotary evaporator maintained at the temperature of 65°C. The product was then filtered using Whatman filter paper.

2.5 Response Surface Methodology (RSM)

The optimum parameters of the procedure (i.e. methanol to oil, reaction time and concentration of its catalyst) for the oil mixture of CPO-RBO oil were determine by using RSM which was based on the experimental design from Box-Behnken from Design-Expert® software version 11. The coded and

uncoded parameters are presented in **Table 1**. The quadratic model is given by the following polynomial, shown in Eq. 1:

$$Y = a_0 + \sum_{i=1}^{k} a_i x_i + \sum_{i=1}^{k} a_{ii} x_i^2 + \sum_{i>1}^{k} a_{ij} x_i x_j + e \quad (1)$$

In Eq. 1, *Y* is the response factor; while the intercept and the first order coefficient are symbolized by a_0 and $a_{i,}$ respectively. Here,x_i the ifactor is the independent aspect and aii is the quadratic ifactor coefficient. For the the relationship amongst I and j indicators is personified by aij, while k is the number of scenarios and e is the experimental error allocated to the response factor *Y*.

Table 1. Selected variable parameters and levels

Parameters	Unit	Level	
		Minimum	maximum
X_1: Methanol to oil ratio	wt.%	30	60
X_2: Catalyst concentration	%	0.5	1
X_3 : Reaction Time	min	10	20

2.6 Physicochemical Properties of CPO-RBO Methyl Ester

Chemical-physical characteristics of Ceibapentandra crude oil (CPO), rice bran crude oil (RBO) and 50 percent Ceibapentandra mixed with 50 percent rice bran methyl ester were calculated using ASTM D6751 (CP50RB50ME).The density of CP50RB50ME using the DM40 LiquiPhysics TM weighing balance from Mettler Toledo, Greifensee, Switzerland, was determined, whereas Parr 6200 Isoperibol (USA) was utilized to find the calorific value. Stabinger Viscometer Anton Paar SVM3000 (Graz, Austria) was utilized in measuring the viscosity of biomechanics. Using TENSOR 27, Bruker Optics Inc, the CP50RB50ME was presented in terms of Fourier transform infrared (FT-IR) (USA). Profit of Acid of the methyl ester was determined using rondo 20 Automatic Titration 20 (Mettler Toledo, Switzerland). The yield (cents per dollar) of CP50RB50ME was directly measured using the formula on **Eq. 2[23]**,[24]:

$$\text{CP50RB50ME yield} = \frac{\text{Weight of CP50RB50ME (g)}}{\text{Weight of CP50RB50ME crude oil used (g)}} \times 100\% \quad (2)$$

3 Results and Discussions

3.1 Properties and Analysis of the Oil Mixtures

Table 2 shows the the physicochemical showcases of the CPO, RBO and various blend ratios of those oils. From **Table 2**, it apparent that the kinematic viscosity and the density of CPO60RBO40 and CPO50RBO50 are lower than the value possessed by RBO. However, as the percentage of RBO increases in the CPO+RBO blend, it is found that the blend resulted in a lower acid value compared to the acid value of CPO. From the results, it is shown that the blend of CPO50RBO50 resulted in compared to lower kinematic viscosity, density, and acid value the other blend ratios of CPO+RBO. Similar reporting can also be found from Fadhil et al (2017), where the blending of 50% waste fish oil (WFO) and 50% castor seed oil (CSO) resulted in lower flash point, kinematic viscosity, density, and acid value [23].

Table 2. Properties of CPO, RBO and mixed CPO+RBO.

Property	Unit	CPO	RBO	CPO90% +RBO10%	CPO80% +RBO20%	CPO70% +RBO30%	CPO60% +RBO40%	CPO50% +RBO50%
Kinematic viscosity at 40°C	mm^2/s	34.90	40.97	41.62	41.32	41.17	40.95	40.31
Density at 15°C	kg/m^3	922.00	924.60	925.00	924.80	924.70	924.60	924.30
Acid value	mg KOH/g	22.20	1.82	23.44	21.67	19.64	18.08	11.08

3.2 Reaction Time's Effect on the Acid Value

Data collection on determination of acid value by means of reaction time for esterified CP50RB50 oil was triplicated and average value was taken for plotting the the graph in Fig. 2. The time interval changed from 10 to 25 milliseconds, it is shownthat acid value significantly decrease 10 to 20 minutes, but after 20 minutes for reaction time, it increases. The increasing of acid value after the 20 minutes reaction time might be due tothe side reactions of H_2SO_4 at longer time that leading to a slow reaction to equilibrium. The acid value of 1.67 mg KOH/g was however taken as the lowest value,

obtained at a total time of 20 minutes and was perfectly adequate for the transesterification process.

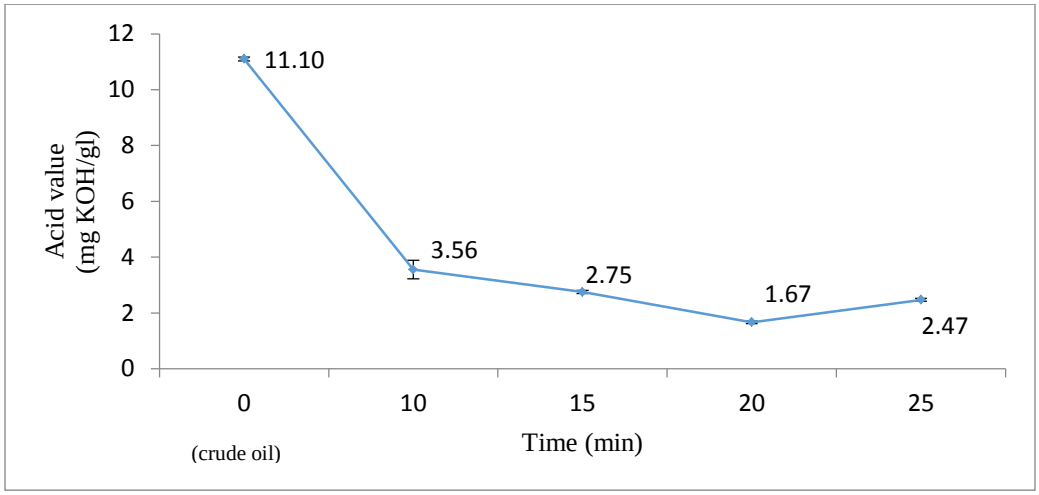


Fig. 2. The effect of esterification reaction time to the acid value of CP50RB50 oil

3.3 Response Surface Methodology

In this study, process parameter for obvious reasons, the methanol / oil ratio, the reaction time and the catalyst concentration used were identified. The experimental matrix was triggered using the experimental design of Box-Behnken (Table 3). The quadratic worldview for predicted RB50CP50ME conversion is shown in the following **Eq. 3**:

$$\text{Yield}\ (\%) = 14.92337 + 1.26666\chi_1 + 73.20750\chi_2 + 1.9751\chi_3 - 0.200467\chi_1\chi_2 + 0.0046\chi_1\chi_3 - 0.226\chi_2\chi_3 - 0.010708\chi_1^2 - 34.91\chi_2^2 - 0.054945\chi_3^2 \quad (3)$$

The model has been tested using Variance Analysis (ANOVA) in 95% confidence interval (Table 4). It is shows that quadratic regression model is significance with the p-value is < 0.0001 (Santos et al., 2013). The value of determination coefficient (R^2= 0.986) indicates that only 1.1 % data cannot explain by the model (Rabelo, 2015). With the small difference of less than 0.2 between the predicted R^2 (0.928) and adjusted R^2 (0.968), it can be implied that the regression is benefit greatly to. The predicted optimum yield for CP50RB50ME based on the developed model were time of reaction (18.58 min), concentration of KOH catalyst (0.987 wt.%), and methanol/oil ratio (55.4%) resulted 98.7% yield.

Table 3. Experimental conditions for methyl ester conversion using Box-Behnken design

Run	X_1: Methanol to oil ratio (%)	X_2: Catalyst concentration (wt.%)	X_3 : Reaction Time (min)	Experimental yield (%)	Prediction yield (%)
1	60	0.75	20	98.13	98.27
2	60	0.75	10	94.20	93.94
3	45	1.00	20	96.64	96.65
4	45	0.75	15	97.16	96.56
5	60	1.00	15	96.82	96.65
6	30	0.50	15	85.63	85.79
7	60	0.50	15	93.67	93.94
8	30	0.75	20	90.68	90.93
9	45	0.75	15	96.83	96.56
10	45	0.75	15	96.92	96.56
11	45	1.00	10	93.16	93.58
12	45	0.50	10	88.82	88.80
13	45	0.75	15	96.67	96.56
14	45	0.5	20	93.43	93.00
15	30	0.75	10	88.13	87.98
16	30	1.0	15	91.79	91.51
17	45	0.75	15	95.26	96.56

3.4 Effects of the Methanol to Oil Ratio, Reaction Time of Polymerization and Catalyst Concentration on the Jerome of Methyl Ester

Fig. 3(a) shows the three dimension response curves for the reaction time versus catalyst concentration interaction. From **Fig.3 (a)**, it shows that the conversion efficiency increased significantly with reaction time 10 to 18 min and then slightly decreased. The decrease of conversion efficiency during longer reaction time is explained by the reversible nature of the transesterification process itself.

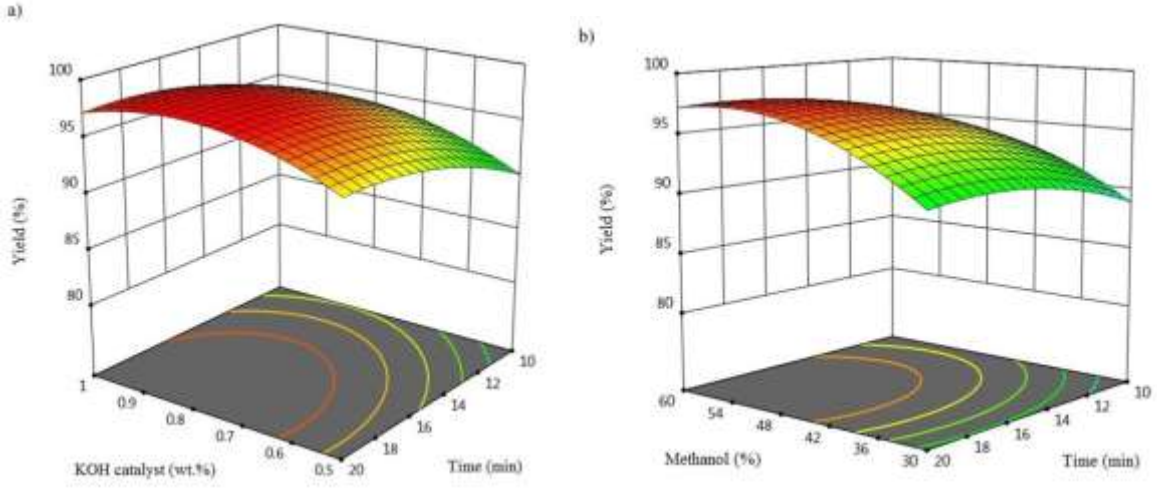
The three dimension surface plot for relationship effects among reaction time and methanol to oil ratio is shown in **Fig. 3(b)**. In order to study the relationships between the two parameters, the methanol to oil ratio were varied in the range of 30% to 60%. From the figure, it seen that the yield of

Table 4. Analysis of variance (ANOVA) for the regression model

Source	Sum of	df	Mean	F-value	p-value	
Model	211.68	9	23.52	54.83	< 0.0001	significant
X_1- Methanol to oil ratio	88.36	1	88.36	206.00	< 0.0001	
X_2- Catalyst concentration	35.55	1	35.55	82.87	< 0.0001	
X_3- Reaction Time	26.54	1	26.54	61.87	0.0001	
X_1X_2	2.26	1	2.26	5.27	0.0553	
X_1X_3	0.4761	1	0.4761	1.11	0.3271	
X_2X_3	0.3192	1	0.3192	0.7442	0.4169	
X_1^2	24.44	1	24.44	56.99	0.0001	
X_2^2	20.04	1	20.04	46.73	0.0002	
X_3^2	7.94	1	7.94	18.52	0.0036	
Residual	3.00	7	0.4289			
Lack of Fit	0.7382	3	0.2461	0.4347	0.7402	not significant
Pure Error	2.26	4	0.5661			
Cor Total	207.239585	16				
R^2	0.986					
Adjusted R^2	0.968					
Predicted R^2	0.928					

CP50RB50ME increases corresponding with term of reaction and ratio of methanol to natural gas. It can be seen in the plot that after touching a peak value, the yield of CP50RB50ME slightly decreases. The study found the ratio of methanol to oil that provides the highest yield of methyl ester is 55.36%.

Fig. 3(c) it shows the relation between the methanol to oil ratio and the catalyst concentration. From **Fig.3(c)**, it shows that the conversion to methyl ester increased significantly when KOH catalyst changed from 0.5 wt.% to 0.8 wt.%. The optimal yield was achieved with catalyst 0.83 wt.%



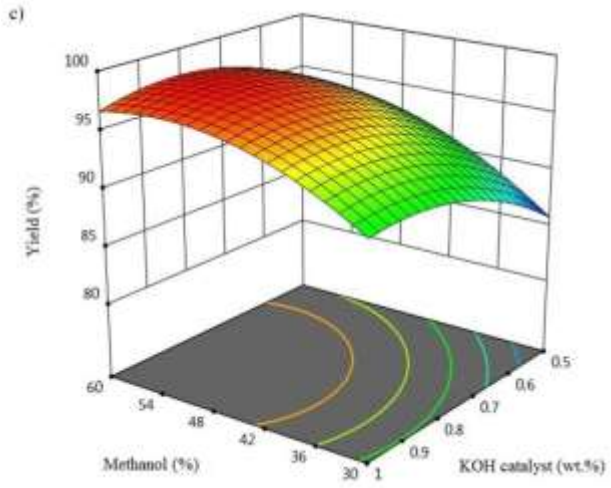


Fig. 3. Three-dimensional response surface plots of (a) time vs catalyst concentration, (b) time vs methanol to oil ratio and (c) methanol to oil ratio vs catalyst concentration.

3.5 Analysis of CP50RB50ME

The obtained CP50RB50ME was characterized using the FTIR spectra shown in **Fig. 4**. Here, it can be seen that the CP50RB50ME consists of long-chain fatty acid methyl esters. Absorption peaks at 880, 1014, 118, 1170, 1196, 1245, 1361, 1436 and 1463 cm^{-1}, which are normally referred to the 'fingerprint' region of biodiesel can beobserved in the **Fig. 4.** Meanwhile, the absorption peak at 1436 cm^{-1} and 1196 cm^{-1}in the figure correspond to the lopsided stretch of-CH3 and O-CH3 stretching, respectively. In particular, the decrease in wave energy between 1000 cm-1 and 1800 cm-1 helps to determine methyl ester content in the CP50RB50ME.

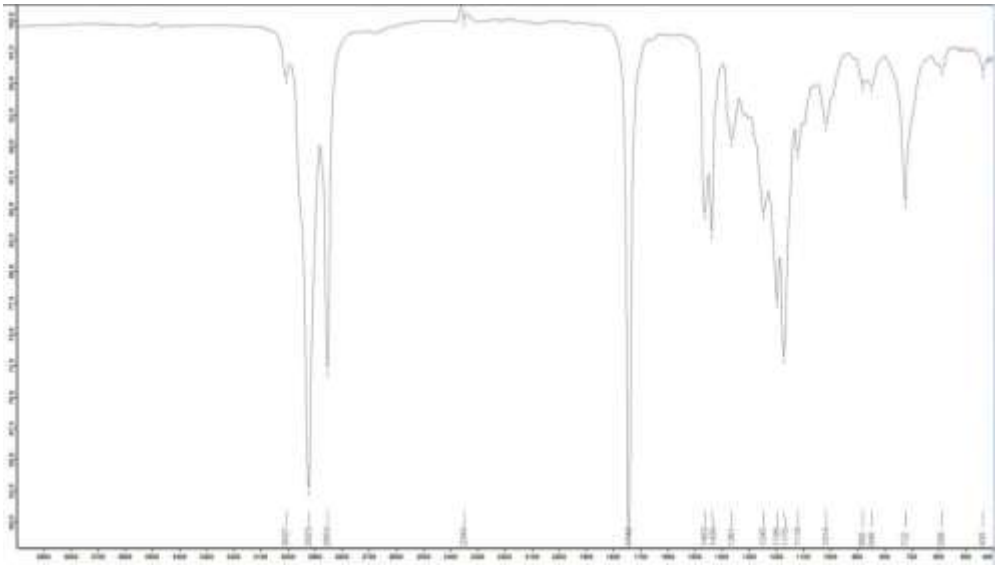


Fig. 4.FTIR spectra of CP50RB50ME.

The properties of CP50RB50ME produced using ultrasound were determined accordingly and presented in**Table 5**; and it can be seen that the properties the values stipulated by the ASTDM D67511 are within the system of values standards. Hence, the biodiesel obtained from the blend of CP50RB50 is suitable to be used in diesel engines.

Table 5. Physical and chemical properties of CP50RB50ME

Property	Unit	Standard test method	ASTM D6751	EN14214	Petro Diesel	WFO50 CSO50 ME[b]	KO50L O50ME[c]	CP50RB50 ME[a]
Kinematic viscosity at 40°C	mm^2/s	D 445	1.9-6.0	3.5–5.0	2.86	4.07	5.61	4.7747
Density at 15°C	kg/m^3	D 1298	860-880	860–900	833	898	8.92	878
Acid value	mg KOH/g	D 664	Max. 0.5	Max 0.5	0.06	0.06	-	0.227
Higher heating value	MJ/kg	D 975	Min. 35	35	45.82	-	37.89	39.5712

[a] Result, [b][25] , [c] [26]

4 Conclusions

The physicochemical properties of *Ceibapentandra* (CPO) and rice bran oil (RBO) consist of various blending ratio of 10:90 to 50:50% w/w were evaluated. The CP50RB50 was considered to be the most effective blend to be used in the transesterification of the alkali base, and the method was optimised focused on the Box-Behnken statistical methodology using methodology of the Response Surface (RSM). Using the optimized values of 0.83% for KOH w/w, 53.36% for methanol to oils ratio, and 18.58 minutes of reaction time, and optimum CP50RB50ME yield of 98.70 wt% was able to be achieved. The properties of the biodiesel obtained correspond with the destination in ASTM D67511 standards. Hence, it can be concluded that CP50RB50ME can be used as a substitute to petro diesel.

Conflict of Interest

There is no conflict of interest.

Acknowledgments

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