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Experimental Optimization of Castor Oil Transesterification by Central Composite Design for Biodiesel Production

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Abstract. Thailand is an agriculture-based country with the potential to cultivate a vast array of plant species, including castor. Castor oil is produced by pressing castor seeds. Castor oil was selected as the preferred vegetable oil for biodiesel production. Castor oil is primarily composed of ricinoleic acid, a hydroxyl fatty acid. Response surface methodology was used in this study to optimize the biodiesel production process parameters. This study varies the molar ratio of methanol to oil, the concentration of the catalyst, the reaction temperature, and the reaction time. As a catalyst, potassium hydroxide was used in the transesterification process. In this study, response surface methodology is utilized in conjunction with central composite design (CCD) experiment design. Therefore, the optimal yield of castor oil transesterification is 4.02 methanol to 1 oil, a catalyst concentration of 0.90%, a reaction temperature of 49.87 ºC, and a reaction time of 59.21 minutes. These optimal conditions resulted in a %fatty acid methyl ester (FAME) yield of castor oil biodiesel of 88.25 %, which is within 5% of the predicted %FAME yield. Transesterification under optimal conditions demonstrates that the physiochemical properties of castor oil biodiesel are enhanced. The viscosity of castor oil is approximately 235 cSt at 40 ºC. After transesterification, the viscosity of castor oil decreases to 15.2 cSt at 40 ºC under optimal conditions. The density and flash point of castor oil biodiesel is 0.92 g/cm^3 and 196 ºC, respectively. It discovers that the flash point of castor-oil biodiesel complies with the American Society for Testing and Materials (ASTM) standard, whereas its viscosity and density do not. However, castor oil biodiesel can be blended with diesel petroleum to reduce its viscosity and meet ASTM specifications.

Keywords. Castor oil, biodiesel, response surface methodology, homogenous catalyst

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Nomenclature	List of abbreviations
ASTM	American Society for Testing and Materials
ANOVA	Analysis of variance
CCD	Central composite design
CaO	Calcium oxide
FAME	Fatty acid methyl ester
KOH	Potassium hydroxide

1. Introduction

Diesel fuel is in high demand in Thailand. According to the paper, diesel fuel might be utilized in multiple industries, including the generation of energy, transportation, and agriculture. Thailand consumed roughly 58,000 millionliters of diesel fuel per day [1], which was insufficient. Diesel fuel must be imported from another nation. The importation of oil fuel was classified into two categories: refined oil and crude oil. The distillation of crude oil produced refined oil. Each year, oil imports cost a significant amount of money and foreign currency. The present finding that the price of crude oil has increased tends to mirror the worldwide market. This is establishing a picture of alternative energy and renewable energy, particularly because biodiesel is an agricultural product, with the majority of biodiesel derived from palm, bean, and jatropha seeds. Instead of diesel, biodiesel might create renewable energy [2].

Biodiesel has a viscosity comparable to diesel oil; its viscosity is steady and changes minimally with temperature. In addition, biodiesel has a high flash point, indicating that it is safe to use. The cetane number is an indicator of the quality of an oil's flammability, and biodiesel has a higher cetane number than diesel fuel. It was discovered that biodiesel has the potential to replace diesel oil as a source of renewable energy [3]. The production of biodiesel begins with the reaction of vegetable oil and alcohol in the presence of catalysts such as acids, bases, or enzymes. This reaction involving vegetable oil and alcohol is known as transesterification [4].

Transesterification by using base catalysts was widely used for biodiesel production since base catalysts could react at a lower temperature and reaction time than acid catalysts [5]. Nevertheless, a reaction with a base catalyst produces soap between the base and methyl ester in the saponification reaction, in which water is involved. Soap from transesterification is not the primary product in the production of biodiesel, which could be avoided by eliminating water before the production of biodiesel [6].

Thailand is an agricultural country, as Thailand can cultivate a variety of plants, including oil crops such as jatropha seeds, coconut, sesame, and castor [7]. Castor oil was used to produce biodiesel in this study. Resin lubricant and grease industries reaped the benefits of castor oil. High viscosity and an allergen-inducing substance are disadvantages [8].

Castor oil is made from castor seeds (figure 1) [9] and contains 85-90% hydroxyl fatty acid, ricinoleic (Figure 2) [10] and 10% non-hydroxyl fatty acid, such as oleic and linoleic acid, which can be used to make biodiesel [11]. Furthermore, while castor seeds are not suitable for human consumption, their use as an energy source does not compete with food production. That is the reason for castor seed prices being lower than other vegetable oils [12]. Therefore, in this study, we synthesized biodiesel from castor oil through the transesterification process.

Figure 1. Castor seed [10].

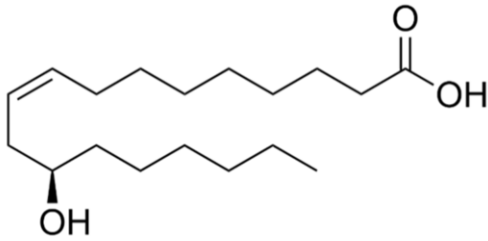

Figure 2. Ricinoleic structure [13].

Castor oil transesterification, such as through homogeneous catalysts, necessitates a lot of work. Kartikkumar Thakkar et al. [14] used multi-response optimization of castor oil with a hydrodynamic cavitation reactor. It was found that the optimum yield (92.31%) in the conditions of methanol:oil (10.3:1), 1.05% wt. catalyst concentration and 58.94 min of reaction time was achieved. Kun Liu et al. [15] synthesize castor oil biodiesel using mixed metal oxide as a catalyst. The optimum yield (91.1%) was obtained with a methanol:oil catalyst concentration of 8%wt, a reaction temperature of 70 ºC, and a reaction time of 4 hours. Sana Gohar Khan et al. [16] produce castor oil biodiesel using CaO nano-catalyst from mussel shell based. The optimum yield (87.42%) was obtained from 2.5wt% catalyst concentration, methanol:oil (8:1) and a reaction temperature 65 ºC. Mostafa R. Abukadra et al. [17] used response surface optimization of castor oil by using CaO nanorods. The optimum yield (95.4%) was achieved through 2.5 h of reaction time, 120 ºC reaction temperature, and a 15:1 ethanol:oil molar ratio. The objective of this study is to produce biodiesel from castor oil by using a potassium hydroxide-based catalyst through the response surface method in the following ranges: methanol:oil (4:1-10:1), catalyst concentration (0.5-1.5), reaction temperature (40-60 ºC), and reaction time (30-60 min). This study varies four independent parameters, namely the molar ratio of methanol to oil, the concentration of the catalyst, the reaction temperature, and the reaction time, in contrast to other studies that vary only three independent parameters. This research uses potassium hydroxide as a transesterification catalyst which used a temperature lower than the CaO catalyst in the transesterification reaction. CaO catalyst cannot produce pure glycerol from vegetable oil, but potassium hydroxide can. In addition, this study maintained a constant mixing speed of 600 rpm.

2. Materials and Method

The transesterification reaction in this study used refined castor oil supplied by the Tropicalife company. Castor oil has a high viscosity and is processed using 99% analytical methanol and potassium hydroxide as catalysts. Castor oil has high viscosity compared to other vegetable oils. Table 1 shows the fatty acid compositions of castor oil [14].

Table 1. Fatty acid composition [14].

Fatty acid composition	Percentage (%)
Palmitic, C16:0	1.3
Stearic, C18:0	1.2
Oleic, C18:1	5.5
Linoleic, C18:2	7.3
Linolenic, C18:3	0.5
Ricinoleic, C18:1	84.2

2.1. Determined Acid Values

Acid values are the amount of sodium or potassium hydroxide that can react with fatty acids in vegetable oil when compared to the percentage of oleic acid (1 gram) [18]. Due to the high concentration of ricinoleic acid in castor oil, the acid values of the fatty acids in castor oil should be compared to those of ricinoleic acid. In this study, sodium hydroxide was compared with ricinoleic acid in castor oil using the titration method with a phenolphthalein indicator. This equation is used to determine acid values: (1)

$$\text{Acid value} = \frac{\text{volume of KOH} * \text{concentration of KOH} * 56.1}{\text{amount of Sample}} \quad (1)$$

2.2. Experiment Set-Up

Castor oil is used as a reactant with methanol as an alcohol, and potassium hydroxide as a catalyst in transesterification. Figure 3 shows how the experiment was set up using a 250-millilitre reactor with a reflux condenser and a magnetic stirrer. Begin by adding castor oil to the reactor and adjusting the temperature to the desired level. After the temperature of the oil reached the temperature threshold, the reactor was mixed with potassium hydroxide and methanol at 600 RPM. Continuously stir the mixture until the desired time is reached. Load the biodiesel into the separator funnel to separate the glycerol and biodiesel. When the biodiesel has been separated, store it in a bottle for later analysis.

Figure 3. Transesterification reaction experiment setup.

2.3. Transesterification Response Surface Optimization

This study used the Design-Expert software version 13 (Trial version) for the design experiment. Optimization of the response surface of a transesterification reaction using castor oil as a reactant and potassium as a catalyst. As a castor oil potassium catalyst transesterification process, a transesterification response surface optimization experiment utilizing a central composite design (CCD), the experiment, model, and castor oil methyl ester yield was designed. In this study, four independent variables were coded into three levels: methanol:oil molar ratio, catalyst concentration, temperature, and time. The axial points (alpha) are coded as alpha and +alpha about the central point shown in Table 2.

Table 2. The design of experiment of the transesterification response surface optimization experiment.

Variable	Unit	Coded factor levels				
		-alpha	-1	0	1	+alpha
Methanol:oil	molar ratio	1.95	4	7	10	12.05
Catalyst	%w/w	0.16	0.5	1	1.5	1.84
Temperature	ºC	33.18	40	50	60	66.82
Reaction time	min	19.77	30	45	60	70.23

Multiple regression was initially used to fit the response to the factors. Using the coefficients of determination and analysis of variance, the quality of model fit was determined. To equation (2), the quadratic response surface model was fitted.

$$Y = \beta_{k0} + \sum_{i=1}^{3} \beta_{ki} x_i + \sum_{i=1}^{3} \beta_{kii} {x_i}^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{kij} x_i \, x_j \quad (2)$$

Where Y is the response variable or fatty acid methyl ester contents, β_0 is the intercept, β_i is the first-order model coefficient, β_{ii} is the quadratic coefficient for the factor i, and β_{ij} is the linear model coefficient for the interaction between factors i and j and x_i is the *i*th independent variable.

3. Result and Discussion

3.1. Acid Value

Using titration with potassium hydroxide and phenolphthalein as indicators, the acid value of castor oil was calculated. The average acid value is 0.32%, which is lower than the recommendation; if the acid value is greater than 2%, the oil must reduce free fatty acid through an esterification reaction with acid [19].

3.2. Response Surface Optimization of Transesterification

A CCD was used in this study to generate the 21 experimental conditions in Table 3 that were used to investigate the effect of the four factors on castor oil methyl ester. Four

centre points were used to approximate the proposed model's lack of fit and pure error [20]. Multiple regressions were used to fit the coefficient of the quadratic polynomial regression response model. In the transesterification experiment, the castor oil methyl ester yield ranged from 35.9% to 88.8%, with the highest content resulting from reaction conditions of methanol:oil 1.95:1, potassium hydroxide concentration of 1% w/w, reaction temperature of 50 ºC, and reaction time of 45 minutes.

Table 3. CCD with actual castor oil methyl ester yield.

Run	Molar ratio	% Catalyst (%w/w)	Temperature (ºC)	Time (min)	%FAME Yield
1	10	1.5	60	30	78.08
2	7	1	50	45	74.00
3	7	1.84	50	45	47.03
4	7	1	50	19.77	71.21
5	7	1	50	45	74.50
6	12.04	1	50	45	63.02
7	4	0.5	40	30	50.30
8	10	1.5	40	30	63.18
9	7	1	33.18	45	71.08
10	1.95	1	50	45	88.77
11	10	0.5	40	60	74.25
12	7	1	50	45	78.00
13	7	1	50	70.22	86.75
14	7	1	50	45	78.00
15	4	0.5	60	30	48.61
16	7	1	50	45	78.00
17	4	1.5	40	60	60.05
18	10	0.5	60	60	68.90
19	7	1	66.82	45	75.61
20	7	0.16	50	45	35.90
21	4	1.5	60	60	72.37

The response variable is the yield of castor oil methyl ester, which is influenced by four parameters: the methanol:oil molar ratio, the percentage of catalyst concentration, the reaction temperature, and the reaction duration. The equation was developed to describe the relationship between the yield of castor oil methyl ester and input parameters. The significance of the response is validated based on a low p-value, indicating that the p-value should be less than 0.05. The molar ratio of methanol to oil, the concentration of the catalyst, the reaction temperature, and the reaction duration were all significant in this investigation. The variance analysis resulting from the experiment data is represented in Table 4.

The adjusted R^2 value of the response surface optimization was 0.9791 (Table 5) and was used to recognize the sufficiency and adaptability of the model [21]. The difference between adjusted and predicted R^2 is less than 0.2, indicating that the response surface optimization in this study is reasonable. Equation 3 represents the highest castor oil methyl ester model.

$$\%\ \text{FAME Yield} = -59.26856 - 1.80080 * \text{Molar Ratio} + 146.48631 * \text{Catalyst} + 0.551685 * \text{Temperature} + 2.15558 * \text{Time} + 0.137642 * \text{Molar Ratio} * \%\text{Catalyst} - 0.004051\ \text{Molar Ratio} * \text{Temperature} - 0.013982\ \text{Molar Ratio} * \text{Time} + 0.854025 * \%\text{Catalyst} * \text{Temperature} - 1.90823\%\text{Catalyst} * \text{Time} - 0.005281 * \text{Temperature} * \text{Time} - 0.004208 * \text{Molar Ratio}^2 - 48.83087 * \%\text{Catalyst}^2 - 0.009368 * \text{Temperature}^2 + 0.004696 * \text{Time}^2 \quad (3)$$

All positive values can increase the yield, and negative values have a reduced impact on the castor oil. The linear correlation between experimental castor oil methyl ester yields and predicted values for castor oil was shown in Figure 4.

Table 4. Analysis of variance (ANOVA) obtained from experiment results.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	3642.64	14	260.19	67.85	< 0.0001
A-Molar Ratio	332.06	1	332.06	86.59	< 0.0001
B-%Catalyst	61.96	1	61.96	16.16	0.007
C-Temperature	56.2	1	56.2	14.65	0.0087
D-Time	120.81	1	120.81	31.5	0.0014
AB	0.1413	1	0.1413	0.0368	0.8541
AC	0.1182	1	0.1182	0.0308	0.8664
AD	1.31	1	1.31	0.3421	0.5799
BC	145.87	1	145.87	38.04	0.0008
BD	678.73	1	678.73	176.98	< 0.0001
CD	5.02	1	5.02	1.31	0.2961
A^2	0.0214	1	0.0214	0.0056	0.9428
B^2	2227.09	1	2227.09	580.73	< 0.0001
C^2	13.12	1	13.12	3.42	0.1139
D^2	16.69	1	16.69	4.35	0.0821
Residual	23.01	6	3.83		
Lack of Fit	6.01	2	3	0.707	0.5458
Pure Error	17	4	4.25		
Cor Total	3665.65	20			

Table 5. ANOVA of the regression models.

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2
Linear	0.6234	0.0006	-0.0711	-0.5544
2FI	0.7255	0.0003	-0.2602	-3.632
Quadratic	< 0.0001	0.5458	0.9791	0.8758
Cubic	0.5458		0.9768	

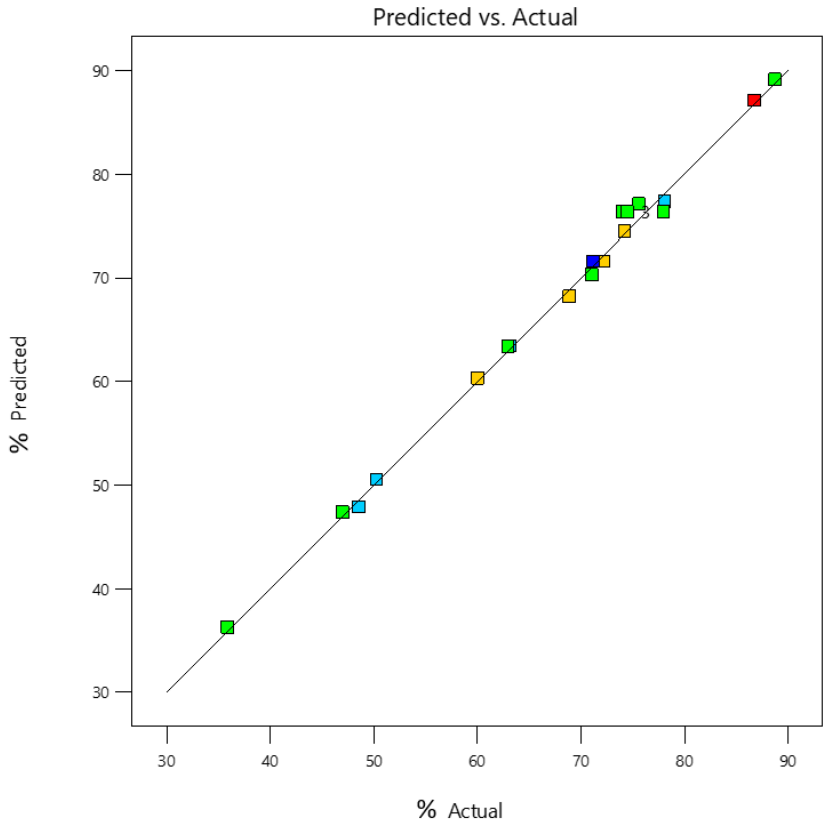

Figure 4. The linear correlation between experimental and predicted values.

3.2.1. The Effect of Molar Ratio and %Catalyst Concentration

Potassium hydroxide is the most effective base homogeneous catalyst for transesterification when compared to other catalyst types. At low catalyst concentrations, the castor oil methyl ester yield was low, resulting in an incomplete transesterification reaction and a low %FAME yield. Because methanol and oil are completely soluble in %FAME, the molar ratio of methanol to oil affected the %FAME decrease at low catalyst concentrations. Similarly, increasing the catalyst concentration to 1.5% and the methanol-to-oil molar ratio to 10:1 caused oil and methanol to become heterogeneous. %FAME decreases at high catalyst concentrations and methanol-to-oil molar ratios. The relationship between the molar ratio and % catalyst concentration is seen in Figure 5.

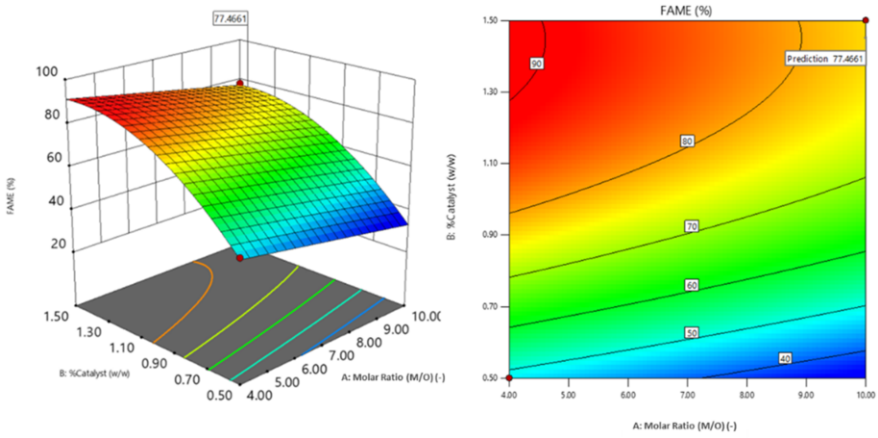

Figure 5. The effect of molar ratio and %catalyst concentration.

3.2.2. The Effect of Reaction Temperature and %Catalyst Concentration

Temperature is one variable that has a lower impact on FAME yield than another. Because the oil and methanol mixture were more concentrated in this study at high temperatures, the %FAME yield increased. However, because methanol has a boiling point of 65 ºC, if the temperature of the reaction increases until it reaches the boiling point, methanol will be boiled, causing the volume of the mixture to decrease [22]. The effect of decreasing mixture volume results in a decrease in %FAME yield. The optimal conditions consist of a reaction temperature of 60 ºC and a concentration of 1.5% catalyst. The impact of reaction temperature and catalyst concentration is illustrated in Figure 6.

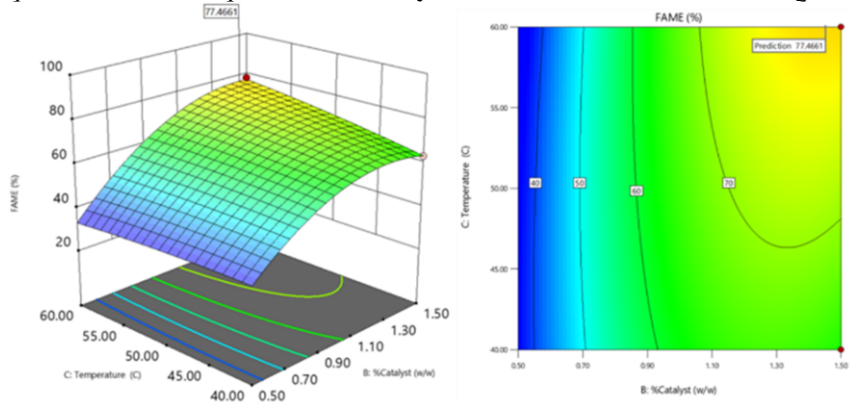

Figure 6. The effect of reaction temperature and % catalyst concentration.

3.2.3. The Effect of Reaction Time and Molar Ratio

The optimum reaction time condition is 60 min with a low molar ratio of methanol to oil at 4:1. The high molar ratio caused the %FAME yield to decrease due to soap that occurred in the reaction. The excess methanol caused triglyceride to react with methanol, resulting in a soap that %FAME yield a decrease. By the way, the short reaction time affected the transesterification reaction incompletely. It was found that in long

reactions, %FAME yield is higher than in short reaction times. The effects of reaction time and the molar ratio is illustrated in Figure 7.

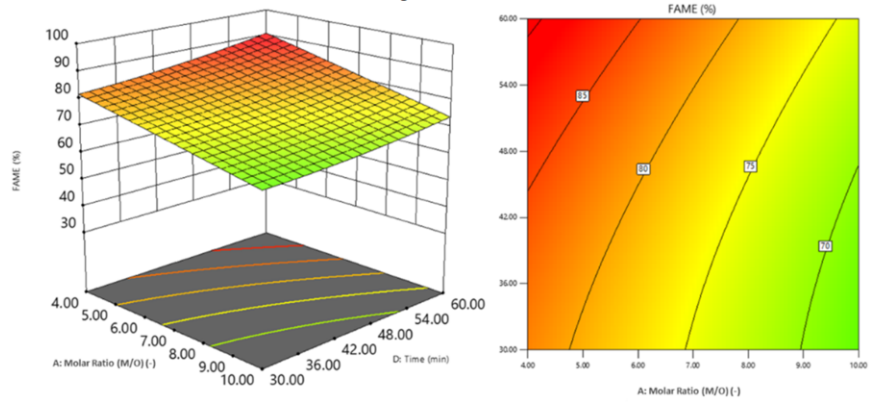

Figure 7. The effect of reaction time and molar ratio.

3.2.4. The Effect of Reaction Time and %Catalyst Concentration

Transesterification at low catalyst concentrations and short reaction times decreases %FAME yield because higher catalyst concentrations require longer reaction times to complete. The increase in catalyst concentration affected the increase in %FAME yield. While a high catalyst concentration with a long reaction time affected %FAME yield, the backward reaction was caused by the long reaction time for transesterification [23]. The long reaction was not advantageous for the transesterification reaction. Figure 8 shows the effect of reaction time and %catalyst concentration.

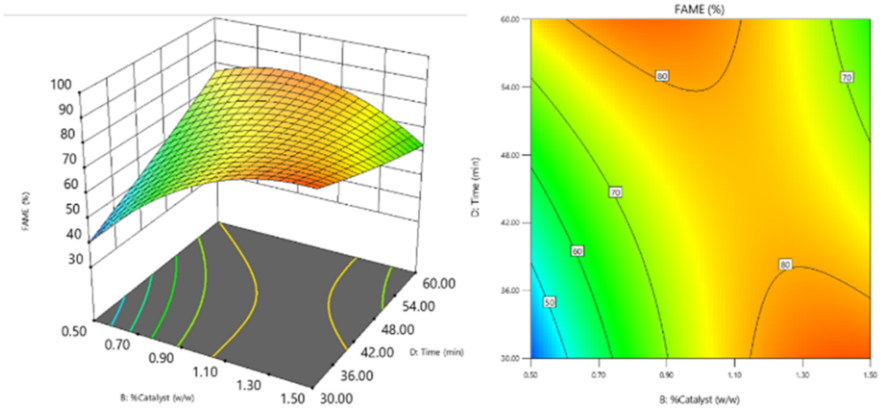

Figure 8. The effect of reaction time and %catalyst concentration.

3.3. %FAME Yield Optimization Condition

In this study, the optimal conditions for the transesterification of castor oil were determined using potassium hydroxide as a catalyst. According to the Design-Expert software version 13 (Trial), the optimal conditions for %FAME yield is a molar ratio of

methanol to oil of 4.02:1, a catalyst concentration of 0.9% by weight, a reaction temperature of 49.87 ºC, and a reaction time of 59.21 minutes. This condition predicts a %FAME yield of 91.368%. Figure 9 shows the optimal reaction conditions for the transesterification of castor oil.

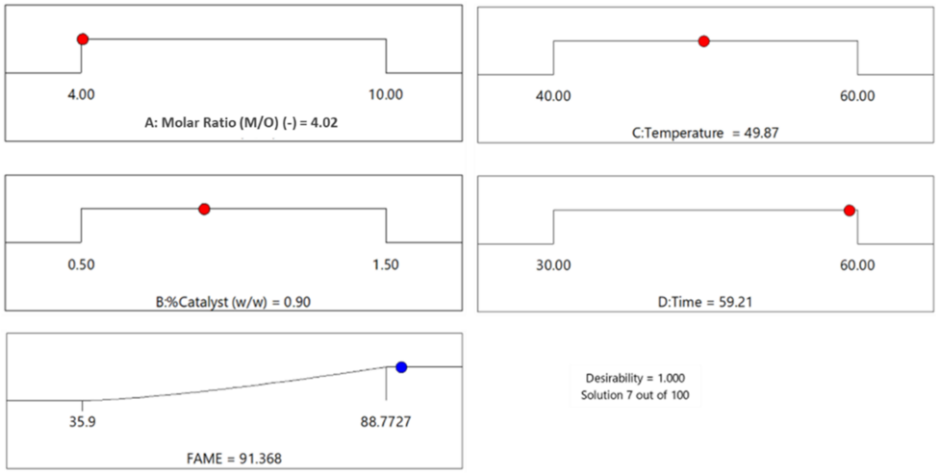

Figure 9. The optimum condition of castor oil transesterification reaction.

The transesterification reaction with the optimum conditions and the predicted %FAME yield gave a 91.368 % FAME yield. The experiment of transesterification in the optimal condition found that the FAME yield reached 89.57%, which is an error value less than 5% from the %FAME yield predicted. The error occurred because the longer transesterification reaction affected the transesterification reverse reaction. The reverse reaction of transesterification decreased the %FAME yield of castor oil.

3.4. Characteristics of Castor Oil Biodiesel

Table 6. Physiochemical properties.

Parameter	Unit	Castor oil biodiesel	ASTM Standard
Density at 15ºC	g/cm^3	0.92	0.86-0.90
Viscosity	cSt	15.2	6.0
Flash point	ºC	196	>120

Table 6 shows the physicochemical parameters of castor oil biodiesel. Transesterification can be used to decrease the viscosity of castor oil. After transesterification, castor oil has a viscosity of 15.2 cSt at 40 ºC, which is greater than the ASTM D 445 standard. The effect of viscosity on internal fluid friction or flow resistance during a change in a dynamic fluid motion. However, combining castor oil biodiesel with petroleum diesel may reduce the viscosity of castor oil [24]. Because biodiesel and petroleum have lower viscosities, they are easier to pump and atomize. The flash point of castor oil is 230 ºC before transesterification and 196 ºC after transesterification. A flash point of a fuel is the temperature at which it can catch fire. It is essential from the perspective of safe handling. It is well known that a high flash point increases storage safety. The ASTM standard recommends that the flash point of

biodiesel be greater than 120 °C. The flash point of biodiesel derived from castor oil was discovered to be 196 °C, indicating that biodiesel has a higher flash point than recommended by ASTM [25]. The density of biodiesel produced under optimal conditions from castor oil was determined to be 0.92 g/cm^3 using the ASTM 7550 Method at 15 °C. Due to the hydroxyl group of ricinoleic acid methyl ester, the density was determined to be greater than the ASTM standard. However, adding up to 20% biodiesel to diesel can reduce its density [26].

4. Conclusion

Transesterification of castor oil using potassium hydroxide as a catalyst can be produced biodiesel. The optimal conditions included a molar ratio of 4.02 methanol to 1 oil, a %catalyst concentration of 0.90, a reaction temperature of 49.87 °C, and a reaction time of 59.21 minutes. Under these conditions, an 88.25 % FAME yield was achieved during the transesterification reaction of castor-oil biodiesel. The ASTM standard is met by the physicochemical properties of castor-oil biodiesel, such as flash point, but not density and viscosity. Castor oil biodiesel can be blended with petroleum diesel to create 20% biodiesel blend (B20) and it is an eco-friendly fuel.

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