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Optimization of Biodiesel Synthesis from Waste Cooking Oil By Transesterification Process in A Microwave

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A B S T R A C T

The rising energy demand in Indonesia is driving the development of biodiesel as a sustainable, renewable fuel. Waste cooking oil has the potential as a biodiesel feedstock due to its abundant availability and relatively low cost. Biodiesel is generally produced via transesterification, but conventional heating methods have limitations, such as long reaction times and high energy consumption. Therefore, microwave-based heating is used as an alternative to improve process efficiency through rapid, even heating. This research aims to optimize biodiesel synthesis from used cooking oil using a microwave-assisted transesterification process with the Response Surface Methodology (RSM) and Analysis of Variance (ANOVA) approaches. The process variables studied include catalyst concentration (1 , 2, 3, 4, and 5%w/w) and reaction time (2, 4, 6, 8, and 10 minutes), with biodiesel yield as the main response. A mathematical model was developed using an appropriate experimental design and statistically validated using ANOVA to assess the model's significance and the effects of interactions between variables. The research results showed the highest biodiesel yield at 4% catalyst with a reaction time of 6 minutes. The RSM method, validated by ANOVA, was effective in determining optimum operating conditions and increasing biodiesel yield. This approach has the potential to be applied as a highly effective and sustainable method for biodiesel production from used cooking oil.

Contribution to Sustainable Development Goals (SDGs):
SDG 7: Affordable and Clean Energy
SDG 12: Responsible Consumption and Production
SDG 13: Climate Action
SDG 15: Life on Land

1. INTRODUCTION

The total energy used in Indonesia is still 42% sourced from fuel oil. According to data from the Ministry of Energy and Mineral Resources (EMR), in 2021 diesel consumption reached 1.59 million kiloliters, an increase of 80.86% from 2020, when it reached77,000 kiloliters [1]. So that fossil fuel reserves are depleting, and replacement alternative fuels are needed. Biodiesel is a candidate for renewable fuels, as it can be produced from vegetable oil or renewable used cooking oil [2].

Used cooking oil has a big potential as a raw material for biodiesel. Used cooking oil waste in Indonesia is estimated at 1.64 billion litres per year, according to a study by the *International Council on Clean Transportation* (ICCT). Used cooking oil waste comes from various sources, such as household waste, restaurants, hotels,nd others [3]. However, the FFA content of used cooking oil must first be lowered below 1% [2]. Reducing levels can be achieved using the two-stage process, namely Esterification and Transesterification [4]. The esterification process reduces the content of free fatty acids by converting them into methyl esters [5].

Meanwhile, transesterification converts triglycerides into methyl esters (Biodiesel) [6]. Transesterification requires a catalyst; a heterogeneous catalyst is generally used because it is easy to separate, cheaper, environmentally friendly,nd reusable [2]. Heterogeneous catalysts that have good catalyst activity, one of which is CaO. To be more reactive, CaO can be modified to become superalkaline, enabling higher biodiesel conversion.[7]. Transesterification heating can be done using alternative heating, such as microwaves. Microwave heating has many advantages over conventional heating because it heats up faster, and control is easy [8]. It is hoped that the biodiesel produced will have a high yield. However, the success of biodiesel synthesis is significantly influenced by various process parameters, including the alcohol-to-oil mole ratio, catalyst concentration, reaction time, and microwave power. Variations in these parameters can significantly affect biodiesel yield, necessitating a practical, systematic optimisation method. The conventional approach of changing one variable at a time *(one-factor-at-a-time)* is considered inefficient because it fails to capture the interactions between process variables. Transesterification is the key chemical reaction in biodiesel production, in which triglycerides react with short-chain (C1/C2) alcohols in the presence of a heterogeneous catalyst. The transesterification reaction is carried out at a temperature around the boiling point of methanol (60-70°C) at atmospheric pressure [9]. A microwave is an appliance that uses microwave energy to heat. It includes selective heating, diffusion radiation, emission reduction and *clean energy* [10]. Reaction time and the percentage of catalyst play an important role in the transesterification process. A longer reaction time increases the yield of methyl esters obtained because the contact time between the reactants is extended [11]. The presence of a catalyst enhances the reaction rate; therefore, a higher catalyst loading can accelerate the transesterification reaction more effectively [12].

Response Surface Methodology (RSM) is a statistical method widely used to optimize processes involving multiple variables simultaneously. RSM works by depicting the correlation between independent variables and the resulting response using a mathematical model. RSM uses multiple polynomial regression equations to relate the factors to the response values. The validity and significance of RSM models are generally evaluated using Analysis of Variance (ANOVA), which plays a crucial role in identifying significant influencing factors and assessing the model's suitability for the experimental data [13]. Biodiesel is an alternative fuel for diesel engines that can be produced by reacting vegetable oil with methanol. [14]. Biodiesel is produced through Transesterification using a base catalyst. [2]. A commonly used heterogeneous catalyst is CaO. Waste cooking oil has potential as a feedstock for biodiesel production due to its cost and availability. The free fatty acid (FFA) content of used cooking oil is generally around 5-30% (w/w) [15]. The RSM method helps optimise the process by aiming to understand the nature of the response and identify the appropriate areas for the response. Therefore, the RSM method can assume random error. The RSM method has a regression equation for biodiesel yield with the following equation (1) :

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_{ii} X_i^2 + \sum_{i=1}^{n} \sum_{i=1}^{n-1} \beta_{ij} X_i X_j + \varepsilon_i \quad (1)$$

It is known that Y is the predicted response value, bi and bij represent the second-order linear effects of the obtained regression coefficients, and the interaction of x1, x2, and x3. In contrast, n represents the number of independent parameters, and e represents the random error in the response. The resulting responses are then used to derive an equation. This equation is used to compile the predicted equation. The RSM method is used to optimize the data before being analyzed using analysis of variance (ANOVA). Model significance is confirmed by the F-test and P-values of the model [16].

This research aims to optimise biodiesel synthesis from waste cooking oil via microwave-based Transesterification using the RSM method, validated by ANOVA, with catalyst concentration and transesterification reaction time as variables.

2. MATERIALS AND METHODS

2.1. Material and Tools

The materials used in this research included waste palm oil from street vendor in Mojokerto, East Java, Indonesia. Methanol (CH_3OH, 99%, Merck), Calcium Oxide (CaO, Merck), Sulfuric Acid (H_2SO_4, 98%, Merck), and Activated Charcoal. Distilled water (aquadest) was used throughout all experimental procedures. The Equipment List:

1. Condenser
2. Stirrer
3. Two-neck flask
4. Microwave
5. Clamp and Stand
6. Condenser hose
7. Water reservoir tank
8. Water pump

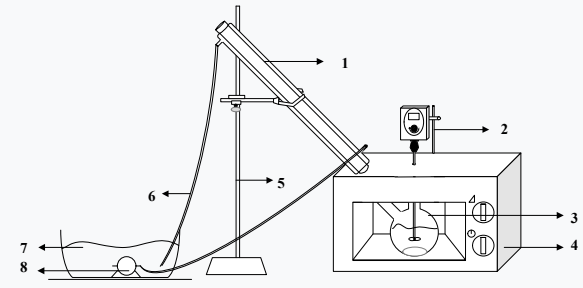

Fig.1. Experimental Setup for Transesterification Biodiesel

2.2. Methods

2.2.1 Pretreatment of Used Cooking Oil

Pre-treatment of used cooking oil by filtering it through filter paper is carried out to remove impurities. The filtrate obtained is analysed for its free fatty acid content by titration with a PP indicator. After that, 7 grams of activated carbon are used to adsorb 40 grams of oil. The adsorption process is conducted at 150°C for 60 minutes to reduce the colour of the used cooking oil. The next step is to esterify 1500 grams of oil from the adsorption process with 78 ml of methanol and 15 ml of sulfuric acid. The mixture is heated to 50-60°C and stirred for 1 hour. The reaction mixture is left to stand for 12 hours and the bottom layer is taken to analyze the free fatty acid content and as a reactant for the transesterification process. as shown in equation (2).

$$FFA\% = \frac{25{,}6 \times V_{NaOH}(ml) \times N_{NaOH}}{m_{Used\ Cooking\ Oil}(g)} \quad (2)$$

2.2.2 Biodiesel Production

The biodiesel production or transesterification process uses 50 grams of esterified oil heated to 60°C. Methanol with a methanol-to-oil ratio of 1:8 is mixed with a catalyst according to the variables (1, 2, 3, 4, and 5%) to form methoxide ions. Transesterification is performed in a three-neck flask heated in a microwave, with oil, methanol, and a catalyst. Heating in the microwave uses 340 watts of power for a given amount of time (2, 4, 6, 8, or 10 minutes) with stirring. The reaction results are left in a separating funnel for 12 hours. The biodiesel product is located in the bottom layer, which is then washed three times with distilled water at 70°C. The water content is separated by heating the mixture to 105°C for 4 hours. The amount of biodiesel formed is measured to determine the percentage yield obtained as shown in equation (3).

$$Yield\ (\%) = \left(\frac{Meth\quad Ester\ Mass}{Oil\ Mass}\right) x100\% \qquad (3)$$

2.2.3 Data Analysis

The results of the study, in the form of per cent yield at varying reaction times and per cent catalyst, were analysed using the Two-Way ANOVA and Response Surface Methodology. The software used is Design Expert version 13. The mathematical model chosen is the quadratic model because it can describe the increase in yield during optimal conditions and its decrease after passing the optimal phase, and it can also describe the relationship between free and bound variables. The contour and surface plots are also used to illustrate the interaction between reaction time and per cent catalyst on biodiesel yield and to determine the combination of variables that produces the optimal biodiesel yield.

3. RESULT AND DISCUSSION

3.1. Yield of Biodiesel

Biodiesel yield is calculated from the volume of biodiesel produced under varying reaction conditions and catalyst loadings. The following are the yield analysis results for biodiesel samples with variations in per cent catalyst and time (Figure 2).

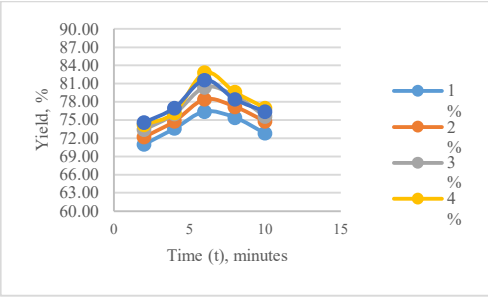

Fig.2. Relationship between Time reaction (minutes) and Percent Catalyst (%) on Biodiesel yield

Figure 2 shows that both reaction time and per cent catalyst affect the biodiesel yield. The longer the reaction time, reaction will decrease the biodiesel yield. This decline is due to a reversible reaction. This reversible reaction will cause the methyl ester to revert to a glyceride as the equilibrium is reached. If equilibrium is reached, the reaction time will not increase the yield but will decrease it due to the reaction's rev*ersibility* [17]. From the research carried out, the best results are obtained with a catalyst percentage of 4% and a reaction time of 6 minutes, yielding 82.80%.

3.2. Two-Way Test ANOVA

The ANOVA test is used to determine whether one or more independent variables can influence a dependent variable. Variable data in the form of reaction time, catalyst percentage, and biodiesel yield were subjected to a normalization test to determine the distribution of the data. The sig value is examined in the ANOVA test. The residual data distribution is usually normal if the p-value is greater than 0.05. Based on Table 1, a p-value of 0.000 was obtained, indicating that the sample data conformed to a normal distribution.

Table 1. Tests of Normality

	Kolmogorov-Smirnov*			Shapiro-Wilk		
	Statistic	df	Sig	Statistic	df	Sig
Standardised Residual for percent_yield	100	75	.000	.815	75	.000

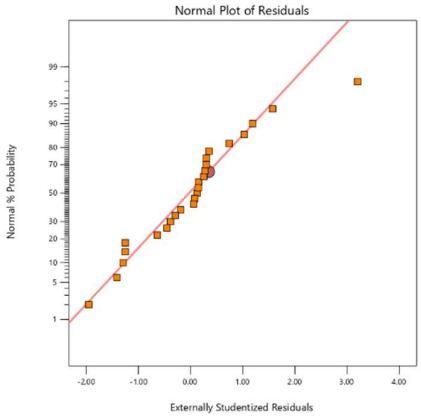

Fig.3. Normal Plot of Residuals

The two-way ANOVA can ignore the sample distribution, allowing the data to be further tested with it. The table that needs to be interpreted is the homogeneity table (Table 2). The data variance is not homogeneous if the p-value is <0.05. Based on Table 2, the sig value is 1.000, indicating that the data variance is homogeneous.

Table 2. Levene's Test of Equality of Error Variances
Dependent Variable: percent_yield

F	df1	df2	Sig
.029	24	50	1.000

The dependent variable is the same across groups because the null hypothesis test results indicate that it is.

a. Design: Intercept + percent_catalyst + reaction_time + percent_catalyst*reaction time

The following important output in the two-way ANOVA test is shown in Table 3: the test of between-subject effects. The percentage values for the factor variables show a significant difference, as the significance value is <0.05 for both reaction time and catalyst percentage. In biodiesel production, reaction

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time and the percentage of catalyst in the transesterification process have a statistically significant effect on the per cent yield obtained. Based on the ANOVA test carried out, a sig value of 0.000 (<0.05) was obtained for the percentage of catalyst, indicating that the percentage of catalyst has a significant effect on the per cent yield. The sig value for reaction time, based on the table, was 0.000 (<0.05), indicating a significant impact on the per cent yield. The table also shows a sig value of 0.000 for percentage_catalyst * reaction_time, indicating an interaction between the percentage of catalyst used and reaction time on the percentage of biodiesel obtained.

Table 3. Tests of Between Subject Effects Dependent variable: percent_yield

Source	Type III Sum of Squares	df	Mean Square	F	Sig
Corrected Model	605.827*	24	25.243	166.509	.000
Intercept	4337222.093	1		2884050.744	.000
Percent_catalyst	172.387	4	43.097	284.280	.000
Reaction_time	414.787	4	103.697	684.01	.000
Percent_catalyst* Reaction_time	18.653	16	1.166	7.690	.000
Error	7.500	50	152		
Total	437835.500	75			
Corrected Total	613.407	74			

3.3. Response Surface on Transesterification Process

This research produced 25 biodiesel value data points that will be optimized using the response surface methodology (RSM). The use of RSM in the optimisation process is very useful for obtaining yield values that meet the target or are optimal. Quadratic equations are used to predict the effect of reaction time and catalyst percent on biodiesel yield. The quadratic equations resulting from the optimization results are as follows:

$$Y = 4.84693A + 2.85879B + 0.0405AB - 2.77714A^2 - 5.95929B^2$$

Optimisation results from RSM can also be visualised using contour and surface plots. The optimization results of the research are presented in the form of a plot (Fig.4).

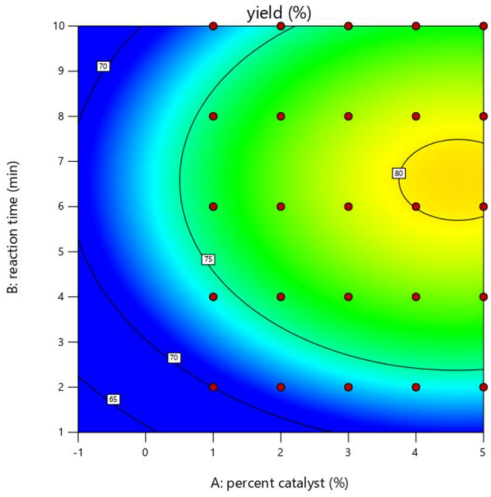

Fig.4. Contour Plot of Yield (%) vs reaction time (min) and percent catalysts (%)

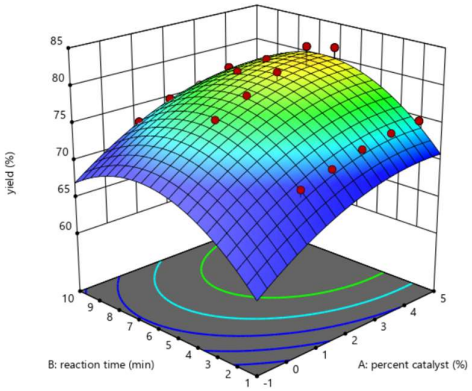

Fig.5. Surface Plot of Yield (%) vs reaction time (min) and percent catalysts (%)

Contour plots are two-dimensional graphs that illustrate the interactions among yield, reaction time, and catalyst percentage. Based on Fig. 4, the optimal conditions lie within the yellow area, with a catalyst percentage of 3–5% and a reaction time of 6–8 minutes. Meanwhile, in the Surface Plot, a three-dimensional graph is formed that can be used to identify the optimum or highest point. This point indicates that optimal and best conditions have been achieved. Based on Fig. 5, the optimum point is reached in the yellow area with the highest peak marked by a red circle. Based on Table 4, the best yield from the Response Surface Methodology analysis is 80.237%, with a catalyst percentage of 4.624% and a reaction time of 6.591 minutes.

Table 4. Solution of Response Surface Methodology

No	Percent catalyst	Reaction time	Yield	Desirability	
1	4.624	6.591	80.237	0.783	**Selected**

4. CONCLUSION

The results of this research indicate that time of reaction and per cent catalyst significantly affect biodiesel yield. The synthesis of waste cooking oil via a microwave-based transesterification process, with variation in time intervals and catalyst percentage, showed an increase in the characteristics toward the standard at a 4% catalyst concentration and a reaction time of 6 minutes, resulting in a yield of 82.80%. The best yield value from Response Surface Methodology (RSM) is 80.237% with a catalyst percentage of 4.624% and a reaction time of 6.591 minutes.

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