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Optimization Synthesis of Biodiesel from Waste Cooking Oil in a Phased Array Ultrasonic Reactor Using Response Surface Methodology

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Abstract: *This research aims to optimize the process of biodiesel synthesis from used cooking oil using Response Surface Methodology (RSM) in an ultrasonic phased array reactor. As a potential waste source, used cooking oil has been identified as a sustainable alternative feedstock for biodiesel production. Using ultrasonic phased array reactors is expected to improve conversion efficiency through cavitation, producing high yields and purity of biodiesel. RSM was employed to obtain the optimal combination of transesterification reaction conditions, including feedstock ratio, catalyst concentration, and reaction time. Methanol and NaOH were transesterified in a reactor filled with used cooking oil. The results showed a biodiesel yield of 90.3250%, an optimum mole ratio of 1:7.59 (oil to methanol), a catalytic concentration of NaOH at 0.14% by oil weight, and a reaction time of 15 minutes. The oil/methanol ratio was identified as the most significant operating parameter based on the ANOVA test. Based on the physical properties of biodiesel, including density, kinematic viscosity, flash point, carbon residue, and GC-MS testing, the biodiesel produced met the SNI 7182:2015 standards.*
Keywords: *Biodiesel, Phased array ultrasonic, RSM, Transducer, Transesterification*

INTRODUCTION

The bioenergy potential in Indonesia has been estimated to reach 57 GW, yet only 2,284 MW have been harnessed as of 2021 (ESDM, 2021). The national energy policy aims for a renewable energy (EBT) mix of 23% by 2025, which is expected to increase to at least 31% by 2050. This potential for utilizing new and renewable energy sources is anticipated to grow further in alignment with the global Net Zero Emission 2060 initiative. Currently, the Indonesian government emphasizes using of biodiesel, specifically the B-30 blend, which combines 30% biodiesel with conventional diesel, as part of its renewable energy strategy.

Biodiesel is an environmentally friendly alternative to petroleum-based diesel fuel, offering several advantages. It is renewable, non-toxic, biodegradable, and contains minimal sulfur, contributing to favourable emission characteristics. Additionally, biodiesel can be used in existing engines without significant modifications (Singh et al., 2020). Biodiesel is typically produced through the transesterification of fats and oils, wherein triglycerides in oil or fat feedstocks react with alcohols to produce alkyl esters of fatty acids (biodiesel) and glycerol as a byproduct (Amenaghawon et al., 2022).

However, the primary limiting factor for large-scale biodiesel production is the high cost associated with its production process, with raw material costs accounting for up to 80% of the total production cost (Etim et al., 2020). Increasing attention is given to non-

edible oil sources, such as castor oil (Frazão do Nascimento et al., 2016; Navas et al., 2018) and used cooking oil (Ghavami et al., 2022). Used cooking oil, in particular, holds significant potential due to its relatively low commercial price. Data shows that biodiesel from used cooking oil contributed 2,765 kL from 2014 to 2018 (ESDM, 2021).

In addition to raw materials, selecting reactor types in biodiesel production is crucial for achieving optimal and efficient results. Biodiesel is produced in transesterification reactors, which come in various types, including batch reactors, stirred tank flow reactors, fixed bed reactors, bubble column reactors, microchannel reactors, membrane reactors, reactive distillation, and hybrid catalytic plasma reactors (Tabatabaei et al., 2019).

Ultrasonic technology has been employed in biodiesel synthesis to enhance the mixing of reactants and accelerate the reaction rate, thus producing high biodiesel yields in shorter reaction times (Tabatabaei et al., 2019). Researchers typically use Ultrasonic Baths and Ultrasonic Horns for this purpose. The ultrasonic cavitation effect can increase the speed of the transesterification reaction by up to 100-fold, resulting in higher biodiesel conversion in a shorter time (Oliveira et al., 2018).

This research produced biodiesel from used cooking oil using a Phased Array Ultrasonic reactor. As far as the literature search indicates, this is the first previous studies have employed this reactor for biodiesel production. The biodiesel production process was analyzed and optimized using Response Surface Methodology (RSM) software from StatEase. Response Surface Methodology (RSM) is a collection of statistical and mathematical techniques proper for developing, improving, and optimizing processes where responses are influenced by several independent variables (Montgomery, 2017). The main goal of this method is to determine the effect of independent variables on the response, model the relationship between these variables and the response, and identify the process conditions that yield the best response. The optimization target is to minimize the effort required while maximizing the desired outcomes (Princess, 2018).

Box-Behnken Design is frequently used in the literature to optimize various processes (Veljković et al., 2019). Accordingly, this study is designed to investigate biodiesel production using Phased Array Ultrasonic reactors and identify the optimal biodiesel production conditions utilizing Response Surface Methodology (RSM). The study aims to determine the influence of critical factors such as the oil/alcohol mole ratio, catalyst concentration, and reaction time on biodiesel yield. This research seeks to identify the most critical operating parameters for efficient and high-quality biodiesel production using Phased Array Ultrasonic reactors. Furthermore, it aims to optimize the operating conditions of the Phased Array Ultrasonic reactor in producing biodiesel from used cooking oil.

RESEARCH METHODS

Materials and Tools

The materials used in this research include cooking oil, NaOH catalyst, distilled water (aquades), and methanol. The equipment utilized in the study comprises an ultrasonic phased array reactor with a transducer frequency of 28 kHz and a power of 100 Watts, along with glassware (beaker, measuring cup, watch glass, and separatory funnel), gloves, digital scales, hot plates, and stirring rods.

Procedures

Optimization of Biodiesel Synthesis in Phased Array Ultrasonic Reactors

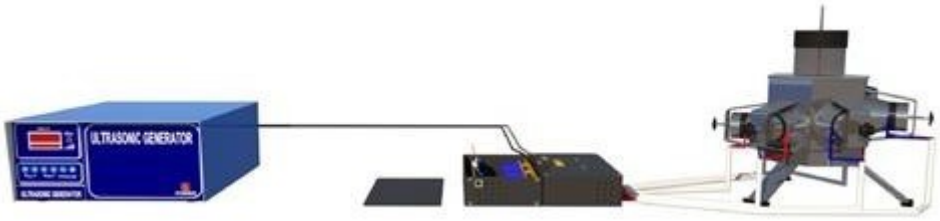

Figure 1. Phased Array Ultrasonic Reactor

The biodiesel from used cooking oil was synthesised in an ultrasonic phased array reactor with base catalyst (NaOH) and methanol. The glass reactor is maintained at room temperature. The molar ratio of alcohols to oils varies between 6:1, 8:1, and 10:1. The concentration of catalysts varies in the range of 0.1%, 0.2%, 0.3% (weight of oil). The reaction time varies between 5, 10 and 15 minutes.

Methanol and NaOH are stirred for approximately 15 minutes to ensure homogeneity. This solution is then added to a glass reactor containing used cooking oil. Upon reaching the desired reaction time, the mixture of biodiesel and glycerol can stand for 24 hours in a separatory funnel. The separation process is then carried out to obtain biodiesel in the lower layer and methanol and glycerol in the upper layer. The obtained biodiesel is subsequently washed using distilled water (aquades) heated to 80°C. The washing process aims to remove unreacted alcohol, catalysts, and any soap remaining in the biodiesel after the reaction. The ratio of aquades to biodiesel used for washing is 1:1. Washing is continued until the pH of the wash water is between 6.8 and 7.2. After washing, the biodiesel is allowed to stand for 24 hours to separate the aquades from the biodiesel. The final stage of the process involves heating the biodiesel to remove any residual water. This is done using an electric heater at 100°C for 20 minutes.

Experiment Design

Response Surface Methodology

Box-Behnken design was chosen for regression and graphical analysis of experimental data in this research. Box-Behnken is a three-level experimental design for RSM in which each parameter is studied at three levels with equidistant intervals (Avinash & Murugesan, 2018). Design Expert software (version 6.0.5) is used to design experiments and perform regression. The code values and actual values of the variables used in the BBD design are given in Table 1.

Table 1. Level and Level Value

Variable Level			Variable
1	0	-1	
1:10	1:8	1:6	Mole oil/methanol ratio (mol/mol)
0,3	0,2	0,1	Catalyst concentration NaOH (weight %)
15	10	5	Reaction time (minutes)

Experimental data from BBD are analyzed to obtain optimal process conditions as in equation (1).

$$Y = \beta0 + \beta1X1 + \beta2X2 + \beta3X3 + \beta12X1X2 + \beta13X1X3 + \beta23X2X3 + \beta11X12 + \beta22X22 + \beta33X32 \quad (1)$$

Information:

Y = response variable you want to optimize
β0 = constant
β1, β2, β3, β12, β13, β23, β11, β22, β33 = regression coefficient
X1, X2, X3 = input variables selected to optimize
X12, X13, X23 = interaction between input variables
X1^2, X2^2, X3^2 = square effect of input variables
While the method to obtain the response variable (biodiesel yield) as in equation (2).

Biodiesel Yield (%) = $\frac{massa\ biodiesel\ (g)}{massa\ minyak\ (g)}\ x\ 100\%$ (2)

The mass of biodiesel produced is the amount of biodiesel successfully produced from the amount of oil used, while the mass of oil is the amount of oil used in the biodiesel production process.

Table 2. BBD matrix of three independent variables along with experimental responses

Run	A	B	C	Yield (%)
	Mole ratio (mol/mol)	%Catalyst (w/w)	Reaction time (min)	
1	1:8	0,2	10	90.36
2	1:10	0,1	10	80.53
3	1:6	0,1	10	89.51
4	1:8	0,2	10	87.41
5	1:10	0,2	5	80.21
6	1:8	0,1	5	83.94
7	1:6	0,3	10	79.66
8	1:8	0,1	15	86.95
9	1:6	0,2	5	81.98
10	1:8	0,3	5	88.53
11	1:6	0,2	15	89.59
12	1:10	0,2	15	90.02
13	1:8	0,2	10	90.81
14	1:8	0,3	15	88.97
15	1:8	0,2	10	83.95
16	1:8	0,2	10	88.81
17	1:10	0,3	10	75.79

RESULTS AND DISCUSSION

Response Model Analysis

Model Selection Test Based on Sequential Model Sum of Squares

Table 3. Analysis *Sequential Model Sum of Squares*

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F	
Mean	1.249E+005	1	1.249E+005			Suggested
Linear	87.57	3	29.19	1.50	0.2609	
2FI	9.39	3	3.13	0.13	0.9410	
Quadratic	115.94	3	38.65	2.12	0.1861	Suggested
Cubic	97.14	3	32.38	4.24	0.0982	Aliased
Residuals	30.51	4	7.63			
Total	1.252E+005	17	7365.73			

Based on Table 3, the model that explains the effect of variations in research variables (oil/methanol mole ratio, catalyst concentration, and reaction time) on the biodiesel yield response is the mean and quadratic models. The best model from the model selection test, based on the sum of squares of the model sequence, is the one that has a probability value of less than 5% (P < 5%). However, for the quadratic model, the P value is 0.1861, which indicates that the model has a marginally significant effect on explaining the intended response (Gantini & Widayanti, 2018). Despite this, the quadratic model is recommended.

Model Selection Test Based on Lack of Fit Tests

Table 4. Analysis *Lack of Fit Tests*

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F	
Linear	222.47	9	24.72	3.24	0.1348	
2FI	213.08	6	35.51	4.66	0.0792	
Quadratic	97.14	3	32.38	4.24	0.0982	Suggested
Cubic	0.000	0				Aliased
Pure Error	30.51	4	7.63			

The lack of fit tests aims to compare residual errors with "pure errors" from replicated design points. The desired model is "insignificant" or Suggested with a probability value of more than 5% (P>5%). Based on Table 4, the model suggested by Design Expert software is quadratic because it has the most insignificant value.

Model Selection Test Based on Model Summary Statistics

Table 5. Model Analysis *Summary Statistics*

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	4.41	0.2572	0.0857	-0.3757	468.51	
2FI	4.94	0.2847	-0.1444	-1.9297	997.74	
Quadratic	4.27	0.6252	0.1433	-3.7036	1601.84	Suggested
Cubic	2.76	0.9104	0.6416			Aliased

This test is based on the R-Squared Adjusted and R-squared predicted values, where a good value is close to 1. Based on the table above, the recommended model is the quadratic model, although the R-squared adjusted value of the cubic model is closer to 1.

ANOVA Test

Table 6. ANOVA Test

Source		Sum of Squares	DF	Mean Square	F Value	Prob>F	
Type		159.26	3	53.09	3.81	0.0370	Siginificant
	A	25.17	1	25.17	1.80	0.2021	
	C	54.44	1	54.44	3.90	0.0698	
	A2	79.65	1	79.65	5.71	0.0327	
Residuals		181.29	13	13.95			
	Lack of Fit	150.78	9	16.75	2.20	0.2332	Not Significant
	Pure Error	30.51	4	7.63			
Total Cast		340.56	16				

Table 6 shows that variable B is the oil/methanol mole ratio, and variable C is the reaction time. For a factor to be considered significant, the value of Prob>F must be less than 5% (Arifin et al., 2022). The ANOVA test results indicate that one of the three factors is significant based on the ANOVA test criteria. The interaction involving the oil/methanol mole ratio is a significant factor, indicating that this variable significantly influences biodiesel yield.

An F model value of 3.81 suggests that the quadratic model is significant, with only a 3.7% chance that this significance could be due to noise. Additionally, Table 6 shows that the lack of fit value is insignificant compared to the possibility of pure error, with a 23.32% chance that the lack of fit is caused by noise. An insignificant lack of fit value is desired in a model, indicating that the model is appropriately capturing the relationship between the variables and the response.

Table 7. The Value of Design Accuracy

Std. Dev.	3.73	R-Squared	0.4677
Mean	85.71	Adj R-Squared	0.3448
C.V.	4.36	Pred R-Squared	0.0185
PRESS	334.26	Adeq Precision	6.253

Based on the ANOVA test in Table 7, the R^2 coefficient of determination is 0.4677, indicating that the independent variables explain 46.77% of the variability in biodiesel yield response. The quadratic model cannot explain 53.23% of the variability. The Adjusted R-Squared value is 0.3448, and the Predicted R-Squared value is 0.0185. The significant difference between these values (exceeding 0.2) suggests that the experimental data for biodiesel yield is not statistically consistent, and the chosen quadratic model is not entirely suitable for modelling this process.

Adequate Precision measures the signal-to-noise ratio with a desired value greater than 4. A ratio of 6.253 indicates an adequate signal, suggesting that the model can distinguish between the signal and noise. The standard deviation indicates data variability. If the standard deviation exceeds the mean value, the mean is a poor representation of the data. Based on Table 7, the standard deviation is smaller than the mean value, suggesting that the mean is a good representation of the overall data and illustrating the model's accuracy.

Table 8. Biodiesel Yield Response Model

Factor	Coefficient Estimate	DF	Standard Error	95% Clen Low	95% Clen High	VIF
Intercept	87.75	1	1.24	85.06	90.44	
A-Oil Mole Ratio	-1.77	1	1.32	-4.63	1.08	1.00
C-Time	2.61	1	1.32	-0.24	5.46	1.00
A2	-4.34	1	1.81	-8.26	-0.42	1.00

Based on Table 8, the model equation for biodiesel yield response is as follows:
Biodiesel yield = 87.75 – 1.77A + 2.61C – 4.34A2
Information:
A = Mole Oil/Methanol Ratio
C = Reaction Time

Based on the equation above, it is shown that the yield response of biodiesel increases directly proportional to the reaction time. The positive sign of the reaction time variable constant characterizes this. Conversely, the yield response of biodiesel decreases as the oil/methanol mole ratio increases and as the interaction between the oil/methanol mole ratio increases, which is characterized by a negative constant value. The condition to be achieved is maximum biodiesel yield, indicating that reaction time significantly influences increasing biodiesel yield.

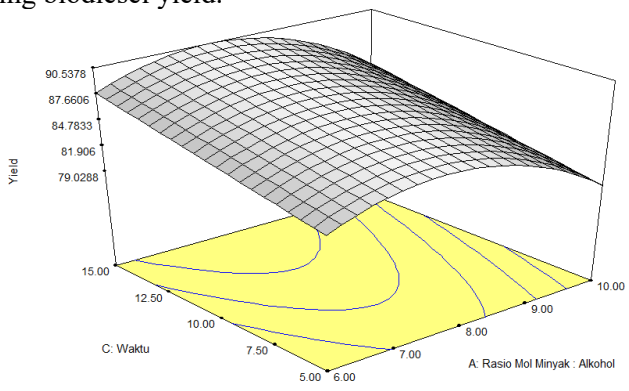

Figure 2. 3D Graph of Process Variable Interaction

Actual Biodiesel Yield Versus Prediction

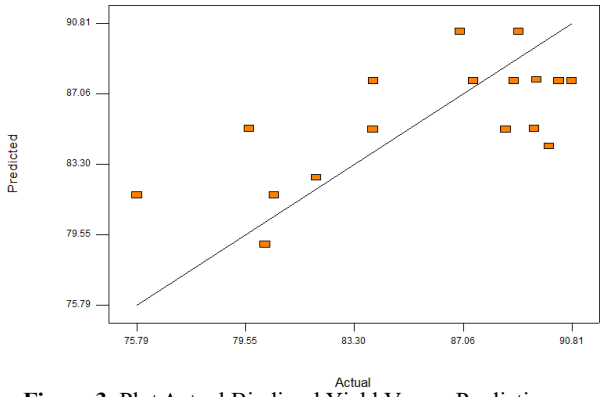

Figure 3. Plot Actual Biodiesel Yield Versus Prediction

The experimental data were analyzed to examine the correlation between actual and predicted biodiesel yields, with the plot of these yields shown in Figure 3. The actual biodiesel yield values are partially close to the predicted biodiesel yield value line, with a coefficient of determination (R^2) value of 0.4677. From this R^2 value, the correlation coefficient (R) can be calculated, indicating the strength of the relationship. An R-value of 0.6839 is obtained. Iqbal (2013) states that this value is considered entirely meaningful or moderate. Therefore, the conclusion is that there is a reasonably good relationship between the predicted and actual response values, indicating that the chosen quadratic model is reasonably adequate in predicting the yield response of biodiesel.

Model Verification

After various analyzes, the Design Expert software will provide optimization suggestions from the given design, which are as follows:

Table 9. Optimizations performed by RSM

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Oil Mole Ratio	Is in range	6	10	1	1	4
Catalyst Concentration	Is in range	0.1	0.3	1	1	4
Time	Is in range	5	15	1	1	4
Yield	Maximize	80	100	1	1	5

Solutions

Number	Oil Mole Ratio	Catalyst Concentration	Time	Yield	Desirability	
1	7.59	0.14	15.00	90.5379	0.527	Selected
2	7.59	0.12	15.00	90.5379	0.527	
3	7.59	0.26	15.00	90.5379	0.527	
4	7.59	0.12	15.00	90.5379	0.527	
5	7.59	0.24	15.00	90.5379	0.527	
6	7.59	0.19	15.00	90.5379	0.527	
7	7.59	0.11	15.00	90.5379	0.527	
8	7.59	0.16	15.00	90.5379	0.527	
9	7.59	0.20	15.00	90.5379	0.527	
10	7.67	0.10	15.00	90.5316	0.527	

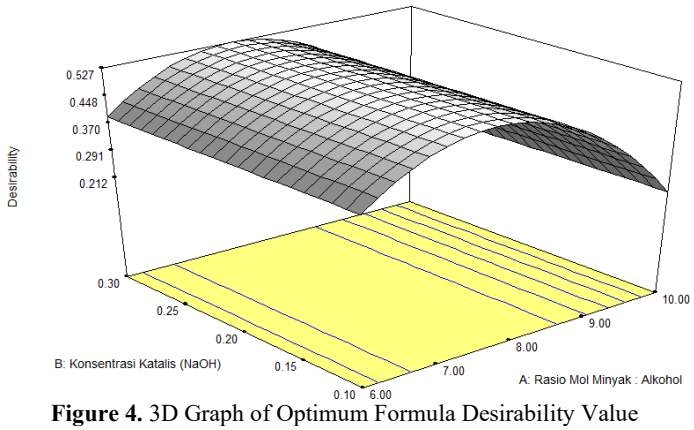

Figure 4. 3D Graph of Optimum Formula Desirability Value

There are ten optimization suggestions provided. The optimization validation selected has the highest desirability value (close to 1), specifically at an oil/alcohol mole ratio of 1:7.59 and a NaOH catalyst weight of 0.14%. After verification by calculating biodiesel yield according to the variations suggested by RSM, the biodiesel yield did not differ significantly. Verification was conducted twice, yielding an average biodiesel yield of 90.3250%, resulting in a percentage error of 0.2357%.

Cetane Number Analysis of Biodiesel

The results of the cetane number analysis revealed that the produced biodiesel has a cetane number of 44.2. According to SNI 7185:2015 standards, the minimum cetane number required is 51. A low cetane number indicates that the biodiesel can ignite at higher temperatures (Andalia et al., 2018). Therefore, the cetane number that produces biodiesel does not meet established standards.

Analysis of Physical Properties of Biodiesel

Table 10. Analysis of Physical Properties of Biodiesel

Parameters	Biodiesel produced	SNI 7182:2015
Density (kg/m3)	870.2	850 – 890
Viscosity (cSt)	5.5	2.3 – 6.0
Flash point (°C)	244	Min. 100
Carbon residue in 10% distillation pulp (%-mass)	0.26	Max. 0.3

Density is the ratio between weight and volume at a given temperature. Low density in biodiesel is caused by the increasing intensity of fracturing in the fat group, resulting in a lighter fraction of the carbon chain (biodiesel) and a heavier fraction of glycerol during the transesterification reaction. The purification stage influences high and low density, as inadequate purification can result in low biodiesel density. Density impacts the quality of biodiesel: a higher density value correlates with a higher calorific value. Biodiesel with a density exceeding the specified limits can result in incomplete combustion, leading to increased emissions and engine wear (Hartono et al., 2023).

Viscosity is one of the most critical parameters in determining the feasibility of using biodiesel. Low viscosity can cause leakage in the fuel injection pump, while excessively high viscosity can affect the performance of the fuel injection system and complicate fuel atomization. The viscosity of biodiesel is generally higher than that of conventional diesel fuel due to the longer carbon chains in the fatty acids that compose biodiesel. Diesel typically has a maximum carbon chain length of 16, whereas biodiesel from used cooking oil can have carbon chains up to 21, resulting in higher viscosity (Afandi, 2023).

The flash point is the lowest temperature at which fuel oil can ignite when its surface is near a flame. It determines the temperature at which oil will start to ignite (burn) when mixed with air, which is crucial for fuel storage and handling safety. The flash point should be sufficiently high to avoid fire hazards at average ambient room temperature (Hartono et al., 2023).

Carbon residue in biodiesel is the amount of residual carbon that accumulates after combustion under certain conditions. It indicates biodiesel quality, as a high carbon residue content can clog the combustion chamber and the ejector tip (Thangaraj et al., 2019). Based on the results of the analysis of the physical properties of biodiesel, the four physical quality parameters of biodiesel have met the SNI 7182:2015 standards.

Biodiesel Hydrocarbon Composition Analysis

This analysis is a qualitative and quantitative assessment used to determine the type and quantity of fatty acids in biodiesel (Azhari et al., 2023). The Gas Chromatography (GC) method provides detailed information on the fatty acid derivatives in the sample. The Mass Spectroscopy (MS) method is used to determine the fragmentation patterns of saturated and unsaturated fatty acids and the location of double bonds in the fatty acid molecules (Ningtyas et al., 2013).

The principle of "like dissolves like" is fundamental in this analysis, where esters with longer chains have longer retention times. Short-chain esters are more polar than long-chain esters (Greever, 1995). The GC detector first reads compounds with short hydrocarbon chains because they are more easily carried by the mobile phase (Mukminin et al., 2022). This is due to the weaker interaction of short carbon chains with the column than long chains. As a result, polar short chains elute earlier than non-polar long chains. Fatty acids have a single carboxylic group and a non-polar hydrocarbon chain and are insoluble in water (Greever, 1995).

Biodiesel methyl esters that have been analyzed with GC-MS show four dominant peaks as presented in figure 5.

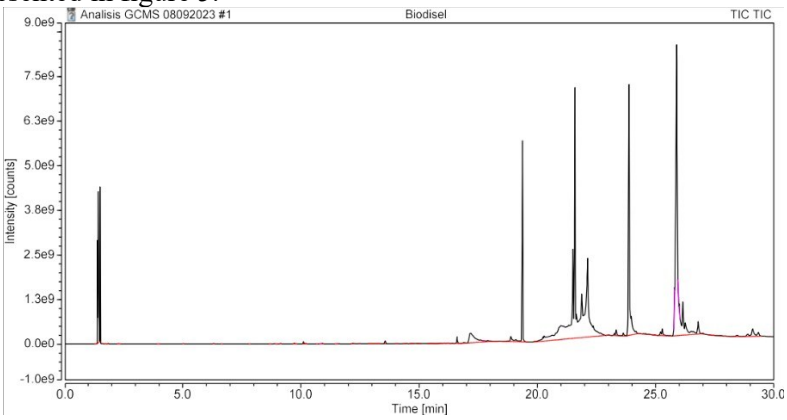

Figure 5. GC-MS Analysis Results

Figure 5 is a chromatogram of methyl ester composition analysis using GC-MS. The chromatogram shows that the biodiesel produced contains methyl esters corresponding to the fatty acids in used cooking oil. Based on the results of GC- MS, there are four methyl ester peaks. The first peak is the saturated fatty acid methyl palmitate ($C_{16}H_{32}O_2$) by 6.52%. The second peak is saturated fatty acids, namely methyl stearate ($C_{18}H_{36}O_2$) of 8.72%. The third peak is saturated fatty acids, namely methyl myristate ($C_{14}H_{28}O_2$) of 10.88%. In the last peak, the fourth peak, there is a monounsaturated fatty acid, methyl oleic ($C_{18}H_{34}O_2$) of 13.46%.

Based on the chromatogram area, methyl oleate has a larger area than the other three methyl esters. According to Gultom (2001) and Ma & Hanna (1999), palmitic acid is the most significant saturated fatty acid, while oleic acid, containing one double bond, is the most essential unsaturated fatty acid. Biofuels with high saturated fatty acid content are methyl esters resistant to oxidation from air and have a high octane number (Gultom, 2001). The GC-MS test analysis results indicate that the overall methyl ester content in the biodiesel is 96.98%, exceeding the minimum requirement of 96.5% as specified by SNI 7182:2015.

CONCLUSIONS

Based on the ANOVA test, the oil/methanol mole ratio was the most critical factor in this study because it significantly influences biodiesel yield. The greater the ratio of moles of oil to methanol, the yield of biodiesel will decrease. At the same time, the yield response of biodiesel will be directly proportional to the reaction time. The longer the reaction time, the more biodiesel yield increases. The optimization formula suggested by RSM is a mole oil/alcohol ratio of 1: 7.59 and a catalyst weight of 0.14% NaOH. The results of testing the physical properties of biodiesel on density, kinematic viscosity, flash point and carbon residue in 10% distillation pulp were 870.2 kg/m3, 5.5 cSt, 244°C, and 0.26%, respectively. Meanwhile, based on the results of GC-MS testing, the overall methyl ester content in biodiesel is 96.98%. The biodiesel produced meets SNI 7182:2015 biodiesel standards.

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