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Parameters Optimization of Palm Oil Biodiesel Production at Various CaO Concentrations Using Response Surface Methodology

Abdul Hadi[1,*], Nur Fadzila Mohamad[1], Mahidin[2]

[1] Center for Chemical Engineering Studies, College of Engineering, Universiti Teknologi MARA Cawangan Pulau Pinang, 13500 Permatang Pauh, Malaysia
[2] Department of Chemical Engineering, Faculty of Engineering, Universitas Syiah Kuala, Banda Aceh 23111, Indonesia

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ABSTRACT

The transesterification of biodiesel using heterogeneous catalysts has recently caught interest because of its potential to overcome the limitations of homogeneous catalysts. Amongst the heterogeneous catalysts, CaO is well known for its superiority in the transesterification process due to its effectiveness, low cost, and low solubility in methanol. The paper covers the analysis of the transesterification process variables to identify the optimum condition of the process. The effect of process variables, which are catalyst concentration, methanol to oil molar ratio, and reaction temperature, and the interaction between variables on the yield of biodiesel from the esterification of palm oil using CaO catalyst was studied. Response Surface Methodology based on a three-variable central composite design was executed to optimize the process variables. It was found that the concentration of the catalysts is the primary variable affecting the reaction, the molar ratio of methanol to oil is the secondary and the temperature is the ternary. While the interaction between the two variables is not significant in the production of the biodiesel yield. The optimum yield of biodiesel of 98.56% was obtained at a catalyst concentration of 9.63 wt%, methanol to oil ratio of 15.30:1, and reaction temperature of 64.40 °C.

1. Introduction

The exponential growth in the world population will lead to an increase in world energy demand. The supply of fossil fuel was restricted, and prolonged use will lead to resource depletion [1]. Biodiesel is a renewable fuel composed of fatty acid methyl esters (FAME). Compared with petroleum diesel, biodiesel has many advantages, such as safety, non-toxic and biodegradable [2]. The use of biodiesel can reduce particulate emissions by up to 75% [3] and also reduces the volume of unburned hydrocarbons, carbon monoxide, and particulate matter, including sulphur dioxide in exhaust emissions [4]. Biodiesel can be used for many applications, such as heating oil, clean oil spills, etc. Biofuels can also replace the energy needs from car fuel and central home heating.

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[*] *Corresponding author.*
E-mail address: hadi9598@uitm.edu.my

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The production of biodiesel in Malaysia in the year 2019 was about 1.56 million liters with a capacity utilization rate is 67%. The production reduced to be 1.05 million liters in 2020 due to the Covid-19 pandemic [5]. Malaysia still has 16 palm biodiesel refineries running today; Selangor (5), Johor (5), Sabah (2), Perak (2), Sarawak (1), and Pahang (1). However, Malaysia's biodiesel production is quite poor, because the total output of palm biodiesel in 2017 only accounted for 40% of its total annual production capacity (2.34 million liters) [6].

Transesterification is the most widely used process in biodiesel production. The molar ratio of alcohol to oil, type, concentration of the catalyst, reaction temperature, and reaction time can affect the transesterification process [7]. In addition, in alkali-catalyzed transesterification, the use of oil with a higher FFA content can lead to soap formation and difficulty in the purification of biodiesel. The difficulty in the purification of biodiesel causes an increase in the overall production cost [8].

The use of heterogeneous catalysts will allow the design of a reliable, continuous process and improve the economy of biodiesel production [9]. In comparison to homogeneous catalysts, the removal of heterogeneous catalysts from biodiesel is easy and does not generate wastewater. Among these, CaO was the most popular and demonstrated generally better performances [10,11]. However, transesterification reaction using CaO catalyst was slow and required a longer reaction time for higher conversion than homogeneous catalysts. Therefore, high concentration of CaO was required to produce high yield of biodiesel. The effect of CaO concentration on the biodiesel yield was crucial as reported by several researchers [12,13].

The production of biodiesel carries a high cost since the raw material and catalyst used are quite expensive and the condition of the reaction involves a complex and costly process [14]. Therefore, in order to ensure the production still gives a profit, it is important to determine the optimum conditions of the process for the highest yield of biodiesel. Most of the articles on the production of biodiesel deduced that the concentration of catalyst gives a tremendous effect on the biodiesel yield [15-17]. This paper reports the results of the study to investigate the effect of catalyst concentration on biodiesel yield and to determine the optimum process condition using RSM. CaO heterogeneous catalyst was selected due to its superiority in transesterification as reported by [18-20], while palm oil was used as the raw material.

2. Methodology

2.1 Identification of the Problem

In this study, the main problem that has been identified is the catalyst concentration gives a significant effect on the biodiesel yield. A little increase in the concentration of catalyst will significantly boost the production of biodiesel. The increase in the catalyst concentration that exceeds the optimum condition however may reduce the yield of biodiesel.

Other than that, in order to determine the optimum condition where the yield of biodiesel is at the highest percent, the experiment needs to be run many times. The cost to run the experiment is quite high and may cause losses. As reported by Ibrahim *et al.,* [21], the optimum condition of transesterification reaction for producing biodiesel can be obtained by using RSM method. Using this method, high accuracy of results can be obtained even with a smaller number of experiments. RSM is commonly used for the optimization of the chemical process and experimental design [22].

2.2 Data Collection

About 68 data of historical experimental were collected. The data were collected based on the scope of this research. Only studies that use calcium oxide catalysts and palm oil as their raw material were collected. The study of catalyst concentrations was limited to a range of 1 wt% to 15 wt%, reaction temperatures of 55 ºC to 75 ºC, methanol to oil ratio of 5:1 to 25:1, and constant reaction time of 3 hours.

2.3 Determination of the Variables

The dependent variable in this study is the yield of biodiesel. The factors that give effects the yield of biodiesel was classified as independent variables. Thus, the independent variables in this study are catalyst concentration, methanol to oil molar ratio, and reaction temperature.

2.4 The Decision of Factor Level

From a practical perspective, the actual levels allocated to the variables are important. This is due to the fact that the quadratic may lose its validity when the ranges between the two extreme levels of some variables are too wide. On the other hand, when the range is too narrow, some parameter estimations may lose their significance. So, in this study, the selected level variable is a five-level variable.

2.5 Selection of Proper Design

In order to decide the optimum condition for the transesterification of palm oil, Response Surface Methodology (RSM) via Central Composite Design (CCD) will be used. Catalyst concentration (X_1), the molar ratio of methanol to oil (X_2), and temperature of the reaction (X_3) will be the independent variables. This experiment would study the influence of each independent variable and the relationships between these variables on the production of the dependent variable FAME. The CCD for 3 variables and 5 levels is depicted in Table 1.

Table 1
Central Composite design (CCD) independent variables

Factors	Unit	Level				
		- α	-1	0	+1	+ α
Catalyst concentration (X_1)	%	1	4	8	12	15
Methanol to oil molar ratio (X_2)		5	9	15	21	25
Reaction temperature (X_3)	°C	55	58	65	72	75

In the figure, the CCD can be made rotatable by selecting an axial spacing α, in which the value of α can be obtained in equation 1. The independent variables will be manipulated for about five different values.

$$\alpha = (2^{k})^{0.25} \quad (1)$$

As the value of k = 3 (number of independent variable), $\alpha = (2k)^{0.25} = 1.682$. The total number of experiments can be calculated using Eq. 2.

$$The\ total\ number\ of\ experiments = 2^{k} + 2k + n \quad (2)$$

Where; *k* is referred to the number of independent variables and *n* is the number of repetitions and is 6, because six sources were used for one data for each variable. Therefore, the total number of data experiments used was 20.

2.6 Evaluation of the Model

The model was analyzed statistically in order to evaluate the analysis of variance (ANOVA) and the suitability of the empirical model. The ideal confidence level in this research was 95%. As a tool for assessing the relevance of each coefficient, P-values (probability of error values) are employed to indicate the interaction strength of each parameter.

2.7 Optimization of the Model

The optimized model will be generated from Minitab software. The optimum process condition can be generated from Minitab software. The independent variables will be manipulated for about five different values. Then all the different value of the variable was classed into points of factorial design, axial points, and central point.

3. Results

Table 2 represents the experimental layout for a three-variable, five-level combination in a central composite design (coded). The experimental layout was given points which are factorial design points, axial points, and central points.

Table 2
The experimental layout for a three-variable, five-level combination in a central composite design (coded)

Std		Catalyst concentration, wt% (X_1)	Methanol to oil ratio, mol/mol (X_2)	Reaction temperature, ˚C (X_3)	FAME Yield, %
1	Axial point	- α	0	0	65
2	Factorial design point	-1	+1	+1	56.16
3	Factorial design point	-1	+1	-1	63.05
4	Factorial design point	-1	-1	-1	56.62
5	Factorial design point	-1	-1	+1	52.79
6	Central point	0	0	0	97.57
7	Central point	0	0	0	96.89
8	Central point	0	0	0	97.85
9	Axial point	0	0	- α	72
10	Axial point	0	0	+α	65
11	Axial point	0	- α	0	64
12	Axial point	0	+α	0	67
13	Central point	0	0	0	96.23
14	Central point	0	0	0	97.62
15	Central point	0	0	0	96.77
16	Factorial design point	+1	+1	+1	68.51
17	Factorial design point	+1	+1	-1	74.97
18	Factorial design point	+1	-1	-1	70.17
19	Factorial design point	+1	-1	+1	66.22
20	Axial point	+α	0	0	85

Eq. 3 shows the regression equation in the uncoded units.

$$Yield = -1253.9 + 9.040\ X1 + 11.113\ X_2 + 38.015\ X_3 - 0.0141\ X_1X_2 - 0.0014\ X_1X_3 - 0.01658\ X_2X_3 - 0.4620\ X_{12} - 0.32180\ X_{22} - 0.29342\ X_{32} \quad (3)$$

3.1 Analysis of Variance (ANOVA)

The analysis of variance (ANOVA) was carried out to examine the relevance and fitness of the quadratic polynomial regression model. As for ANOVA, the model's adequacy was measured by F-value, P-value, and the significance of the “Lack of Fit”.

Table 3 shows the analysis of variance with the interpretation of the significance level. The greater the F-value and the lower the ‘P' value (Prob. > F) results in the higher significance of the related coefficient. Furthermore, the model terms are only significant when the P-values are less than 0.05. X_1, X_2 and X_3 are significant model terms in this study. Due to the catalyst concentration (X_1) has the highest F-value (484.74), it is the primary variable affecting the FAME yield, whereas the molar ratio of methanol to oil (X_2) and reaction temperature (X_3) are the secondary and tertiary variables, respectively.

Table 3
Analysis of variance with interpretation of significance level

Source	DF	Adj SS	Adj MS	F-Value	P-Value	
Model	9	4897.10	544.12	500.81	0.000	Significant
Linear	3	642.22	214.07	197.03	0.000	Significant
Catalyst concentration	1	526.66	526.66	484.74	0.000	Significant
Methanol to oil ratio	1	35.35	35.35	32.53	0.000	Significant
Reaction temperature	1	80.21	80.21	73.83	0.000	Significant
Square	3	4250.08	1416.69	1303.92	0.000	Significant
Catalyst concentration	1	905.62	905.62	833.53	0.000	Significant
Methanol to oil ratio*Methanol to oil ratio	1	1891.01	1891.01	1740.49	0.000	Significant
Reaction temperature*Reaction temperature	1	1862.68	1862.68	1714.41	0.000	Significant
2-Way Interaction	3	4.81	1.60	1.48	0.280	Not-significant
Catalyst concentration*Methanol to oil ratio	1	0.92	0.92	0.84	0.380	Not-significant
Catalyst concentration*Reaction temperature	1	0.01	0.01	0.01	0.918	Not-significant
Methanol to oil ratio*Reaction temperature	1	3.88	3.88	3.57	0.088	Not-significant
Error	10	10.86	1.09			
Lack-of-Fit	5	8.92	1.78	4.58	0.060	Not-significant
Pure Error	5	1.95	0.39			
Total	19	4907.97				

The weighted total of the squared deviations between the mean response at each parameter level and the corresponding fitted value represents any lack of fit. The P-value for lack of fit is 0.060, which is greater than 0.05 indicating that there was no significant contribution to a pure error. No significant lack of fit means that the model is good. After fitting the model to the experimental data, an F-value of 4.58 implies a 6.00% likelihood of poor fit due to noise. The coefficient of determination (R^2) was used to determine the quality of the model fitness at the relative of this term. R^2 also implies that the independent parameters are well correlated.

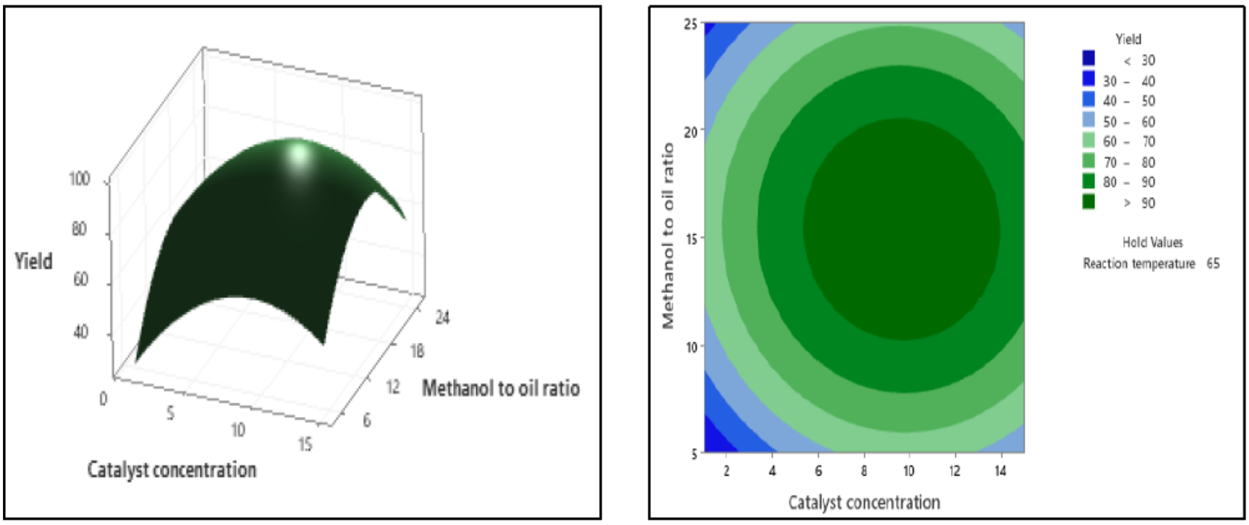
The determination coefficient (R^2) of 0.9978 indicates that the model can account for 99.78% of the response variability observed in the experimental data. The reasonable agreement between the Pred R^2 and the Adj R^2 values of 0.9849 and 0.9958, respectively, indicates that the regression model is very significant. The standard deviation of the model was 1.04235. A low standard deviation suggests a good model that closely matches predicted and actual response values.

3.2 Response Surface Plots for Palm Oil Biodiesel Production

In order to understand the effect of the independent variables used in the optimization process, the interactive effect of the two factors on the transesterification process for biodiesel production was observed.

3.2.1 Interaction effect of catalyst concentration and methanol to oil molar ratio

Figure 1 shows the effect of catalyst concentration and methanol to oil molar ratio on the yield of biodiesel. The catalyst concentration and methanol to oil molar ratio were varied between 1 to 15 wt. % and 5:1 to 25:1 respectively. The reaction temperature was kept constant at 65 °C.



(a) 3D response surface plot (b) 2D contour plot

Fig. 1. Effect of catalyst concentration and methanol to oil ratio on the palm oil biodiesel yield at 65 °C

According to Figure 1(a) and (b), the yield of biodiesel increases as the catalyst concentration is increased by 9.2 wt%. It is usually assumed that raising the catalyst concentration results in a higher yield of triglycerides to fatty acid methyl esters due to greater active site availability [23].

However, this presumption is not always correct. It was discovered that the FAME yield decreases linearly with increasing catalyst concentration for given methanol to oil molar ratios and reaction temperatures. For example, raising the catalyst concentration to 15 wt% and methanol to oil ratio to 15:1 could result in a biodiesel yield of less than 80%.

The lowest biodiesel yields were observed when the maximum concentrations of methanol and catalyst were utilized in the transesterification process. Increases in catalyst concentration to above

10 wt % results in a drop in biodiesel yield. This is because the catalyst particles become saturated inside the reaction mixture. Thus, reducing the surface area of interaction between the methanol and oil molecules [24]. According to Kouzu *et al.,* [25] sufficient amount of CaO can improve the purity of methyl ester. However, an excess amount of CaO can cause the production of diglyceride and soap production which reduces the yield of biodiesel. The formation of soap due to the high basicity of the mixture also influences the decrease in FAME yield as reported by Abdullah *et al.,* [26].

When the methanol to oil molar ratio was 15:1, the yield of oil to biodiesel was found to be lower at 15 wt% catalysts than at 9 wt% catalysts. This is because as the catalyst concentration was increased further, particle cohesion and agglomeration will lower the active surface area and increase the viscosity of the solution, thus lowering the biodiesel yield [24]. According to Silva *et al.,* [27] higher concentration of catalyst could result in the production of emulsion and phase separation which reduces the biodiesel yield.

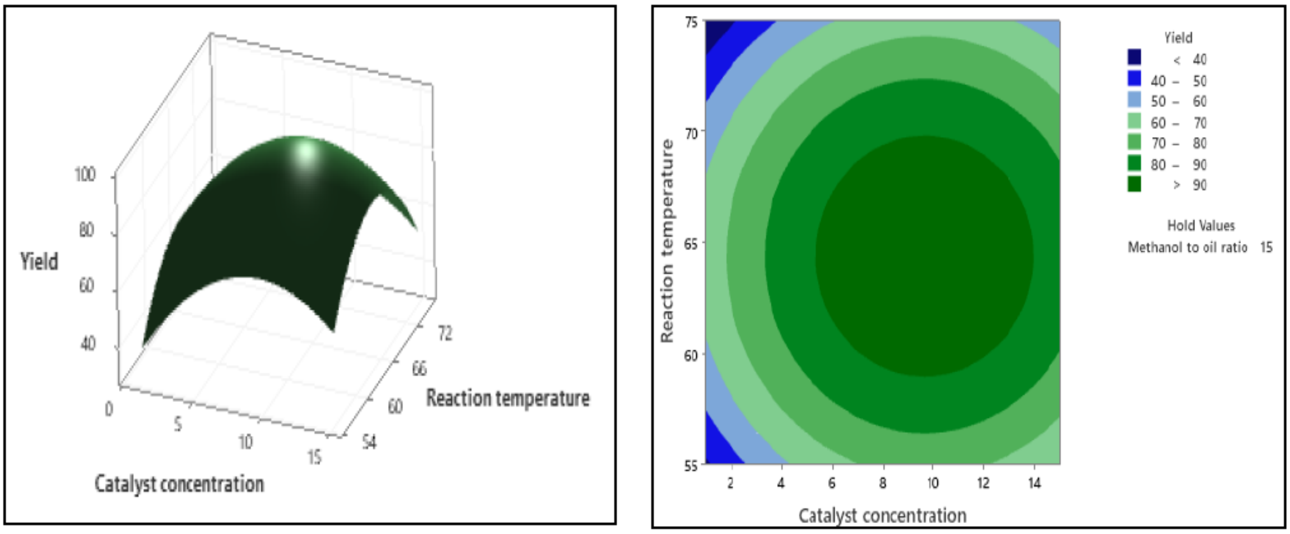
The biodiesel yield decreases as the increase of methanol to oil ratio exceed the optimum condition. The excess of methanol could hinder the glycerol separation process. This drop could be attributed to an overabundance of methanol, which interferes with the separation of alkyl ester and glycerol by increasing the solubility of glycerol. A higher amount of methanol also lowered the concentration of oil and decreased the biodiesel yield as reported by Wijittra *et al.,* [28]. This finding was also reported by Verma & Sharma [29].

3.2.2 Interaction effect of catalyst concentration and reaction temperature

Figure 2 shows the combined effect of catalyst concentration and reaction temperature on the biodiesel yield. This study was carried out at different temperatures from 55-75 ^{o}C with different catalyst concentrations from 1-15 wt%. The molar ratio of methanol to oil was 15:1. The reaction rate was slow at low temperatures due to the diffusion resistance, as the heterogeneous catalyst forms a three-phase system, oil-methanol-catalyst. It was observed that temperature has a positive effect on the biodiesel yield. For example, the percentage of FAME yield increases with the increase in the reaction temperature up to 64 ^{o}C at 9 wt% of catalyst and 15:1 methanol to oil molar ratio.

However, the percentage of FAME yield decreases as the increase in reaction temperature of above 65 °C at 10 wt% of catalyst and 15:1 methanol to oil molar ratio. The reaction temperature at high temperatures must be avoided since there is a chance of methanol loss due to vaporization at high temperatures [26]. According to Britannica [30], the boiling point of methanol is 65 °C. This means that the methanol will be vaporized at 65 ^{o}C and above. As the methanol was vaporized, there will be insufficient methanol to react with triglyceride in the transesterification process which results in less percentage of biodiesel produced. High biodiesel yield was obtained around 64 ^{o}C with a catalyst concentration of approximately 9.2 wt% and a methanol to oil molar ratio of 15:1. This was in good accordance with the result obtained by other researchers [31-33].

Other than that, the increase in the concentration of the catalyst will result in the increase in the percentage of FAME yield and the FAME yield will start to decline as it exceeds the optimum point. As a result of increasing the catalyst concentration exceeding the optimum point, the mixture of catalyst and reactants could become too viscous and may cause problems in the interaction between the reactants and active sites [34]. This limitation of the mass transfer can lead to a decrease in the yield of biodiesel [26]. On the other hand, when the catalyst loading amount was not enough, the maximum production yield could not be reached. High biodiesel yield was obtained at around 9.2 wt% catalyst concentration at 64 °C. This was in accordance with the result obtained by Viola *et al.,* [35]. However, this researcher used fried vegetable oil as feedstock for biodiesel production. As the catalyst used was CaO, then the increase in the reaction temperature above 64 °C will not give effect to the catalyst.



(a) 3D response surface plot (b) 2D contour plot

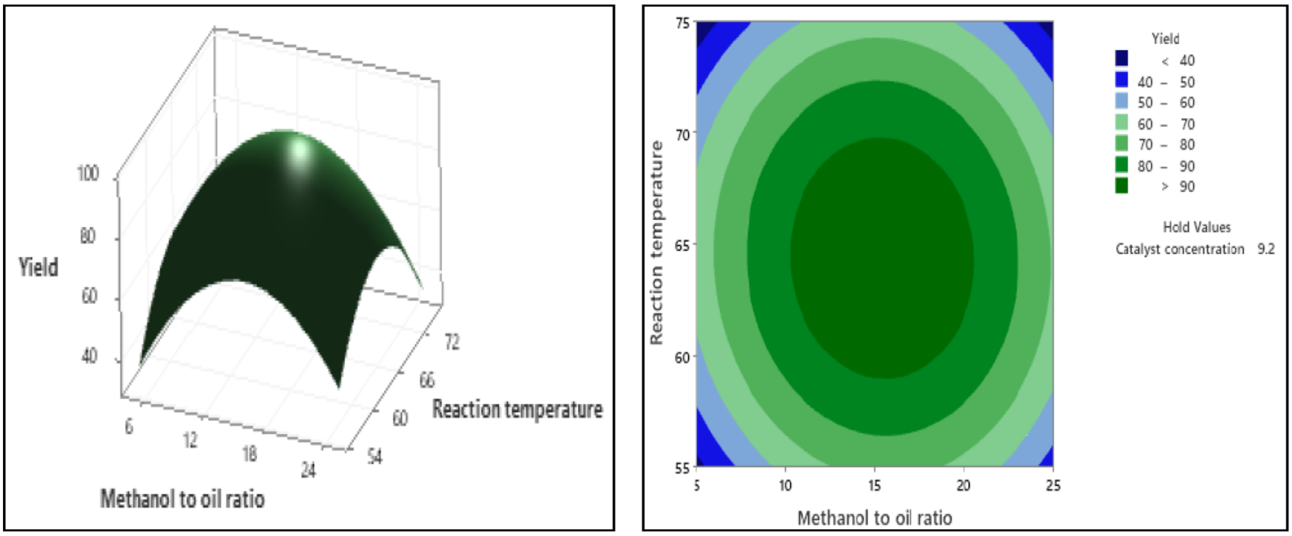
Fig. 1. Combined effect of catalyst concentration and reaction temperature at 15:1 methanol to oil ratio

3.2.3 Interaction effect of methanol to oil molar ratio and reaction temperature

Figure 3 depicts the effect of temperature and the methanol to oil molar ratio on biodiesel yield. The increase in the methanol to oil molar ratio will also result in an increase in the yield of biodiesel at a constant temperature. As shown in the figure, the yield of biodiesel at 5:1 methanol to oil molar ratio is 55% and at 15:1 methanol to oil molar ratio, the yield of biodiesel is 98 % at a constant reaction temperature and catalyst concentration of 65 °C and 9 wt% respectively. This trend shows an increase in biodiesel yield.

The molar ratio of methanol to oil has a significant impact on the percentage conversion of oil. The transesterification chemical equation's stoichiometry balance required three moles of methanol to react with one mole of triglyceride. However, in this reversible reaction, a high methanol to oil molar ratio would favour the conversion of oil to biodiesel. The result in Figure 3 shows that the percentage yield of biodiesel increases as the methanol to oil ratio increases. At a low methanol-to-oil ratio (5:1), the methanol present was insufficient to drive the equilibrium reaction to completion, resulting in a low percentage of FAME yield [26]. Many previous studies agreed that exceeding the best ratio of methanol would dilute the system and cause a decrease in yield due to less contact between the active site of the catalyst and the oil [34].

The percentage of biodiesel yield increases with increasing temperature up to 64 °C and then slightly decreases with further increase in temperature. As a result, the optimal temperature for this transesterification reaction was around 64 °C. High temperatures give molecular reactants more kinetic energy [25], which eventually speeds up the reaction rate. The decrease in the biodiesel yield above the optimum temperature can be attributed to methanol evaporation. Methanol has a boiling point of 65 °C, so it vaporizes easily at 65 °C. The reaction temperature influences both the reaction rate and the yield of biodiesel [36]. The increase in temperature will only affect the composition of methanol in oil as the methanol will start to vaporize at its boiling point. Because the transesterification reaction is reversible, the transesterification could be sped up by increasing the amount of methanol [37].



(a) 3D response surface plot (b) 2D contour plot

Fig. 1. Combined effect of methanol to oil ratio and reaction temperature at 9.2 wt% catalyst concentration

The estimation of the optimum process conditions for these three variables was based on the findings in these three figures. As shown in the figures, the center of the contour will be the area for 90%. It covers many points of the combination of both process parameters for each figure. Therefore, based on the estimation from the figures, the optimum condition for the biodiesel synthesis was at the catalyst concentration of 9.63 wt%, methanol to oil molar ratio of 15.30:1, and reaction temperature of 64.40 °C. The yield of the biodiesel produced will be 98.65%.

3.3 Validation of the Response Surface Methodology Results

The optimum operating conditions for biodiesel yield from the palm oil by transesterification process were 9.63 wt% catalyst concentration, reaction temperature 64.40 °C, and methanol to oil ratio 15.30:1. The maximum yield obtained is 98.6514%. As in this study, the heterogeneous CaO catalyst was used, and then the result obtained was compared with several previous studies that use CaO as their catalyst by referring to Table 4. A study by Qu *et al.,* [36] obtained a successful conversion of palm oil to biodiesel at 9.21 wt% of heterogeneous base catalyst concentration, the molar ratio of methanol to palm oil of 15.54 and a temperature of 64.2 °C with 98.46% FAME yield. Thus, it can be said that the result obtained from this study is valid as the result obtained was slightly similar to the previous researcher.

Table 4
Comparison from other related studies

Catalyst	Feedstock	Catalyst conc (wt%)	Methanol to oil molar ratio	Temp (°C)	Yield (%)	Reference
KNO_3 /CaO	Rapeseed oil	1	6:1	65	98	[27]
CaO/wollastonite	Palm oil	9.21	15.54	64.2	98.46%	[9]
CaO/Al_2O_3	Palm oil	5.97	12.14:1	64.29	98.64	[28]
CaO/mesoporous silica	Soybean oil	5	16:1	60	95.2	[29]
CaO	Sunflower oil	1	6:1	80	91	[30]
Heterogeneous base catalyst (CaO)	Palm oil	9.63	15.30:1	64.40	98.65	This study

4. Conclusions

From this study, it is observed that the increase in catalyst concentration increases the percentage of biodiesel yield. However, the percentage of biodiesel yield decreases after the reaction exceeds the optimum concentration of the catalyst. This is because of sufficient amount of CaO can improve the production of biodiesel, but the excess amount of CaO will reduce the yield produced. It is predicted due to the production of diglyceride and soap production. The possible optimum condition for the biodiesel synthesis obtained using RSM method was at the catalyst concentration of 9.63 wt%, methanol to oil molar ratio of 15.30:1, and reaction temperature of 64.40 °C. Under the mentioned condition, the yield of the biodiesel produced was recorded at 98.65%.

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