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# Microwave irradiation-assisted transesterification of ternary oil mixture of waste cooking oil – *Jatropha curcas* – Palm oil: Optimization and characterization

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 Response surface  
 methodology

**Abstract** Palm oil is an incredibly efficient crop, but our dependence on this crop as a primary biodiesel feedstock has threatened food insecurity as it is still perceived as the main source for vegetable oil throughout the world rather than being utilized for fuel. Therefore, the idea of utilizing non-edible food crops and waste vegetable oils could help to overcome the major problems faced by the first generation of biodiesel feedstock. In this study, a ternary oil mixture comprises 50 vol% of waste cooking oil, 15 vol% of *Jatropha curcas* oil and 35 vol% of palm oil were premixed and developed into biodiesel via esterification and microwave irradiation-assisted transesterification using a modified household microwave in the presence of methanol and potassium hydroxide catalyst. The parameters affecting biodiesel yield were optimized via response surface methodology based on central composite design. The operating parameters were optimized at 0.78 wt% of catalyst concentration, 9.86:1 of methanol/oil molar ratio, 10.5 min of reaction time and 478 rpm of stirring speed with the predicted and experimental yields are at 96.81 and 96.91 %, respectively. The results indicate that the synergistic mixture of ternary oil in WJP biodiesel gives better cold flow properties as well as improves oxidation stability and cetane number.

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## Nomenclature

WC	Waste Cooking Oil	CCD	Central Composite Design
JC	<i>Jatropha curcas</i> Oil	WC50-JC15-PO35	Ternary oil mixture containing 50 vol% of waste cooking oil, 15 vol% <i>Jatropha curcas</i> oil and 35 vol% palm oil
PO	Palm Oil	WJP	Waste cooking oil – <i>Jatropha curcas</i> – palm biodiesel
FFA	Free Fatty Acid		
FAME	Fatty Acid Methyl Ester		
RSM	Response Surface Methodology		

## 1. Introduction

Exponentially growth of energy demand in transportation sector has raised concerns to energy security and greenhouse gas (GHG) emissions, and there is a broad of consensus that transportation sector will continue to grow in the coming decades driven by the expanding commercial transportation activity. In fact, it is estimated that the global transportation-related energy demand is expected to grow by more than 25 percent from 2017 to 2040 [1]. Besides that, about 25 percent of carbon dioxide emissions from this sector are caused by the burning of petroleum-based fossil fuels, mainly gasoline and diesel. Prior to that, renewable and carbon neutral efficient biofuels is required to replace fossil-derived fuels in near future towards energy conservation and environmental sustainability.

Biodiesel is an alternative biofuel for diesel powered vehicles due to its renewability, biodegradability, and carbon neutrality. It is mainly derived from vegetable oil or animal fats through transesterification process in the presence of catalyst. Great thing about biodiesel is it can be produced from various sources such as edible oils [2,3], non-edible oils [4,5], animal fats [6], waste cooking oil (WC) [7], and algae oil [8]. The use of biodiesel in transportation is not a new concept, where it is widely used for more than 60 countries worldwide [9]. Many countries are promoting biodiesel fuel in order to reduce their reliance on non-renewable fossil fuels. Edible oil such as palm oil (PO) is the most promising candidates due to its high yield oil supply and best suited for usage in diesel engine. Nowadays, the utilization of biodiesel from non-edible crops gains more attention as the future raw material for biodiesel production. This will reduce the dependency on edible oils and to release stress on human food supply in long term. The non-edible crops such as *Jatropha curcas* (JC), *Moringa*, *Pongamia*, *Mahua* and *Calophyllum inophyllum* have drawn attention by many fellow researchers [10–13].

Although *Jatropha* is ranked behind palm according to the annual oil yield/hectare, it is favored as a non-edible feedstock. *Jatropha curcas* is a promising raw material to complement palm oil in near future. The plant indigenous to the genus *Euphorbiaceae*, which is native in tropical American and has been widely spread all over the tropics and subtropics regions of Africa, India and South-East Asia [14]. The crops are suitable for Malaysia given that the plants grow well within temperature of 20 to 30 °C and in a wide range of humidity conditions. Like palm oil, the crude *Jatropha curcas* oil can be extracted from the seeds and kernels which is around 40 and 60% respectively, through oil pressing. Besides that, the oil has the total unsaturated and saturated fatty acid content of 79 and 21 percent respectively [15]. High amount of unsat-

urated fatty acids, i.e., oleic and linoleic acid indicates that the oil has good cold flow properties as biodiesel.

Besides using non-edible oil as biofuel, converting waste cooking (WC) oil into biofuel is a three-win alternative, which can improve food security, increase energy security and reduce pollutions [16,17]. WC oil is commonly found in food processing industry, restaurants, fast foods or at consumer level, within households. In most developing countries, WC oil is disposed simply everywhere either on the earth itself or into the water ecosystem, which will then cause serious environmental pollutions, social and health problems to the society. However, WC oil contains high impurities, such as free fatty acid (FFA) and water which must be removed before the transesterification process take place.

Meanwhile, the catalyst holds an important part to enhance and facilitate the chemical catalysed reaction [18]. The catalyst used in biodiesel production can be categorized into three main groups: (i) homogeneous, (ii) heterogeneous and (iii) enzyme-based catalyst. The most common catalyst used in the biodiesel production industry is homogenous base catalyst such as sodium hydroxide (NaOH) and potassium hydroxide (KOH). They are low in cost and easily available in the market. These catalysts are able to produce high biodiesel yield under a short time duration, almost 400 times faster compared to those of acid catalyst [19]. In comparison, KOH is much stronger base than the NaOH where KOH losing OH<sup>-</sup> ion more easily than Na. In fact, KOH can be dissolved extremely fast in methanol and KOH based glycerine is very easy to handle by comparison. Therefore, these are the reasons why most researchers tend to use KOH in the biodiesel production [20].

Response Surface Methodology (RSM) was effectively used for design, optimization and analysis of experiments that is often been used in biodiesel production process. RSM can provide the details of interaction and quadratic effects between the process variables involved in the biodiesel production process and to predict the optimal parameters by reducing the large number of experiments that need to perform via conventional experiment methods. Thus, RSM helps to boost productivity as well as to minimize the time and cost consumption required for optimization. The two most common design used in response surface modelling are Box-Behnken Design (BBD) and Central Composite Design (CCD) which can study the process variables at three and five levels, respectively. The use of BBD is very popular in industrial research due to economic benefits as BBD has lesser number of experimental runs than the CCD. However, CCD is more effective response surface design that can fit a full quadratic model. CCD contains an embedded factorial or fractional factor design with a centre point that is augmented with a group of axial points which can

provide excellent prediction for the second-order model or quadratic model with optimum experimental runs [21]. Ngan et al. [22] found that CCD predicts better responses closer to the actual values as compared to the BBD to optimize fullerene loaded palm-based nano-emulsions for cosmeceutical application. Several other researchers also used CCD experimental design to optimize process variables involves in biodiesel production [23–25].

To ensure the competitiveness of biodiesel, the technology used for its production is equally important. With the current technology nowadays, biodiesel can be produced through advance methods which is more energy efficient such as microwave (MW) irradiation technology replacing the conventional heating technique which consumes much more energy and longer reaction time. The use of the MW heating system in a transesterification process can increase the biodiesel production rate by reducing the processing time, which is obtained from improved heat transfer efficiency and chemical reactions and thus results in energy saving (energy saving up to 48%) [26].

Milano et al. [9] adopted microwave irradiation-assisted transesterification to produce biodiesel from mixture of WCO and *Calophyllum inophyllum* oil (W70CI30) with optimized RSM based on BBD in 9.15 min of reaction time with 97.65% of biodiesel yield. The results indicate that the microwave radiation provides unique thermal effects for chemical synthesis by improving biodiesel yields, increasing the rate of chemical reaction, and reducing the net energy to produce biodiesel. Microwave has shorter reaction time with an effective temperature effect, thus leading to higher fatty acid methyl ester (FAME) yields. Besides that, Dharma et al. [27] also studied the optimization of biodiesel production for mixture *Jatropha curcas* - *Ceiba Petandra* biodiesel using RSM based on BBD. The maximum yield of the biodiesel was 93.33% at optimum operating parameters of 60 °C over a period of 2 h with 30% methanol/oil ratio, 1300 rpm agitation speed and 0.5 wt% KOH.

Silitonga et al. [28] also examined microwave irradiation-assisted transesterification to produce *Ceiba pentandra* biodiesel with optimized Extreme Learning Machine (ELM) from Cuckoo Search (CS) model. The results show that the microwave radiation can accelerate the rate of reaction and temperature within a shorter period. The maximum yield of *Ceiba pentandra* biodiesel was 96.19% at an optimum reaction time of 388 s, 800 rpm stirring speed, 60% methanol/oil ratio and 0.84 wt% KOH. In addition, Jaliliannosrati et al. [29] also performed a synthesis of fatty acid ethyl esters (FAEEs) from *Jatropha curcas* seeds by in situ two-step process, i.e. esterification and transesterification using microwave system. The highest conversion of biodiesel was 97.29% at the optimum conditions; <0.5 mm seed size, 12.21 min reaction time, 8.15 mL KOH catalyst loading and 331.52 rpm agitation speed at a constant 110 W microwave power.

Although MW heating has been used for transesterification, the optimum reaction condition to convert the mixture of WC, JC and PO into biodiesel has not been extensively studied. The current commercial biodiesel in Malaysia only uses PO as the feedstock in which the feedstock is also mainly used in food industry, so there is a stiff competition for availability. Since JC oil is a promising aspect as a non-edible oil in Malaysia, thus there will be no competition from food industry. Moreover, to fully utilize the WC oil from being disposed as

a waste, these three feedstocks are being mixed to be developed into biodiesel. In the current study, a household microwave was modified to be a biodiesel reactor and the microwave irradiation-assisted transesterification method was used in this study. The modified microwave biodiesel reactor will be discussed in the next section. Response Surface Methodology (RSM) based on Central Composite Design (CCD) was used to evaluate and optimize the effect of catalyst loading, methanol/oil ratio, reaction time and stirring speed to maximize the biodiesel yield. The procedure, mathematical model and optimization analysis used for biodiesel production using MW heating technique are described in detail in the next section. The physicochemical properties of the resulting biodiesel were determined, and verification was also done based on the biodiesel standards ASTM D6751 and EN14214. The novelty of this study lies in the optimization of the operating parameters for MW irradiation-assisted transesterification using RSM based on CCD in order to maximize the yield of waste cooking oil – *Jatropha curcas* – palm oil biodiesel.

## 2. Experimental procedure

### 2.1. Materials

WC oil was collected from households at Kuala Lumpur, Malaysia. JC and PO oils were obtained from Bionas (Kuala Lumpur, Malaysia) and Sime Darby Oils – Jomalina Refinery (Selangor, Malaysia), respectively. The following chemicals were used in this study; methanol (99.9% purity, ACS reagent grade), sulphuric acid (98% purity, AR grade), potassium hydroxide pellets (99% purity), anhydrous sodium sulphate (99.84% purity, AR grade), FAME mix C<sub>8</sub>-C<sub>24</sub> (Sigma-Aldrich), methyl nonadecanoate, C19 (Sigma-Aldrich, >99.5% purity).

### 2.2. Selection and preparation of feedstocks

Using WC oil as a biodiesel feedstock is an alternative way to utilize WC oil efficiently and economically. However, converting untreated WC oil into high quality biodiesel poses a significant challenge due to the unfavourable properties of the oil, which subsequently lead to low biodiesel yield with poor quality. Therefore, combining the WC oil with other crude oils derived from our tropical biodiversity such as *Jatropha curcas* and palm oil will improve the feedstock properties, thus resulting in a high-performance biodiesel for transportation.

Therefore, in this current study, WC, JC and PO oils were mixed together in order to obtain the best combination of a crude oil mixture with superior physicochemical properties, which able to improve the biodiesel quality and yield. The crude oil mixtures contained fixed amount of 50 vol% of WC oil with a varied amount of JC and PO, i.e., WC50-JC35-PO15, WC50-JC30-PO20, WC50-JC25-PO25, WC50-JC20-PO30 and WC50-JC15-PO35. The physicochemical properties of the crude oils and their mixtures are tabulated in Table 1.

Based on Table 1, the density, acid value, FFA content decreases as the PO content in crude oil mixture increases. Meanwhile, the kinematic viscosity, higher heating value and oxidation stability increases as the PO content in the crude oil mixture increases. From the observation, WC50-JC15-

**Table 1** Physicochemical properties WC, JC, PO and their crude oil mixtures.

Properties	Unit	Crude Oil			Crude Oil Mixture				
		WC	JC	PO	WC50-JC35-PO15	WC50-JC30-PO20	WC50-JC25-PO25	WC50-JC20-PO30	WC50-JC15-PO35
Kinematic viscosity, at 40 °C	mm <sup>2</sup> /s	41.193	34.933	40.682	39.036	39.382	39.547	39.901	40.200
Density, at 15 °C	kg/m <sup>3</sup>	916.2	917.7	915.1	916.9	916.6	916.5	916.4	916.2
Acid value	mg KOH/g	0.43	47.69	0.39	15.48	12.53	10.56	8.27	6.20
FFA content	wt%	0.22	23.84	0.19	7.74	6.26	5.28	4.14	3.10
Higher heating value	MJ/kg	39.6	39.2	39.7	39.4	39.4	39.4	39.5	39.5
Oxidation stability, 110 °C	hr	4.86	2.73	24.45	2.79	3.97	5.38	7.25	11.86

PO35 oil mixture gives favourable oxidation stability values with 11.86 hr, and it has the lowest acid number and FFA content as compared to other oil mixtures, thus requires minimum conditions in acid catalysis esterification as a pre-treatment step to reduce the FFA content. Therefore, WC50-JC15-PO35 oil mixture (hereinafter referred to as “WJP”) is selected for this study.

### 2.3. Production of WJP biodiesel

#### 2.3.1. Acid-catalyzed esterification of crude oil mixture

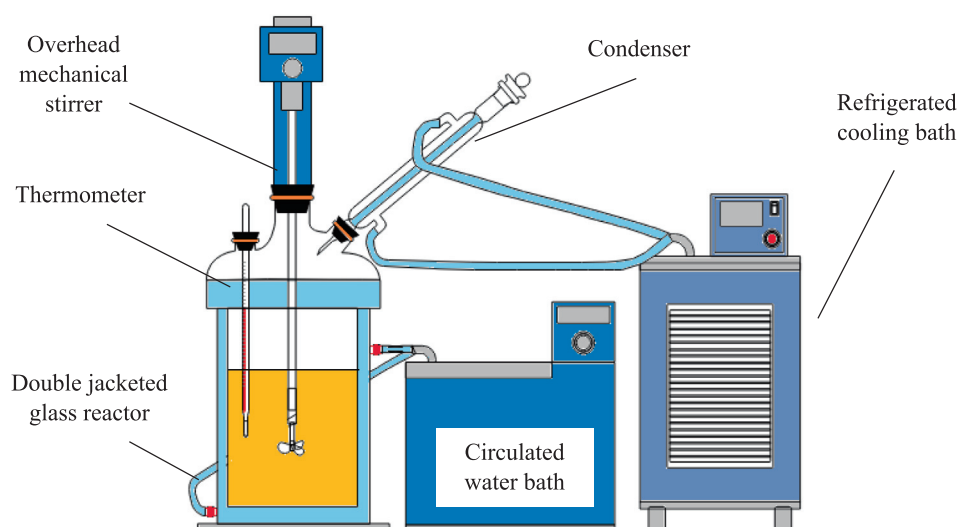
In the first step of biodiesel production, the oil reacted with alcohol and acid as the catalyst, to convert fatty acid into esters. The esterification process was carried out in a double jacketed glass reactor equipped with condenser and mechanical stirrer as shown in Fig. 1. In this study, WJP oil mixture was esterified using concentrated sulphuric acid of 1 wt% and methanol to oil molar ratio of (12:1) for 2 hr at stirring speed of 1000 rpm and reaction temperature of 65 °C. After the reaction completed, the oil was then settled in a separating funnel for at least 6 hr. Two separation layers were formed, whereas the top layer contains excess methanol and esterification by-products, while the bottom layer is the WJP esterified oil. The esterified oil was then collected and reheated in a rotary vacuum evaporator at 50 °C for 30 min. Upon comple-

tion, FFA level of the esterified oil was measured and the value of the FFA must be at the acceptance level (FFA < 1%).

#### 2.3.2. Microwave irradiation-assisted transesterification of WJP biodiesel

The transesterification step was conducted by employing a base catalyst and alcohol to convert the triglycerides into biodiesel. Fig. 2 shows the microwave reactor used in this experiment. The reaction was carried out in a 500 mL flat-bottom flask equipped with a reflux condenser and glass mechanical stirrer (Duran, Germany) and the reactor was placed inside a household microwave oven (Sharp, Japan). The fixed 50 g of the esterified oil and desired amount of the potassium hydroxide (KOH) catalyst and methanol were added into the reactor at respective methanol/oil molar ratios as described in the next section. The lowest MW power was employed at 10% of total output power due to the high volatility of methanol and to consume as little energy as possible. Thus, the reaction mixture was irradiated at 80 W for all the experiments at different reaction times and stirring speeds, and it was instantly stopped by rapid cooling in an ice bath.

The reaction mixture was then settled in a separating funnel for at least 6 hr upon completion. There were two separating layers observed, where the top layer is the WJP biodiesel, while the bottom layer is a mixture of glycerol, methanol and other

**Fig. 1** Esterification of crude oil mixture.



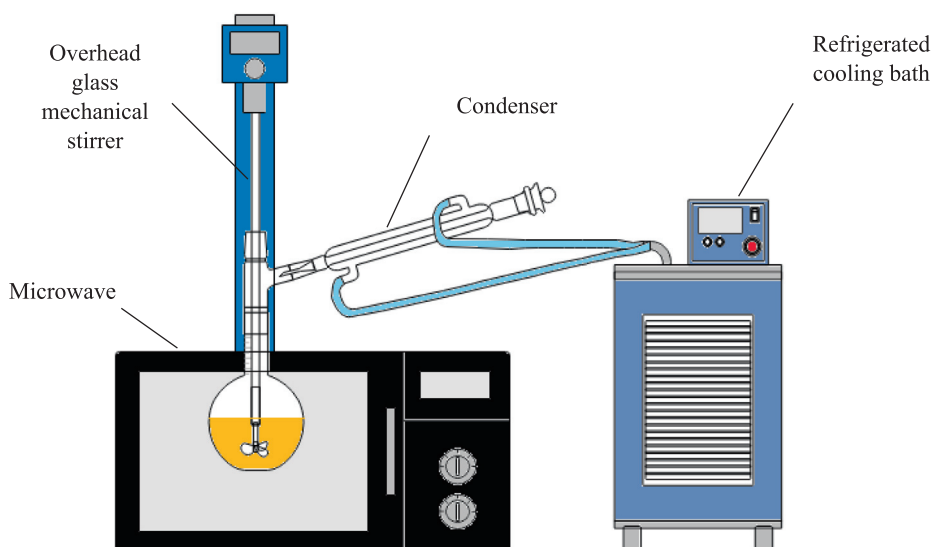


Fig. 2 Microwave irradiation-assisted transesterification of WJP oil.

impurities. The bottom layer was removed, while the top layer containing WJP biodiesel was being washed for several times using warm distilled water (40 °C) until crystal clear. Then, the WJP biodiesel was reheated in a rotary vacuum evaporator at 50 °C for 30 min to remove any moisture remaining in the biodiesel. Anhydrous sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) was added into the biodiesel to further absorb excess moisture. Finally, the WJP biodiesel was filtered via vacuum filtration to remove insoluble foreign impurities. The yield of WJP biodiesel was calculated by using the following equation (1):

$$WJP_{\text{yield}} = \frac{\text{Weight of WJP (g)}}{\text{Weight of oil used (g)}} \times 100\% \quad (1)$$

#### 2.4. Modelling using Central Composite design (CCD)

In this study, Design-Expert® software version 10 (Stat-Ease, Inc., USA) was used for the statistical DOE and analysis of data. A central composite design (CCD) tool was used to construct batch trials to optimize selected process variables involved in this biodiesel production. Response Surface Methodology (RSM) was subsequently employed to achieve the optimum conditions and to evaluate the response patterns. Four input factors including catalyst concentration (A), methanol/oil molar ratio (B), reaction time (C), and stirring speed (D) were identified for transesterification reaction with five levels as summarized in Table 2. These levels include plus and minus 1 (factorial points), a centre point, as well as plus and minus alpha (axial points). The total number of experimental runs is 30, since there are four process parameters with five levels for each. The response variable for the design experiment is the yield of WJP biodiesel which was measured at the end of every run as reported in Table 3.

#### 2.5. Statistical analysis

The optimum condition that correlates the yield of WJP biodiesel as a function of input factors is predicted using quadratic model as expressed in the following equation (2):

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i,j>j}^k \sum_{i=1}^k \beta_{ij} x_i x_j + e \quad (2)$$

where,  $Y$  is the response factor (yield of biodiesel),  $x_i$  and  $x_j$  are the process variables,  $\beta_0$  is constant coefficient,  $\beta_i$ ,  $\beta_{ii}$  and  $\beta_{ij}$  are the interaction coefficients of linear, quadratic and second order terms, respectively,  $k$  is the number of process variables, and  $e$  is the value that attribute to the uncertainty of  $Y$ .

For graphical data analysis, analysis of variance (ANOVA) was used to study interaction between the process factors and the response. The coefficient of determination ( $R^2$ ) was used to indicate the quality of the fitted quadratic model, while the F-value and Adequate Precision were used to validate its statistical significance.

#### 2.6. Measurement of physicochemical properties of WJP biodiesel

The physicochemical properties of the optimized WJP biodiesel produced via MW irradiation-assisted transesterification were measured according to the ASTM D6751 and EN 14,214 standards and the results were compared with those for waste cooking oil (WC) biodiesel, *Jatropha curcas* (JC) biodiesel and palm oil (PO) biodiesel separately. Table 4 lists all the instruments and recommended test methods used to measure the physicochemical properties of the biodiesels. Table 5 shows the operating conditions used in gas chromatography (GC) analysis to measure the composition of FAME and linolenic acid methyl ester content according to the EN 14103:2011 standard test method. Cetane number of the biodiesel was computed based on fatty acid weight composition proposed by E. G. Giakoumis and C. K. Sarakatsanis [30].

### 3. Results and discussions

#### 3.1. Prediction model for WJP biodiesel yield

Four parameters, i.e., catalyst concentration, methanol to oil molar ratio, reaction time and stirring speed were optimized

**Table 2** Ranges and levels of four input factors used in microwave assisted transesterification reaction.

Input Factor	Symbol	Units	Levels				
			+ 1 level	−1 level	Center	− $\alpha$	+ $\alpha$
Catalyst concentration	A	wt. %	0.75	1.25	1.00	0.50	1.50
Methanol/oil molar ratio	B	mol/mol	9:1	15:1	12:1	6:1	18:1
Reaction time	C	min	7.5	12.5	10.0	5.0	15.0
Stirring speed	D	rpm	400	600	500	300	700

**Table 3** Experimental design matrix with four input factors and one response variable.

Run	Space type	Factor 1 A: Catalyst concentration (wt. %)	Factor 2 B: Molar Ratio (mol/mol)	Factor 3 C: Reaction time(min)	Factor 4 D: Stirring speed(rpm)	Response 1 Biodiesel yield(%)
1	Factorial	1.25	15:1	7.5	600	93.12
2	Center	1.00	12:1	10.0	500	96.40
3	Factorial	0.75	15:1	12.5	600	93.21
4	Factorial	0.75	9:1	12.5	600	96.45
5	Center	1.00	12:1	10.0	500	96.25
6	Axial	1.00	12:1	10.0	700	94.86
7	Factorial	1.25	15:1	12.5	400	93.87
8	Factorial	1.25	9:1	12.5	400	90.64
9	Factorial	0.75	9:1	7.5	400	94.28
10	Factorial	0.75	15:1	12.5	400	93.69
11	Factorial	1.25	9:1	12.5	600	90.75
12	Axial	1.00	18:1	10.0	500	92.49
13	Center	1.00	12:1	10.0	500	96.52
14	Factorial	0.75	15:1	7.5	600	91.65
15	Axial	1.00	12:1	10.0	300	94.59
16	Factorial	1.25	15:1	7.5	400	92.78
17	Factorial	0.75	9:1	7.5	600	95.05
18	Factorial	1.25	9:1	7.5	400	89.16
19	Center	1.00	12:1	10.0	500	96.62
20	Factorial	1.25	9:1	7.5	600	89.53
21	Axial	1.00	12:1	5.0	500	90.69
22	Center	1.00	12:1	10.0	500	96.39
23	Factorial	0.75	9:1	12.5	400	96.75
24	Axial	1.00	12:1	15.0	500	94.49
25	Axial	1.50	12:1	10.0	500	90.16
26	Factorial	0.75	15:1	7.5	400	91.55
27	Axial	1.00	6:1	10.0	500	91.46
28	Factorial	1.25	15:1	12.5	600	94.09
29	Axial	0.50	12:1	10.0	500	94.07
30	Center	1.00	12:1	10.0	500	96.21

in this study to maximize the yield of WJP biodiesel via MW irradiation-assisted transesterification. The yield of the WJP biodiesel obtained from each run is provided in Table 3. Multiple regression analysis was conducted on the findings obtained from the CCD experimental design, and it was found that the quadratic response surface regression model is the best predictor for this experiment as given in Equation (3).

$$\begin{aligned}
 Y = & 96.40 - 1.10A + 0.14B + 0.83C + 0.07D - 1.06A^2 \\
 & - 1.10B^2 - 0.954 - 0.41D^2 + 1.64AB - 0.18AC \\
 & + 0.059AD - 0.051BC - 0.048BD - 0.13CD
 \end{aligned} \quad (3)$$

where Y represents the yield of WJP biodiesel and A, B, C, and D represent catalyst concentration, methanol to oil molar ratio, reaction time and stirring speed, respectively.

### 3.2. Optimization of the MW irradiation-assisted transesterification process parameters using RSM

Table 6 shows the statistical ANOVA results, of the quadratic response surface regression model as well as the significance of the regression coefficients in order to maximize the yield of WJP biodiesel. The F-value and p-value of the model are 196.66 and less than 0.0001 respectively. Therefore, the quadratic regression model is significant to predict the yield of WJP biodiesel. In addition, the lack of fit F-value is 3.07, which is not significant when compared to pure error. This means that the lack of fit F-value is attributed to noise by 11.39% probability, and its associated p-value is 0.1139, showing that the model fits the experimental data well. Fig. 3 shows a comparison of WJP biodiesel yield predictions with experimental

**Table 4** List of instruments and recommended test method to measure physicochemical properties and FAME composition of biodiesel.

Property	Equipment	Test Method
Kinematic viscosity, 40 °C	Stabinger viscometer SVM 3000(Anton Paar, Austria)	ASTM D 445
Density, 15 °C	Stabinger viscometer SVM 3000(Anton Paar, Austria)	ASTM D 4052
Flash point	PMA 5 Pensky-Martens flash point tester (Anton Paar, Austria)	ASTM D 93
Cloud point	NTE 450 CP tester (Norma Lab, France)	ASTM D 2500
Pour point	NTE 450 PP tester (Norma Lab, France)	ASTM D 97
Cold filter plugging point (CFPP)	Calisto 100 CFPP tester (Anton Paar, Austria)	ASTM D 6371
Copper strip corrosion, 3 h at 50 °C	Seta copper corrosion bath (Stanhope-Seta, UK)	ASTM D 130
Oxidation stability, 110 °C	873 Biodiesel Rancimat (115 V)(Metrohm AG, Switzerland)	ASTM D 7462
Higher heating value	C2000 basic Calorimeter-automatic(IKA, UK)	ASTM D 240
FAME and linolenic acid methyl ester content	GC-FID analyzer (Agilent Technologies 7890) (Agilent Technologies, USA)	EN 14103:2011

**Table 5** Operating conditions used for FAME composition analysis.

Parameters	Specifications
Capillary Column	HP Innowax column, 30 mm × 0.25 mm × 0.25 µm
Oven temperature	60 °C, hold 2 min10 °C/min up to 200 °C, 0 min5 °C/min up to 240 °C, 0 min240 °C hold for 7 minPost run, 255 °C, 0.5 min
Carrier gas	Helium
Flow rate	1.5 mL/min
Injection temperature	250 °C
Detector temperature	250 °C
Type of Injector	Split/splitless
Type of detector	Flame ionization detector
Injection volume	1 µL
FAME standard	FAME mix C8–C24, Sigma-Aldrich, 100 mg
Internal standard (IS)	Methyl nonadecanoate, C19, Sigma-Aldrich, >99.5 %

results. The actual values of data are specified in Table 3, whereas the predicted values are obtained by the model from Equation (3). The standard deviation of the model was found to be small i.e., 0.24 indicates better predicting response of the

model developed. The coefficient of determination ( $R^2$ ) is 0.9946 close to unity, indicating that this model gives 99.46% of the variability in WJP biodiesel yields. This shows that the predicted WJP biodiesel yield values are close to those obtained by CCD experimental design, indicating that the model is reliable. It is also found that the mean square error (MSE) and root mean square error (RMSE) value is 0.028891 and 0.158242, respectively. Therefore, the smaller values of MSE and RMSE implies higher accuracy of the regression model, however greater value of  $R^2$  is desirable. Moreover, the adequate precision is a measure of the signal to noise ratio and it is determined to be at 44.831, which is much greater than 4, indicating that the model is acceptable to navigate through the design space and to predict WJP biodiesel yield. The difference between the predicted  $R^2$  (0.9721) and adjusted  $R^2$  (0.9895) is less than 0.2, indicating that the regression polynomial is in reasonable agreement.

Besides that, the term that has a probability value less than 0.05 ( $p < .05$ ) would be considered as a significant effect to the model. Based on this criterion, A, B, C, AB, AC,  $A^2$ ,  $B^2$ ,  $C^2$  and  $D^2$  are the significant model terms, indicating that these model terms have a significant effect on the yield of WJP biodiesel. However, the p-values for D, AD, BC, BD and CD are greater than 0.05, indicating that these model terms have no significant effect to the yield of WJP biodiesel.

### 3.3. Effect of process parameters on WJP biodiesel yield

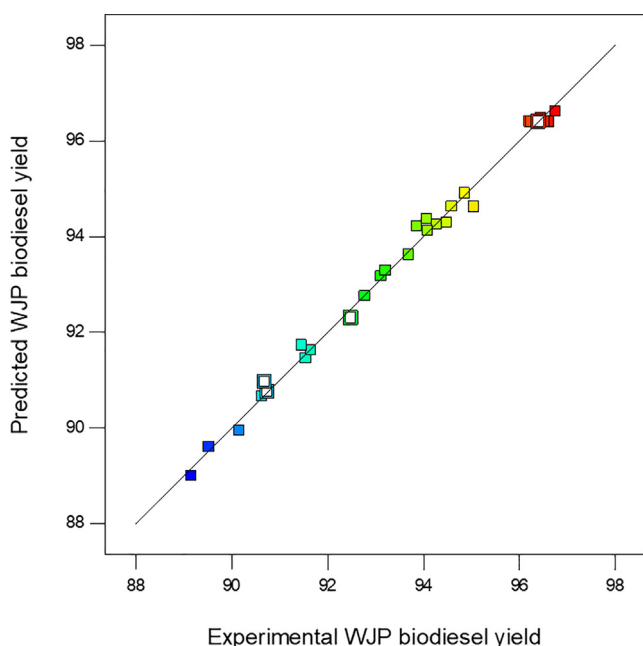
The effect of each process parameter on the yield of WJP biodiesel is discussed in detail in this section to understand how these process parameters affect the MW irradiation-assisted transesterification process. Fig. 4 shows the effect of process parameters (a) catalyst concentration, (b) methanol/oil molar ratio (c) reaction time, and (d) stirring speed, on the WJP biodiesel yield.

#### 3.3.1. Effect of catalyst concentration

Potassium hydroxide (KOH) is a superb homogeneous catalyst that promotes high biodiesel yield due to greater catalytic activity in methanol solution. Additionally, it is critical to determine the accurate catalyst concentration to maximize the biodiesel yield. Fig. 4 (a) shows the effect of varying the KOH catalyst concentration from 0.75 to 1.25 wt% on WJP biodiesel yield. The catalyst concentration, reaction time and stirring speed are kept constant at 12:1, 10.0 min and 500 rpm, respectively. The WJP biodiesel yield increases significantly with the increase of KOH catalyst concentration from 0.75 to 0.85 wt%, and it decreases with further increase of catalyst concentration up to 1.25 wt%. The excessive amount of KOH catalyst used in transesterification reaction will cause a massive collision of ions due to overwhelming free ions in the mixture, thus reduces the activation energy needed by the oil to increase the rate of reaction, and eventually reduces biodiesel yield [31]. In addition, excessive amount of KOH catalyst also resulting in the formation of soap (saponification reaction), thus it complicates the process to separate the biodiesel from soap and excess glycerol during the purification process, and eventually reduces the biodiesel yield. Similarly, Hasni et al. [32] also found that the increase of sodium hydroxide (NaOH) catalyst concentration up to 1.25 wt% would result in higher *Brucea javanica* biodiesel yield. However, a

**Table 6** ANOVA for response surface quadratic model.

Source	Sum of Squares	df	Mean Square	F Value	p-value
Model	159.08	14	11.36	196.66	< 0.0001
A-Catalyst concentration	29.28	1	29.28	506.78	< 0.0001
B-Molar Ratio	0.48	1	0.48	8.39	0.0111
C-Reaction time	16.55	1	16.55	286.43	< 0.0001
D-Stirring speed	0.12	1	0.12	2.01	0.1766
AB	42.94	1	42.94	743.06	< 0.0001
AC	0.49	1	0.49	8.54	0.0105
AD	0.056	1	0.056	0.98	0.3388
BC	0.041	1	0.041	0.71	0.4128
BD	0.037	1	0.037	0.64	0.4357
CD	0.26	1	0.26	4.46	0.0519
A <sup>2</sup>	30.87	1	30.87	534.31	< 0.0001
B <sup>2</sup>	32.94	1	32.94	570.14	< 0.0001
C <sup>2</sup>	24.35	1	24.35	421.39	< 0.0001
D <sup>2</sup>	4.58	1	4.58	79.19	< 0.0001
Residual	0.87	15	0.058		
Lack of Fit	0.75	10	0.075	3.07	0.1139
Pure Error	0.12	5	0.024		
Correlated totalsum of squares	159.95	29			
Standard deviation	0.24			Adjusted R <sup>2</sup>	0.9895
Mean	93.59			Predicted R <sup>2</sup>	0.9721
Coefficient of variation	0.26			Adequate precision	44.831
R <sup>2</sup>	0.9946				

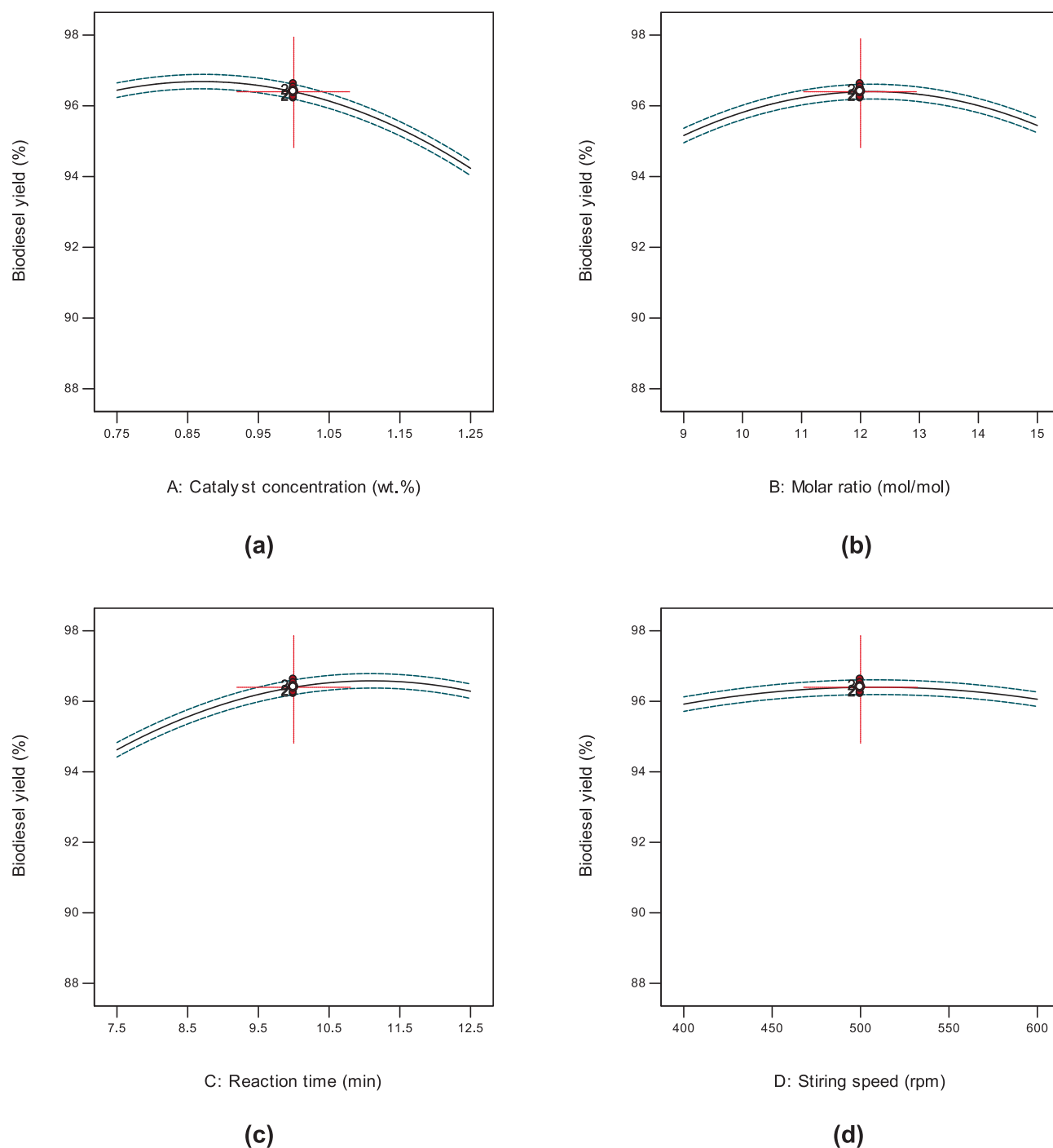
**Fig. 3** Comparison between the predicted and experimental of WJP biodiesel yield.

reversed trend was observed when the concentration of catalyst is too high. The results indicate that the excess amount of catalyst used in the reaction will produce high amount of glycerine due to saponification, thus reduce biodiesel yield. Therefore, further increase in catalyst concentration will not increase the biodiesel conversion yield. In fact, extra costs were required to remove the catalysts from the reaction medium during washing process.

### 3.3.2. Effect of methanol to oil molar ratio

Molar ratio of methanol to oil is indeed important to optimize the biodiesel yield produced as well as to reduce biodiesel production cost. The transesterification process is reversible reactions where the stoichiometric molar ratio of alcohol to triglyceride is 3:1. However, higher molar ratio is required to increase the yield of alkyl esters and to allow phase separation from the glycerol. In this study, the transesterification reaction was conducted under MW heating in a closed reactor which prevents evaporation of methanol. For this reason, a significant quantity of methanol is required to optimize the quantity of biodiesel produced. Fig. 4 (b) shows the effect of methanol/oil molar ratio on WJP biodiesel yield. The methanol/oil molar ratio was varied from 9:1 to 15:1, whereas catalyst concentration, reaction time and stirring speed were kept constant at 1.0 wt%, 10.0 min and 500 rpm respectively. It was observed that the yield of WJP biodiesel increases with increase of methanol/oil molar ratio from 9:1 to 12:1. Nonetheless, a reduction in the conversion yield was observed when the methanol to oil molar ratio was increased to a level above 12:1. This could be due to an excess of methanol in the reaction, which interferes the separation of the alkyl ester and glycerol by increasing the glycerol solubility. As a result, some diluted glycerol remains in the alkyl ester phase, resulting in an apparent loss of alkyl ester final product due to soap formation. This is in line with the result reported by Ong et al. [33] whereby *Calophyllum inophyllum* - *Ceiba pentandra* biodiesel yield increases as the methanol to oil ratio increases from 30 to 37 vol%, but it decreases when the methanol to oil ratio is furtherly increased. The biodiesel yield decreases significantly most likely due to emulsification of the alkyl ester and glycerol, which in turn complicate the separation of biodiesel from glycerol thus results in lower yield of biodiesel.





**Fig. 4** Effect of process parameters (a) catalyst concentration, (b) methanol to oil molar ratio, (c) reaction time, and (d) stirring speed on the WJP biodiesel yield.

### 3.3.3. Effect of reaction time

Biodiesel produced via MW irradiation-assisted transesterification requires shorter reaction time with respect to conventional heating due to improve energy transfer (via penetrative radiation) and the reverse thermal effect, i.e. heat starts from the interior of material body [34]. In this study, the transesterification reaction was conducted at the lowest MW power (80 W) to hinder evaporation of methanol to occur and to consume as low energy as possible. Reaction time is

also a crucial operating parameter to ensure complete diffusion of the oil, methanol, and catalyst. Therefore, a suitable reaction time is required to optimize the quantity of WJP biodiesel produced. Fig. 4 (c) represents the WJP biodiesel yield with variation of reaction time from 7.5 to 12.5 min. The catalyst concentration, molar ratio and stirring speed were kept constant at 1.0 wt%, 12:1 and 500 rpm respectively. In Fig. 4 (c), the WJP biodiesel yield increases with the increase of reaction time from 7.5 to 11.0 min. The transesterification reaction

between methanol and oil is completed when it reaches an equilibrium state after 11.0 min. However, the WJP biodiesel yield decreases with further increase in reaction time. This indicates that the excessive reaction time may increase the reaction temperature, thus resulting in greater solubility of glycerol. The glycerol apparently dissolved with methyl ester thus complicates the removal of glycerol during water washing process. Similar results was found by Chen et al. [35], whereby the increase in reaction time from 1 to 3 min at very high microwave power of 750 W caused a significant increase in biodiesel yield, and then the yield decreased with further increase of reaction time.

### 3.3.4. Effect of stirring speed

Although stirring speed is not statistically significant as shown in Table 3, this parameter is very essential in transesterification reaction. Stirring keeps the reactants particles in motion, thus increase the chances (frequency) of collision of the particles and eventually increase the rate of reaction. Fig. 4 (d) shows the effect of stirring speed on the WJP biodiesel yield. The reaction speed was varied from 400 to 600 rpm, whereas catalyst concentration, molar ratio and reaction time were kept constant at 1 wt%, 12:1 and 10.0 min respectively. It was observed that the WJP biodiesel yield is slightly increased from 400 to 500 rpm over a period of 10.0 min of MW heating. This result indicates that this exothermic reaction reaches an equilibrium state with an optimum operating stirring speed of 500 rpm right after 10.0 min. However, the biodiesel yield gradually decreases with further increase of stirring speed from 500 to 600 rpm. Excessive stirring speed may cause the mixture to rapidly revolve like a cyclone which can minimize the chance of the reaction to occur. Besides that, the reaction temperature is also gradually increase at a very high stirring speed, which then leads to an unnecessary reaction, eventually resulting in lower biodiesel yield.

### 3.4. Validation of the quadratic response surface regression model

The numerical optimization was used to determine the optimum parameters which can maximize the yield of WJP biodiesel. The process parameters which comprised of catalyst concentration, methanol to oil molar ratio, reaction time and stirring speed were set between the lower and upper limits at a setting of 3 pluses (+++), indicating equally important goals, whereas the WJP biodiesel yield was set at maximum value of 5 pluses (+++++) , indicating the most important goal. The optimization constraints for the WJP biodiesel production parameters are shown in Table 7.

Three experiments were repeated based on the optimized process parameters acquired from the optimization tool to verify the quadratic response surface regression model. The predicted and the experimental yield of WJP biodiesel are listed in Table 8.

It was found that the average WJP biodiesel yield obtained from the experiment is 96.94%, which is slightly higher than predicted WJP biodiesel (96.81%). The difference between the predicted and experimental yield is 0.1% with a standard error of 0.0808. This suggests that the RSM based on the central composite design is a useful tool for predicting the ideal

process parameters of the MW irradiation-assisted transesterification to optimize the biodiesel yield.

### 3.5. Physicochemical properties and FAME composition of WJP biodiesel

The physicochemical properties of WJP biodiesel produced via MW irradiation-assisted transesterification are presented in Table 9. The physicochemical properties results were compared with respect to WC, JC and PO biodiesel. Table 10 shows the FAME and linolenic acid methyl ester content in WJP, WC, JC and PO biodiesel.

As mentioned previously, the optimized WJP biodiesel comprises 50 vol% of WC oil, 15 vol% of JC oil and 35 vol % of PO oil which was produced via esterification and MW irradiation-assisted transesterification. The physicochemical properties in Table 9 shows that WJP biodiesel has fulfilled the fuel specifications stated in the ASTM D6751 and EN 14,214 standards. Viscosity and density are crucial properties of a fuel, as it will directly affect the engine performances. Very high as well as very low viscosity of the fuel will impact the engine performances [36]. As such, too high viscosity of fuel will form larger droplets during injection which will resort in poor combustion quality and eventually produce higher exhaust emissions. Meanwhile, low fuel viscosity will not provide enough lubrication which then introduce wears and leakages. It was observed that the kinematic viscosity of the optimized WJP biodiesel ( $4.586 \text{ mm}^2/\text{s}$ ) is in the middle range between these single biodiesels, i.e. WC ( $4.793 \text{ mm}^2/\text{s}$ ), JC ( $4.394 \text{ mm}^2/\text{s}$ ) and PO ( $4.642 \text{ mm}^2/\text{s}$ ). However, all measured values are still within the range of permissible limits specified in ASTM D6751 ( $1.0\text{--}6.0 \text{ mm}^2/\text{s}$ ) and EN 14,214 ( $3.5\text{--}5.0 \text{ mm}^2/\text{s}$ ). Similarly, the density of the WJP, WC, JC and PO biodiesel were observed to be at 877.9, 877.6, 879.1 and  $876.4 \text{ kg/m}^3$  respectively, which means that they are also within the range specified in EN 14,214 ( $860\text{--}900 \text{ kg/m}^3$ ).

Besides that, flash point of the biodiesel was measured to determine the safety measure of fuel for storage purpose. It was observed that the optimized WJP biodiesel has the highest flash point ( $185.6^\circ\text{C}$ ) with respect to that of WC, JC and PO biodiesel. This indicates that the WJP biodiesel is safer for handling and storage. In addition, biodiesel is easily oxidized at higher temperature and when in contact with air due to the different chemical compositions in FFA. It was observed that the oxidation stability of the optimized WJP biodiesel (11.55 hr) is higher compared to JC (7.93 hr) and WC (10.07 hr) and but slightly lower than that of PO (13.11 hr). Indeed, the 15 vol % composition of JC oil in the crude oil mixture slightly reduces the oxidation stability because it bears the double bond molecules in the FFA. However, the biodiesel still achieves the minimum permissible limits of oxidation stability which must be greater than 3 hr (US standard) and 6 hr (European standard).

Besides that, cold flow properties of biodiesel were also measured in terms of pour point (PP), cloud point (CP), and cold filter plugging point cloud (CFPP). These cold flow properties are very crucial to determine the quality of the biodiesel produced and decide whether the biodiesel is suitable under cold weather conditions [37]. It was observed that JC biodiesel has better cold flow properties, i.e., PP, CP, and CFPP with respect to PO and WC biodiesel. The presence of JC oil in

**Table 7** Optimization constraints for the WJP biodiesel production parameters.

Parameters	Units	Goal	Lower limit	Upper limit	Importance
Catalyst concentration	wt. %	In range	0.75	1.25	+++
Methanol/oil molar ratio	mol/mol	In range	9:1	15:1	+++
Reaction time	min	In range	7.5	12.5	+++
Stirring speed	rpm	In range	400	600	+++
Biodiesel yield	%	Maximize	89.16	96.75	++++

**Table 8** Predicted and experimental yield of the optimized WJP biodiesel.

Run	Catalyst concentration (wt. %)	Methanol/oil molar ratio (mol/mol)	Reaction time (min)	Stirring speed (rpm)	Predicted yield (%)	Experimental yield (%)
1	0.78	9.86	10.5	478	97.06	96.91
2	0.78	9.86	10.5	478	97.06	96.77
3	0.78	9.86	10.5	478	97.06	97.05
<b>Average</b>					<b>96.81</b>	<b>96.91</b>
<b>Standard error</b>						<b>0.0808</b>

**Table 9** Physicochemical properties of WJP biodiesel with respect to WC, JC, PO biodiesel and standard specification of biodiesel fuel (ASTM D6751 and EN14214).

Property	Unit	WJP biodiesel	WCbiodiesel	JCbiodiesel	PObiodiesel	ASTM D 6751	EN 14,214
Kinematic viscosity, 40 °C	mm <sup>2</sup> /s	4.586	4.793	4.394	4.642	1.9–6.0	3.5–5.0
Density, 15 °C	Kg/m <sup>3</sup>	877.9	877.6	879.1	876.4	n/a	860–900
Flash point	°C	185.6	174.0	182.5	180.2	93 min	120 min
Pour point	°C	5.4	8.3	−3.9	9.1	–	–
Cloud point	°C	11.2	13.9	2.7	14.6	Report	Location and season dependant
CFPP	°C	7.9	10.6	−0.4	11.2	–	–
Oxidation stability, 110 °C	hr	11.55	10.07	7.93	13.11	3.0 min	6.0 min
Higher heating value	MJ/kg	39.98	40.03	38.75	40.14	n/a	n/a
Cetane number*	–	59.3	60.9	55.0	61.3	47 min	51 min
Total acid number	mg KOH/g	0.05	0.05	0.10	0.05	0.50 max	0.50 max
Copper strip corrosion, 3 h at 50 °C	Rating	1a	1a	1a	1a	No. 3 max	Class 1
FAME content	wt. %	98.86	97.17	89.64	94.13	n/a	90 min.
Linolenic acid methyl ester content	wt. %	0.25	0.30	0.16	0.21	n/a	1–15

Note: \*Cetane number of the biodiesel were computed based on fatty acid weight composition proposed by E. G. Giakoumis and C. K. Sarakatsanis [30].

the crude oil mixture improves the cold flow properties of the WJP biodiesel where the PP, CP, and CFPP were observed to be at 5.4, 11.2, and 7.9 °C respectively, which can be considered that the fuel is significantly improved compared to PO and WC biodiesel.

Additionally, higher heating value is another crucial property to determine its competency as a diesel fuel substitute that produces the power output to the engine. Greater amount of higher heating value is clearly desirable. It was observed that higher heating value of the optimized WJP biodiesel (39.98 MJ/kg) is slightly lower than PO (40.14 MJ/kg) and WC biodiesel (40.03 MJ/kg). However, there were no significant differences of higher heating values between the biodiesels.

Besides that, the FAME and linolenic methyl ester content of the optimized WJP, WC, JC and PO biodiesel were determined in accordance with the EN 14103:2011 standard test method and the results are summarized in Table 10. The optimized WJP, WC and PO biodiesel satisfy the requirement mentioned in the EN 14,103 standard test method where the FAME content must be greater than 90 wt%. The total FAME of the optimized WJP, WC and PO biodiesel are 98.86, 97.17 and 94.13 wt% respectively. Besides that, the linolenic acid content for WJP, WC, JC and PO biodiesel are lower than the permissible range of 1–15 wt% mentioned in the EN 14103:2011 test method. It was observed that the linolenic acid content of the optimized WJP, WC and PO biodiesel are at 0.25, 0.30, 0.16, 0.21 wt% respectively.

**Table 10** FAME and linolenic acid content in WJP, WC, JC and PO biodiesel.

Name of FAME	Molecular formula	Carbon structure	FAME content (wt.%)			
			WJP	WC	JC	PO
Methyl Laurate	C <sub>11</sub> H <sub>23</sub> COOCH <sub>3</sub>	C12:0	0.17	0.20	0.00	0.22
Methyl Tetradecanoate	C <sub>13</sub> H <sub>27</sub> COOCH <sub>3</sub>	C14:0	0.78	0.94	0.00	0.90
Methyl Palmitate	C <sub>15</sub> H <sub>31</sub> COOCH <sub>3</sub>	C16:0	32.66	35.99	12.88	37.31
Methyl Palmitoleate	C <sub>15</sub> H <sub>29</sub> COOCH <sub>3</sub>	C16:1	0.39	0.47	0.68	0.18
Methyl Stearate	C <sub>17</sub> H <sub>35</sub> COOCH <sub>3</sub>	C18:0	4.26	3.95	6.33	3.86
Methyl Oleate	C <sub>17</sub> H <sub>33</sub> COOCH <sub>3</sub>	C18:1	40.75	43.32	40.00	40.78
Methyl Linoleate	C <sub>17</sub> H <sub>31</sub> COOCH <sub>3</sub>	C18:2	19.22	11.58	29.33	10.27
<b>Methyl Linolenate</b>	<b>C<sub>17</sub>H<sub>29</sub>COOCH<sub>3</sub></b>	<b>C18:3</b>	<b>0.25</b>	<b>0.30</b>	<b>0.16</b>	<b>0.21</b>
Methyl Arachidate	C <sub>19</sub> H <sub>39</sub> COOCH <sub>3</sub>	C20:0	0.38	0.41	0.26	0.40
Saturated FAME			38.25	41.49	19.46	42.69
Unsaturated FAME			60.61	55.68	70.18	51.44
<b>Total FAME</b>			<b>98.86</b>	<b>97.17</b>	<b>89.64</b>	<b>94.13</b>

Additionally, it can be found that all the biodiesels contain more unsaturated fatty acid methyl esters (FAME) with double or triple bonds compared to saturated FAME whereby the carbon atoms are linked by single bonds. As such, the JC biodiesel shows the highest polyunsaturated FAME content with 70.18 wt%, followed by WJP, WC and PO biodiesel with 60.61, 55.68 and 51.44 wt% respectively. Greater amount of polyunsaturated FAME content in the biodiesel will improve the cold flow properties of the biodiesel [38]. As evidence, JC biodiesel shows better cold flow properties in terms of cloud point, pour point and CFPP in comparison to PO and WC biodiesel due to high content of linoleic acid. Therefore, the presence of JC oil in WJP biodiesel which contains higher unsaturated FAME at least enhances the cold flow properties of the biodiesel. Nonetheless, the presence of higher polyunsaturated FAME will reduce the oxidation stability and cetane number of the resulting biodiesel [39]. The oxidation stability and cetane number of JC biodiesel are slightly lower than PO biodiesel due to presence of high content of polyunsaturated FAME. However, the oxidation stability and cetane number of the WJP biodiesel have surpassed the minimum limits stated in US and European biodiesel standards.

Meanwhile, the PO biodiesel shows the highest monosaturated FAME content with 42.69 wt%, followed by WC, WJP and JC biodiesel with 41.49, 38.25 and 19.46 wt% respectively. Greater amount of monounsaturated FAME will result into higher cetane number. Thus, higher cetane fuel clearly has shorter ignition delay which leads to more complete fuel combustion. It was revealed that the PO biodiesel has the highest cetane number with 61.3, followed by WC, WJP and JC biodiesel with 60.9, 59.3 and 55.0 respectively. Similar results were reported by Mofijur et al. [40] whereby the cetane number of PO and JC biodiesel were at 59 and 51 respectively. The cetane number of palm biodiesel is high due to the higher saturated FAME composition in the biodiesel, whereas the lower cetane number of JC biodiesel may be due to higher amount of linoleic acid. However, the cetane number for all biodiesels are within the specified standard limits where it must be greater than 47 in US standard and 51 in European standard.

In summary, JC biodiesel has a good cold flow properties but poor oxidation stability. Meanwhile, PO biodiesel has a good oxidation stability but poor cold flow properties. Therefore, synergistic combination of the WC oil with enhanced

**Table 11** Energy consumption for MW irradiation assisted transesterification of WJP biodiesel.

Equipment	Power (W)	Time, t (h)	Energy Consumption, Energy (kWh) = P*t
Microwave reactor	150	0.175	0.0263
Refrigerated cooling bath	500	0.175	0.0875
Overhead stirrer	35	0.175	0.0061
Total energy consumption for 50 mL of WJP biodiesel (kWh)			0.1199
Total energy consumption for 100 mL of WJP biodiesel (kWh)			0.2398

crude oil derived from tropical biodiversity, i.e., JC and PO will improve the properties of the feedstock to achieve a high-performance biodiesel for transportation.

### 3.6. Energy consumption for microwave irradiation assisted transesterification

Table 11 shows the energy consumption required in the MW irradiation-assisted transesterification process of WJP biodiesel. In this study, three electrical equipment were used in the transesterification process which includes microwave reactor, refrigerated cooling bath, and overhead stirrer. The total energy consumption is determined to be 0.1199 and 0.2398 kWh (equivalent to 431.64 and to 863.28 kJ) for reactions of 50 and 100 mL of WJP biodiesel, respectively. In comparison with the previous study, Milano et al. [31] found that the energy consumption required for microwave reactor and conventional reactor to produce 100 mL of waste cooking oil - *Calophyllum inophyllum* biodiesel was 74.41 and 1283.10 kJ. MW heating greatly accelerates the transesterification reaction, thus reducing the energy consumption for biodiesel production. The results indicate that MW biodiesel synthesis is beneficial for the industrial scale.

## 4. Conclusion

In this study, a household microwave was modified to be a biodiesel reactor, and MW irradiation-assisted transesterification

was adopted to convert the ternary oil mixture of waste cooking oil – *Jatropha curcas* – palm oil at a volume ratio of 50:15:35 developed into WJP biodiesel. Response Surface Methodology (RSM) based on Central Composite Design (CCD) was used to optimize production parameters, i.e. catalyst loading, methanol/oil ratio, reaction time and stirring speed in order to maximize the WJP biodiesel yield. The following conclusions of this study are drawn based on the results of the study:

1. The process parameters for the WJP biodiesel production from the ternary oil mixture of waste cooking oil – *Jatropha curcas* – palm oil by using microwave assisted transesterification were optimized. The optimized process parameters were: (1) KOH catalyst loading (0.78 wt%), (2) methanol/oil ratio (9.86:1), (3) reaction time (10.5 min), and (4) stirring speed (478 rpm) with the predicted and experimental yields are at 96.81 and 96.91 %, respectively.
2. The remarkable physicochemical properties of the optimized WJP biodiesel have fulfilled the fuel specifications stated in the ASTM D6751 and EN 14,214 standards. Overall, the results indicate that the synergistic combination of waste cooking oil, *Jatropha curcas* and palm oil as the feedstock for WJP biodiesel gives better cold flow properties (pour point, cloud point, and cold filter plugging point) as well as enhances oxidation stability and cetane number.

#### CRedit authorship contribution statement

**M.N.A.M. Yusoff:** Conceptualization, Investigation, Writing – original draft. **N.W.M. Zulkifli:** Writing – review & editing. **N.L. Sukiman:** Supervision. **M.A. Kalam:** Validation. **H.H. Masjuki:** Supervision. **A.Z. Syahir:** Data curation, Software. **M.S.N. Awang:** Investigation, Validation. **M.A. Mujtaba:** Software, Visualization. **J. Milano:** Formal analysis. **A.H. Shamsuddin:** Validation.

#### 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.

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