



Optimization Transesterification Reaction in the Synthesis of Biodiesel from Household Catering Waste Cooking Oil

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Abstract. Biodiesel is an alternative energy source for diesel engines or diesel fuel which is carried out using the transesterification method. Used cooking oil is reacted with methanol which will produce methyl ester and glycerol with the help of a base catalyst in the form of NaOH. The research aims to optimize the biodiesel obtained. The synthesis reaction for biodiesel was carried out at temperatures of 55°C, 60°C, 65°C and with varying times of 60, 90, and 120 minutes, with a volume ratio of used cooking oil-methanol, namely 1:4, 1:5, and 1:6. The highest yield was 61.9404% and ANOVA (*Analysis of Variance*) was proven to be significant and fulfilled as in the *Design Expert 13*.

Keywords: *Used cooking oil, Biodiesel, Transesterification, Optimization, ANOVA*

1. Introduction

The supply of fossil diesel fuel is decreasing by the day due to continuous use. Alternative fuels are a solution that can be used to minimize the use of fossil fuels. Biodiesel has the opportunity to replace diesel fuel because the materials used come from renewable materials [1]. Biodiesel is the name for a type of *fatty ester* and usually comes from vegetable oil or comes from living creatures. Biodiesel itself has much smaller emissions than diesel emissions from petroleum [2].

Biodiesel which is a mixture of mono-alkyl esters of long-chain fatty acids, is a simple fuel used in diesel engines [3]. The benefits of biodiesel itself are as a lubricant to increase engine life and have a high selling price. Biodiesel as an environmentally friendly alternative fuel has the benefit of reducing and overcoming the impact of global warming [4].

The main ingredient for making biodiesel is used cooking oil. Used cooking oil is waste from frying, where the oil contains many dangerous compounds that can pollute the environment and cause disease [5]. Used cooking oil that is used continuously contains a lot of

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free fatty acids which are caused by the high temperature frying process. The formation of free fatty acids results from the hydrolysis process of triglycerides during frying. If the value of free fatty acids in used cooking oil increases, the quality of the used cooking oil will become worse. The free fatty acid content in used cooking oil is generally more than 1% [6]. The chemical content of used cooking oil can be seen in Table 1 below:

Table 1. Chemical Content of Waste Cooking Oil

Parameter	Waste Cooking Oil
Density (gram/cm ³)	0.8989
FFA (%)	9.3
Viscosity (40°C)	46.5
Glyceride Component (%)	56.5
Non-Glyceride Component (%)	43.5

Source: Yozanna, 2016 [7].

The process of making biodiesel from used cooking oil is carried out through several processes, one of which is the transesterification method (conversion of triglycerides into methyl esters) with the help of a catalyst to speed up the reaction [8]. The process of processing used cooking oil into biodiesel, not only requires a catalyst but also requires alcohol as a reactant. The reactant chosen was methanol, because methanol has a cheaper price, is more stable, and has the highest reactivity. The resulting product (if methanol is used) is more often referred to as *fatty acid methyl ester* [9]. The catalysts commonly used are homogeneous acid-base catalysts, such as NaOH and HCl. Homogeneous catalysts tend to have many disadvantages, namely that they can react with *free fatty acids* (FFA) and will form soap, making purification difficult [10].

To maintain and develop the quality of biodiesel production, provisions have been made for biodiesel quality standards. Several countries have set their biodiesel standards or have differences with other countries, one of which is Indonesia the biodiesel standard is set in SNI 7182:2015 with some quality requirements [11], the biodiesel standard can be seen in Table 2 below:

Table 2. Biodiesel Quality Standart

Test Parameters	Unit (min/max)	Standard	Test Method
Acid Number	mg KOH/ gram	0.5	SNI 01-2901-2006
Density at 40°C	kg/m ³	850-890	ASTMD 1298/ASTMD 4052
Cetane Number	Min	51	ASTMD 613

Test Parameters	Unit (min/max)	Standard	Test Method
Flash Point (Bowl Closed)	°C, min	100	ASTMD 7094
Pour Point	°C, max	18	ASTMD9704

Source: SNI 7182: 2015

Table 3. Previous Research

No.	Method	Variable	Catalyst	Result Obtains	Reference
1.	Transesterification	Variations in the ratio of used cooking oil : methanol (1:9, 1:12, and 1:15). The temperature variations used were 30°C, 45°C, and 60°C with time variations of 90, 120, and 150 minutes. The variations of catalyst used are (1.2, and 3%).	CaO	The results of the biodiesel quality test were that the yield was 53% under conditions of a ratio of 1:15, 3% catalyst, reaction temperature of 60°C with a time of 120 minutes.	[12]
2.	Transesterification	The variation in the ratio of used cooking oil:methanol is 1:2. The time variations used are 90 and 120 minutes. The temperature used is 65°C.	NaOH and KOH	The best biodiesel yield of all variations was shown at a time variation of 120 minutes using a KOH catalyst. The test results obtained a yield value of 76.7%, a density of 0.8669 gr/ml, and a viscosity of 5.15 Cst.	[13]
3.	Transesterification	Temperature variations (50°C, 55°C, 60°C, 65°C, and 70°C. Time variations used were 30, 60, 90, and 120 minutes. Transesterification reaction with a used cooking oil:methanol ratio of 1: 5.	KOH	The results obtained were that the amount of biodiesel yield produced in 30 minutes reached 80% and 60 minutes reached 90%. The most optimal temperature is between 60°C to 65°C. The FFA level obtained was 9.67%.	[14]
4.	Transesterification	The ratio of waste cooking oil:methanol is 1:6. The transe-terification process was carried out at varying temperatures of 60°C, 65°C, and 70°C and varying catalysts of 1%, 2%, and 3% for 60 minutes.	CaO- NaOH	The results obtained were the highest yield of 85%, density of 0.857 gr/ml, viscosity of 0.65 Cst at a temperature of 65°C and 1% catalyst.	[15]
5.	Transesterification	The temperature used was 65°C for 60 minutes. The ratio of used cooking oil :methanol is 1:6.	NaOH	The results obtained were biodiesel with a yield of 19%, an acid number of 0.4 and a viscosity of 3.35 Cst	[16]

The main aim of this research is to reduce used cooking oil waste and then reuse it into alternative fuels namely biodiesel [17]. Analysis needs to be carried out to find out the reaction results at a certain temperature and time that are most optimal for biodiesel and apply the BBD (Box-Behnken Design) approach, where the Box-Behnken Design (BBD) is one of the designs that can be accessed in RSM which has been widely used to increase biodiesel production. This is because BBD can be more efficient in parameter tuning, and requires fewer trials than other designs [18].

2. Methods

2.1 Tools and Materials

2.1.1 Tools

The equipment used in this research is a set of transesterification tools consisting of a three-neck flask, heating mantle, magnetic stirrer, thermometer, water bath, reflux condenser, stand and clamp, separating funnel, Erlenmeyer, beaker glass, and measuring cup.

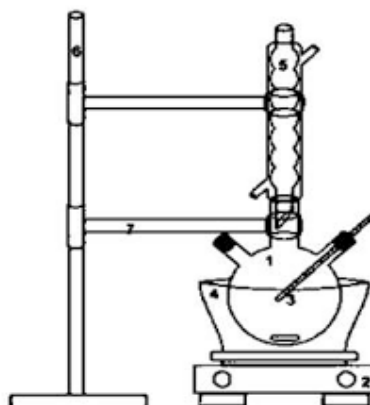


Figure 1. Series of Transesterification Equipment in a Batch Reactor

Source: Mirzayanti et al., 2022 [19]

2.1.2 Materials

The materials used in this research include Aquadest, PP Indicator, NaOH, NaOH 0.025N, Methanol 98%, and Waste Cooking Oil.

2.2 Methodology

2.2.1 Biodiesel Manufacturing Process

2.2.1.1 Filtering Wasted Cooking Oil

The used cooking oil is filtered using a vacuum pump and filter paper. The purpose of this filtering is to remove impurities remaining during frying.

2.2.1.2 Waste Cooking Oil Testing

After the used cooking oil has been filtered, the free fatty acid content of the used cooking oil is tested, namely by calculating the density, viscosity, and also FFA (Free Fatty Acid).

2.2.1.3 Reflux Process

Then 40 ml of used cooking oil was weighed, then put into a three-neck flask. Methanol was added with a volume ratio of 1:4, 1:5, 1:6 (used cooking oil: methanol), and 1% NaOH catalyst was added. Next, all the mixtures were refluxed using temperatures of 55°C, 60°C, and 65°C with stirring using a magnetic stirrer for 60, 90, and 120 minutes.

2.2.1.4 Solution Separation

The resulting mixture from the transesterification method is then cooled, then deposited for 1-2 hours in a separating funnel, until several phase layers are formed. The results obtained are 2 layers, namely pure biodiesel solution at the top and glycerol at the bottom.

2.2.1.5 Washing Solution

The top layer of biodiesel is taken, then the washing process is carried out with hot distilled water, namely at a temperature of 80°C. This washing aims to remove residual glycerol and soap from the transesterification reaction. Washing is carried out with a ratio of distilled water to the oil phase, namely 1:1. Then it was deposited for 3 days, the biodiesel obtained was taken and then FFA testing was carried out and the yield, density, and viscosity were calculated.

2.3 FFA

FFA testing is carried out using the titration method to determine the quality and usability of the oil. The titration is carried out using a 0.025 N NaOH solution which functions to measure several free fatty acids from the oil to be titrated as well as determining the levels of several acidic compounds using an alkaline solution [20].

$$FFA = \frac{V \text{ NaOH} \times N \text{ NaOH} \times BM \text{ fatty acid}}{\text{sample weight} \times 1000} \times 100\% \quad (1)$$

Source: Hadrah et.al., 2018 [5].

2.4 Yield

Yield is used to determine the percentage of results obtained from a process. Yield is a comparison between the weight of biodiesel and the initial weight of oil. Yield can be influenced by many factors, namely temperature, settling time, and stirring [18]. The yield in the solution can be found using Equation 2. The following:

$$\text{yield} = \frac{\text{biodiesel weight}}{\text{waste cooking oil weight}} \times 100\% \quad (2)$$

Source: Prihanto and Irawan, 2017 [21].

3. Result and Discussion

3.1 Optimization

This research was designed using *Response Surface Methodology* (RSM) with Design Expert 13 Software. Based on research carried out using used cooking oil, the experimental data is known as shown in Table 4. The following:

Table 4. Design RSM (*Response Surface Methodology*)

Std	Run	Factor 1 A: Volume Ratio (mL)	Factor 2 B: Time (min)	Factor 3 C: Temperature (°C)	Response Yield (%)
3	1	160	120	60	51.0639
4	2	240	120	60	38.3784
12	3	200	120	65	41.9037
1	4	160	60	60	61.293
17	5	200	90	60	47.3298
7	6	160	90	65	45.6449
5	7	160	90	55	61.9404
8	8	240	90	65	43.9593
13	9	200	90	60	49.4191
15	10	200	90	60	42.9918
10	11	200	120	55	28.8011
9	12	200	60	55	58.6227
16	13	200	90	60	45.0757
11	14	200	60	65	26.7472
6	15	240	90	55	38.053
14	16	200	90	60	48.9123
2	17	240	60	60	47.1447

This model will optimize according to the variable data and response measurement data entered. Optimization is carried out by determining the desired response criteria (goal) with a range that is possible to achieve. The most optimal formula is the formula with the maximum desirability value. The desirability value is a function value for optimization purposes that shows the program's ability to fulfill desires based on the criteria set in the final product. A desirability value that is getting closer to 1.0 indicates the program's ability to produce the desired product is increasingly perfect [22].

Each response from the experimental results is then subjected to an ANOVA (Analysis of Variance) test to determine the significance of the response analysis between variables and to find out the model suggested by the Design Expert 13 Software. Table 5 shows the ANOVA (Analysis of Variance) of the results obtained with the Design Expert 13. The model can be declared to have a significant influence if it has a P value < 0.05. However, if the probability value ($P > F$) is greater than 0.1 then the model shown is not significant.

Table 5. ANOVA (*Analysis of Variance*) of Yield

Source	Sum of Squares	Df	Mean Square	F-value	P-value	
Model	1473.19	9	163.69	28.37	0.0001	Significant
A	343.31	1	343.31	59.50	0.0001	
B	141.63	1	141.63	24.55	0.0016	
C	106.30	1	106.30	18.42	0.0036	
AB	0.5349	1	0.5349	0.0927	0.7696	
AC	123.23	1	123.23	21.36	0.0024	
BC	505.76	1	505.76	87.65	< 0.0001	
A ²	129.81	1	129.81	22.50	0.0021	
B ²	33.68	1	33.68	5.84	0.0464	
C ²	101.05	1	101.05	17.51	0.0041	
Residual	40.39	7	5.77			
Lack of Fit	11.33	3	3.78	0.5197	0.6911	not significant
Pure Error	29.06	4	7.27			
Cor Total	1513.58	16				

The ANOVA results selected are those with the largest R² value. The largest R² shows that the model is recommended and the variable component values have a real (significant) effect on the conversion response [23]. The following is Table 6 of the ANOVA results on Fit Statistics:

Table 6. ANOVA (*Analysis of Variance*) Fit Statistics

Std. Dev.	= 2.40	R²	= 0,9733
Mean	= 45,72	Adjusted R²	= 0,9390
C.V. %	= 5,25	Predicted R²	= 0,8502
		Adeq Precision	= 19,5045

The accuracy of the model can be seen in Figure 2. And the R-squared (R^2) obtained is 0.9733. In the sum of the square test, a model is declared appropriate if the Adjusted R^2 and Predicted R^2 values have a difference in values smaller than 0.2. If the research results are obtained, the value of Adjusted R^2 is 0.9390 and the value of Predicted R^2 is 0.8502, which shows that this model is significant.

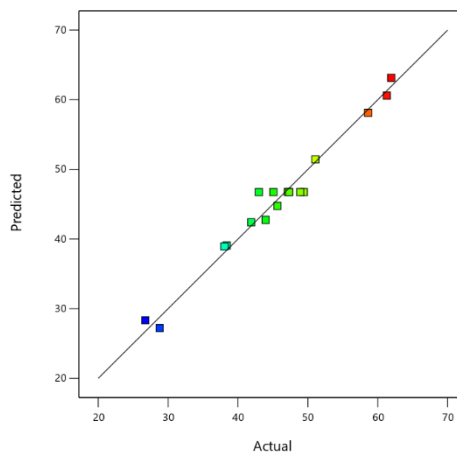
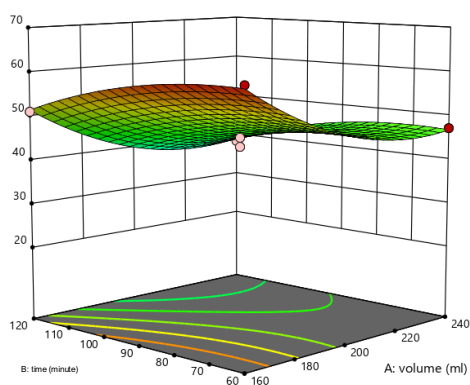
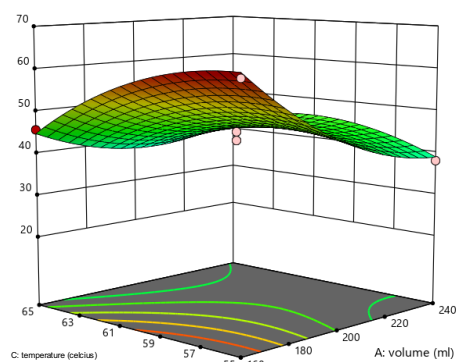
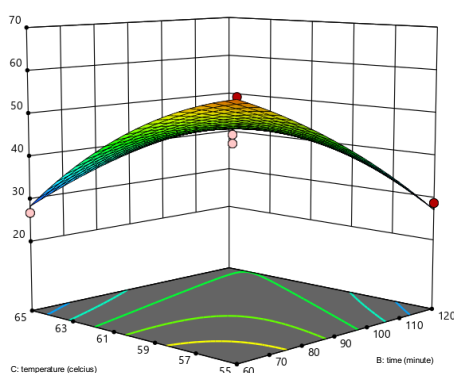


Fig 2. Predicted to Actual

3.2 Yield

The use of high methanol will cause the reaction to shift to the right and will produce maximum conversion [24]. The higher the methanol volume ratio used in the transesterification process, the resulting yield will decrease. This is because using a lot of methanol will form a layer of methanol on the top of the oil because not all methanol can react with the triglycerides contained in used cooking oil [25]. Figure 3 shows that a small reactant ratio (methanol) will produce a greater yield. The optimal used cooking oil: methanol ratio is based on Figure 3, namely at a ratio of 1:4 with 60 minutes the yield obtained is 61.9404%. The decrease in yield was very drastic, up to 26.7472%, which could be caused by the high volume of methanol during the transesterification process.

**Fig 3.** The Effect of Yield on Time and Ratio**Fig 4.** The Effect of Yield on Temperature and Ratio**Fig 5.** The Effect of Yield of Temperature and Temperature

In the transesterification process, changing temperatures can cause the molecules to move faster or the energy possessed by the molecules can overcome their activation energy [27]. Each temperature used in the transesterification process will produce a different yield. Based on Figure 4. The highest yield was obtained at a temperature of 55°C. Low yield results can be caused by several things, such as the temperature used during the mixing process. The temperature used during the process must not exceed the boiling point of methanol, because it will cause the methanol to evaporate and reduce. When the optimal heating temperature is reached, the yield obtained will increase. When the process temperature is 55°C, the raw material from used cooking oil receives perfect heat to react compared to other temperatures.

In Figure 5, it can be seen that the reaction time influences the biodiesel yield obtained. When equilibrium has been reached, the synthesis process should be stopped so that the energy is used more efficiently [26]. This can be proven that the largest yield is obtained at the lowest time, namely 60 minutes, however, if the reaction is continued for up to 90 minutes, 120 minutes the yield conversion in biodiesel decreases. The effect of time on the yield can be said to be a reversible reaction because the equilibrium reaction leads to the formation of fatty acid esters and glycerol. Triglycerides are converted to diglycerides, monoglycerides, and finally to glycerol.

3.3 FFA

Determining the quality of biodiesel can be done by knowing the level of free fatty acids in the biodiesel. The FFA levels in biodiesel must be low because if the FFA levels are high it will reduce the yield and viscosity of the biodiesel [28]. The size of the FFA content is influenced by several factors such as the ratio of used cooking oil: methanol, temperature, and time during the process. The results obtained were FFA values with the highest level of 0.0448 in running 12, with a ratio of 1:5, time 60 minutes, and temperature 55°C. The lowest level of FFA produced was 0.01536 in running 7, with a ratio of 1:4, time of 90 minutes, and temperature of 55°C. The size of the FFA is also inversely proportional to the yield obtained, if the resulting yield value is high then the resulting FFA value will be low and have a greater yield. It is proven in Table 7 that running 12 with the highest FFA content has a smaller yield compared to running 7 which has a low FFA content. Biodiesel which has low FFA content is a good biodiesel to use as fuel because it is non-corrosive and does not cause scale in diesel engines [29].

Based on research by Irepia Refa Dona [30], it is said that good quality biodiesel that is suitable for use is biodiesel that has low FFA levels. The longer the transesterification time will provide greater opportunities for compound molecules to react which can reduce FFA levels [31]. The decrease in FFA occurs as the process time increases, which means that the longer the time required for the transesterification process, the smaller the FFA produced. In the experiments that have been carried out, it was proven that the lowest FFA levels were obtained at 90 minutes at a temperature of 55°C, with a result of 0.01536, and the highest FFA levels were obtained at 60 minutes at a temperature of 55°C, with a result of 0.0448.

Table 7. Biodiesel Test Result

Run	Volume Ratio (ml)	Time (min)	Temperature (°C)	Density (gr/cm ³)	Viscosity (cSt)	FFA
1	1:4	120	60	0.8164	2.67444	0.027495
2	1:6	120	60	0.8196	2.31142	0.03264
3	1:5	120	65	0.8145	2.60282	0.02048
4	1:4	60	60	0.82	2.60185	0.0224
5	1:5	90	60	0.81648	2.45219	0.02752
6	1:4	90	65	0.8178	2.63464	0.01664
7	1:4	90	55	0.8123	2.53517	0.01536
8	1:6	90	65	0.8115	2.4455	0.01728
9	1:5	90	60	0.8135	2.49476	0.0192
10	1:5	90	60	0.8133	2.42793	0.01664
11	1:5	120	55	0.8213	2.5744	0.03136
12	1:5	60	55	0.8188	2.75359	0.0448
13	1:5	90	60	0.8157	2.61588	0.0384
14	1:5	60	65	0.82102	2.56424	0.02688
15	1:6	90	55	0.8216	2.60321	0.04352
16	1:5	90	60	0.8167	2.57845	0.04032

The use of high temperatures in transesterification reactions can result in accelerated movement of compound molecules which will also increase collisions between reactant molecules. The relationship between reaction rate and temperature is directly proportional, the higher the reaction temperature, the higher the reaction rate [32]. In the experiments carried out, the temperature that produced low FFA levels was found to be 55°C, at this temperature the reaction process occurred perfectly and optimally.

3.4 Density and Viscosity

The size of the density is proportional to the FFA content and viscosity, if the FFA content and viscosity are high then the density value will also be high [33]. It was proven in the experiment that the highest FFA content was carried out in running 12, producing a higher density of 0.8188 gr/cm³ compared to running 7, which had the lowest FFA content producing a density of 0.8123 gr/cm³. In Table 7, it can be seen that the density of biodiesel produced in the experiment does not meet the biodiesel quality requirements based on SNI 7182-2015. This is due to the influence of the composition of the mixture used when making biodiesel, the higher the composition of the mixture of used cooking oil and methanol, the higher the density it will produce [34]. The used cooking oil used in the transesterification process has a density of 0.8465 with a viscosity of 32.5733 cSt and an FFA content of 0.5056.

Table 8. Comparison of characteristics of test results with SNI

Testing	Result	Unit	Standart SNI
Rate of Free Fatty Acid	0.01536-0.0448	-	Max 0.2
Density	0.8115-0.8241	gr/cm ³	0.85-0.89
Viscosity	2.31142-2.75359	cSt (mm ² /s)	2.3-6.0

Source: SNI 7182-2015

The longer the transesterification time and increasing temperature, the shorter the methyl ester chain, so the viscosity value will also decrease [35]. The data obtained shows that all samples meet the biodiesel viscosity quality standards. The highest viscosity is 2.75359 cSt, while the lowest viscosity value is 2.31142 cSt. The viscosity value of biodiesel in each sample decreased as the reaction time increased. It was proven in research that the lowest viscosity value was in running 2 with the highest time of 120 minutes. The viscosity value is also proportional to the FFA content, if the FFA content is high then the viscosity is also high. In the research conducted, it was proven that the highest viscosity value in running 12 was 2.75359 cSt with the highest FFA content, namely 0.0448, at the lowest time is 60 minutes.

4. Conclusions

It can be concluded that the Design Expert 13. Software is useful in formulation to make it easier to determine optimal results. The R-squared (R^2), Adjusted R^2 , and Predicted R^2 values meet the requirements of ANOVA (Analysis of Variance). The yield obtained depends on the mole ratio of the reactants mixed. Optimal yield results were obtained at a ratio of 1:4, with a time of 60 minutes, at a temperature of 55°C, a yield of 61.9404% was obtained. To determine the quality of the biodiesel obtained, it is also necessary to test FFA, density, and viscosity, to ensure the suitability of the biodiesel samples obtained.

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