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Research Article

Production of biodiesel (isopropyl ester) from coconut oil by microwave assisted transesterification: parametric study and optimization

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Abstract. Biodiesel, a renewable fuel for diesel vehicle engines, has been commonly produced from transesterification process involving triglycerides from vegetable oil with alcohol. One of the most promising candidates for vegetable oil due to its abundance in Indonesia is coconut oil. However, the short carbon chain present in coconut oil necessitates the use of longer-chain alcohol types to adjust to the biodiesel carbon chain, such as isopropanol. Therefore, this research focused on producing biodiesel (isopropyl ester) from coconut oil using isopropanol and NaOH catalyst through a transesterification process. To enhance this process, microwave technology was utilized for its ability to lower the biodiesel production reaction time from the conventional one-hour timeframe to less than ten minutes, increase energy efficiency, and improve biodiesel quality. The primary objective was to investigate the impact of reaction time, catalyst concentration, and microwave power on the isopropyl ester yield. Further optimization was conducted using Response Surface Methodology (RSM) with Box-Behnken Design (BBD) to illustrate the model's effectiveness and applicability. Based on BBD optimization simulation, the optimal condition for producing isopropyl ester from coconut oil using microwave technology is a 1-minute reaction time, 0.2 wt.% NaOH catalyst concentration, and 443.9 W microwave power, maximizing the yield to 99.89%. This research highlights the potential of microwave assisted transesterification and the reliability of this innovative approach, contributing to the development of isopropyl ester production with enhanced quality that meets the specifications of the Indonesian National Standard (SNI).

Keywords: Biodiesel, Coconut Oil, Isopropyl Ester, Microwave, Transesterification.

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1. Introduction

The heavy reliance on fossil fuels as the primary energy source globally has led to significant environmental and economic challenges (Ansori and Mahfud 2021; Nugroho *et al.* 2023). This dependence notably contributes to escalating greenhouse gas (GHG) emissions due to the combustion of fossil fuel, leading to climate change and global warming (Nguyen *et al.* 2023). Additionally, it also results in degradation of environment, along with biodiversity loss, water and air pollution, and oil spills during the processing of fossil fuel (Huang *et al.* 2021; Latif *et al.* 2021; Nugroho *et al.* 2023). Furthermore, besides its detrimental environmental impact, the availability of fossil fuel production in Indonesia has steadily declined annually, leading to Indonesia becoming a net oil importer in recent years. This finite nature of fossil fuel reserves poses economic risks, as fluctuations in supply and demand can potentially cause price volatility and geopolitical tensions (Agarwal *et al.* 2020; Theresia *et al.* 2022).

Given these challenges, and in alignment with an attempt to mitigate environmental pollution, uphold energy and economic security and independence, and accomplish the global commitment to lower greenhouse gas emissions and fossil fuel production, the government is urged to enhance the prominence of renewable energy sources (Asian Development Bank 2020; Abdul *et al.* 2021; Lyeonov *et al.* 2019). Biodiesel, derived from renewable sources, stands as one such alternative fuel, utilized in diesel engines (Hassan and Kalam 2013; Ieva *et al.* 2020). Biodiesel offers several advantages over fossil fuels, including its renewable raw materials, low sulfur content which prevents acid rain, superior lubricant qualities prolonging machine life, a high flash point reducing the risk of fire, the capability to reduce harmful air pollutants, and its sustainable properties (Demirbas 2005; Bustaman 2009; Hadi 2009; Huang *et al.* 2011).

Biodiesel has been industrially produced through transesterification process using palm oil as the feedstock, with methanol as the alcohol and a base catalyst to accelerate the reaction. However, the availability of palm oil plants in Indonesia is currently concentrated primarily to the islands of Kalimantan, Papua, and Sumatra, posing a drawback to its utilization as a primary feedstock. Therefore, an alternative vegetable oils are needed to replace palm oil. Among these options, coconut oil stands out as a greatly potential raw material for the production of biodiesel in Indonesia due to extensive distribution of coconut trees population across the country, spanning every island in Indonesia. In accordance with

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data released by the Organization of United Nations Food and Agriculture in 2020, Indonesia emerged as the world's leading coconut producer, with an output of 20 million tonnes coconuts annually. This trend continued in 2021, solidifying Indonesia's position as the primary global producer of coconuts, which can be utilized not only in the field of food, but also in the energy sector through biodiesel production, following further research and development (Dauqan *et al.* 2011). This aligns with the increasing demand for biodiesel in remote areas of Indonesia, where access to conventional fuels is limited, yet coconut trees are abundant. Thus, the utilization of biodiesel derived from coconut oil will be beneficial to meet the community energy needs.

However, biodiesel derived from coconut oil has a shortcoming due to its composition, containing approximately 70% medium chain fatty acids (C_{12}-C_{16}), mainly lauric and myristic acid (Dauqan *et al.* 2011; Suryanto *et al.* 2015). Consequently, producing biodiesel with coconut oil requires the addition of alcohol with longer carbon chains in comparison to methanol, which contains only one carbon chain. Therefore, isopropyl alcohol is used in this research to elongate the carbon chain of coconut oil to adjust to the biodiesel carbon chain, aiming to produce biodiesel (isopropyl ester) with improved qualities compared to methyl ester, which exhibits poor cold flow properties, potentially leading to crystallization and blockage of fuel line filters at a temperature near 0°C, and it also has a higher toxicity level (Wang *et al.* 2005). Furthermore, isopropyl alcohol is priced comparably to methanol, making it economically viable. This aligns with the objective of minimizing environmental and economic impact, while also ensuring safety in the production process.

In the process production of biodiesel, various methods are employed to enhance the reaction rate and the obtained biodiesel yield, including the use of catalyst and the application of microwave technology. For maximizing the transesterification reaction, a strong base catalyst in the form of sodium hydroxide (NaOH) is added to speed up the biodiesel production reaction. NaOH has been commonly used globally to facilitates the reaction by breaking down the ester bonds in the triglycerides present in coconut oil (Hadiyanto *et al.* 2020). It serves as an effective catalyst with a high reactivity and relatively low cost, requiring no pretreatment, and can operate under ambient conditions. Therefore, it can efficiently enhance the overall effectiveness of the process, addressing the research objective of enhancing process efficiency (Prayanto *et al.* 2016). Additionally, microwave technology has been extensively utilized due to its ability to improve and accelerate the efficiency of transesterification process (Hsiao *et al.* 2011; Kumar *et al.* 2011; Gude *et al.* 2013; Oliveira *et al.* 2013; Manco *et al.* 2016). Microwave technology has been shown to reduce biodiesel production reaction time from one hour in conventional methods to less than ten minutes (Handayani *et al* 2017). Furthermore, the energy consumption associated with microwave-assisted processes is significantly lower, approximately reduced by a factor of 23 compared to the conventional methods (Pathil *et al.* 2010). This efficiency stems from the superior heat distribution of microwaves, which ensures simultaneous and uniform heating, directly transmitting heat through the material (Kumar *et al.* 2011; Prayanto *et al.* 2016; Mahfud *et al.* 2020). Furthermore, microwave technology also offers numerous advantages, including reduced startup time, accelerated heating, enhanced energy efficiency, decreased production costs, ease of control, and improved quality of the final product (El Sherbiny *et al.* 2010; Kumar *et al.* 2011; Silviana *et al.* 2022). Therefore, the use of microwaves would offer advantages in reducing energy consumption and production costs, thereby addressing the research objective of employing practical, economical, and environmentally friendly solutions for biodiesel production, in line with the broader goal of transitioning towards a more sustainable energy future.

To optimize the transesterification reaction variables, evaluate parameters, and understand variable interactions while minimizing the number of experiments and production cost, a statistical analysis method known as Response Surface Methodology (RSM) is used. One of the designs offered by RSM that is frequently utilized to maximize the production of biodiesel is the Box-Behnken Design (BBD). The BBD approach was chosen over other designs because it has a higher prediction accuracy during parameter tweaking while requiring fewer trials. Moreover, the BBD approach outperforms the Central Composite Design (CCD) in terms of efficiency, considering the ratio of model coefficients to the number of trials (Ferreira *et al.* 2007).

Therefore, this research aims to study the transesterification reaction involving coconut oil and isopropanol with NaOH base catalyst enhanced by microwave technology, while considering key operating variables as reaction time, catalyst concentration, and microwave power. These parameters are further optimized using Response Surface Methodology (RSM) with Box-Behnken Design (BBD) to maximize efficiency, reduce costs, and minimize environmental footprint. Subsequently, product analysis was conducted to verify that the final product has the enhanced quality that meets the specifications of the Indonesian National Standard (SNI). Thus, this research can contribute to accelerating the transition towards a more sustainable energy future by utilizing an efficient process of biodiesel production, mitigating climate change, enhancing energy resilience, and sustaining economic growth.

2. Material and method

2.1 Material

Coconut oil that is used as raw material in this experiment is Barco Co coconut oil obtained from supermarkets. The alcohol used is isopropanol (IPA by Petronas Purity 99%) obtained from UD. Crystal Clean in Jakarta. Whereas NaOH catalyst 99% obtained from UD. Sumber Ilmiah Persada in Surabaya.

2.2 Apparatus and experimental procedures

Biodiesel production was done in a batch reactor utilizing microwave technology. Pyrex glass one-neck round bottom flask of 1000 mL and a diameter of 9.85 cm was used as a reactor with a magnetic stirrer inside. The microwave used as the heat source was an Electrolux Microwave EMM2007X with specifications: power setting ranging from 150 to 800 watts, time setting of 0 to 35 minutes, and a frequency of 2450 MHz.

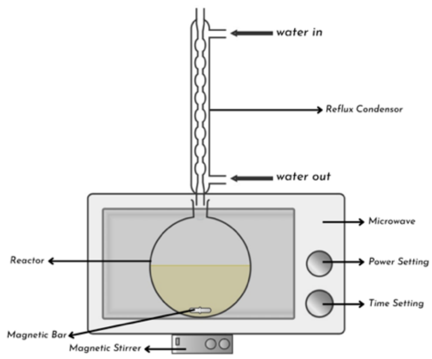

Fig 1. Experimental setup

The reactor and microwave were connected to a reflux condenser measuring 50 cm in length with a $^{24}/_{40}$ ground mouth size. The transesterification reactions were done in the presence of coconut oil, alcohol, and NaOH catalyst (0.2, 0.6, 1 wt.%) with varying reaction times (0.5, 1, 2, 3, 4, 5 minutes), and were carried out using microwaves with power settings of 250, 350, and 450 watts. The process involved introducing 60 mL of coconut oil into the reactor, followed by adding a mixture of 55 mL of alcohol and catalyst with adjusted variables. The mole ratio of coconut oil to alcohol was maintained at 1:9, with the reaction time and microwave power adjusted accordingly. Following the completed reaction, isopropyl ester as the product and glycerol as a by-product along with any residual catalyst were separated using a separating funnel. The product was then washed using warm distilled water and dried at 110°C for 1 hour to remove any remaining water.

2.3 Biodiesel optimization using box-behnken design (BBD)

Response Surface Methodology (RSM) is a statistical technique used to investigate the relationships between parameters while minimizing the number of experimental trials. This statistical technique can help researchers identify patterns, trends, and relationships between variables. Once the relationship of variables is understood, statistical models derived from experimental data can be used to optimize and predict the reaction parameters to achieve maximum yield. RSM begins with designing experiments and obtaining a mathematical model that fits the experimental data.

The Box-Behnken Design (BBD) approach was chosen over other designs because it offers higher prediction accuracy during parameter tweaking while requiring fewer trials (Myers *et al.* 2009; Montgomery 2014). The BBD approach is widely recognized for its effectiveness in optimizing biodiesel production, as demonstrated in the research conducted by Ansori and Mahfud (2021), who used BBD to optimize the interesterification method of biodiesel production from palm oil. Their study resulted in a predicted biodiesel yield of up to 98.64% under specific operating conditions: catalyst concentration of 1.24 wt.%, methyl acetate to oil molar ratio of 18.74:1, reaction temperature of 57.84°C, and reaction time of 12.69 minutes. This research further confirmed the suitability of BBD for optimizing biodiesel production. Therefore, to maximize yield and reduce the number of experiments required in this research, the RSM with BBD approach was employed to optimize the parameters affecting transesterification process: reaction time, concentration of catalyst, and microwave power.

The BBD approach utilized in this study involved three levels and factors, generating a total of 15 experiments aimed at optimizing transesterification parameters. These experiments were conducted to assess the collective effects of the components involved (Jahirul *et al.* 2014). The BBD will provide a matrix design experiment and a response variable in the form of biodiesel yield, as displayed in Table 1. The default coding for the factors is +1 for high levels and -1 for low levels (Kusuma *et al.* 2018). Subsequently, the investigation was conducted with the following parameters: reaction time between 0-1 minute, catalyst concentration between 0.2-1%, and microwave power between 250-450 W. For the statistical technique, Minitab software version 21.4 (Minitab Inc., Pennsylvania) was applied to generate model data, experimental design, and its statistical analysis. The second-order polynomial model in generalized form applied in the RSM simulation which represented by Equation 1 (Iqbal *et al.* 2020).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_{i} x_{i} + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} \beta_{ij} x_i x_j \qquad (1)$$

Table 1
Selected independent factors levels

Independent factors	Levels		
	-1	0	1
Reaction time (min)	0	0.5	1
Catalyst concentration (%)	0.2	0.6	1
Microwave power (W)	250	350	450

Where, Y represents the predicted response, β_0 is the constant coefficient for regression, β_i is the linear coefficient for regression, β_{ii}, is the quadratic coefficient for regression, and β_{ij} is the interaction coefficient for regression. Meanwhile, x_i and x_j are the codes of the parameters.

2.4 Analysis of product

The resulting isopropyl ester product undergoes further analysis to assess its viscosity, density, and flash point to determine the quality of the product obtained. Furthermore, Gas Chromatography with Flame Ionization Detector (GC-FID) analysis was done to identify the glycerides contents.

Density analysis is conducted to determine the mass present within each unit volume of biodiesel produced. Density analysis of biodiesel is performed using a pycnometer. Initially, the empty pycnometer's mass is recorded, followed by recording the final mass of the pycnometer containing biodiesel to determine the mass of biodiesel. The density of biodiesel is obtained by dividing the mass of the biodiesel by the volume of the pycnometer. Viscosity analysis is performed using an ostwald viscometer. This calculation is done by comparing the time required for water and biodiesel to reach a specified line on the viscometer.

Flash point analysis of biodiesel is performed to discover the lowest temperature at which biodiesel ignites and briefly burns. Additionally, flash point testing is also carried out to comply with biodiesel specifications. The testing is performed using the ASTM D 93-02A method with the Pensky-Martens closed cup apparatus (SYD-261D) within normal room conditions, with temperature of 22°C and RH of 58.5%. The testing procedure begins by placing the sample into the test cup and covering it with a lid. The sample is heated and stirred, and then an ignition source is periodically directed into the cup. The test concludes with observing any flash of light that spreads throughout the cup's interior.

Gas Chromatography with Flame Ionization Detector (GC-FID) analysis is done to investigate the glycerides contents of isopropyl ester. The procedure begins with manual injection of the sample from the front part of the instrument using the split method with a ratio of 10:1. The GC system used is the Hawlet Packard (HP-6890) with the HP-INNOWax capillary column. The initial oven temperature program is set at 140°C for 2 minutes, then increased to maximum of 250°C at a rate of 50°C per minute. The column uses helium gas as the makeup flow, and the detector uses a combustion gas consisting of hydrogen and air, with the temperatures of the injector and detector is at 240°C. Glycerides identification is performed by comparing peak areas to standard fatty acid esters or by comparing retention times of standard fatty acid esters with the sample.

Meanwhile, Biodiesel yield is calculated by multiplying the mass analysis by the content of isopropyl ester determined through viscosity analysis, as outlined in Equation 2.

$$\text{Yield (\%)} = \frac{W_{biodiesel}\ x\ Purity}{W_{coconut\ oil}}\ x\ 100\% \qquad (2)$$

Where, $W_{biodiesel}$ is the actual weight of biodiesel obtained (g), $W_{coconut\ oil}$ is the initial weight of coconut oil (g), while purity of isopropyl ester obtained using viscosity comparison approach.

3. Results and discussion

3.1 The relation between microwave power and microwave temperature

The relation between microwave power and temperature can be analyzed by measuring the microwave temperature with a thermocouple inserted into a reactor containing reactants adjusted to the power variable. Temperature checks were conducted according to the reaction time variable until the data in Figure 2 was obtained.

Microwave heating process involves the transmission of electromagnetic waves. When a material is irradiated by electromagnetic waves, it will resonate due to changes in dielectric polarization under an alternating electromagnetic field, leading to heating. As microwave energy hits the target material, the molecules of the material will rotate rapidly, generating heat through molecular friction. The increase in microwave power used will directly affect the rate of molecular rotation, thus also affecting the temperature generated (Kumar *et al.* 2011; Prayanto *et al.* 2016). Consequently, the power used in the microwave will influence the temperature profile during its operation and affect the time taken to reach equilibrium.

During the transesterification process, the reaction temperature, specifically the equilibrium temperature, has a significant influence on reaction kinetics and product yield. Equilibrium temperatures denote the stable thermal conditions reached within the reaction medium, indicating that heat distribution is uniform and consistent throughout the system. Achieving equilibrium temperatures is crucial as it ensures optimal conditions for the transesterification reaction to proceed efficiently as it directly affects reaction kinetics. Uniform heat distribution results in higher temperatures, generally leading to faster reaction rates by providing the necessary activation energy for the reaction to occur, as already proposed by the Arrhenius law. Within the context of the production of biodiesel by microwave assisted transesterification, reaching equilibrium temperatures ensures that the reaction progresses at an optimal rate, maximizing the conversion of triglycerides into alkyl esters. Furthermore, equilibrium temperatures play a critical role in determining product yields. High biodiesel yield can be achieved if the reaction is maintained at appropriate temperatures (Kusuma *et al.* 2018).

In Figure 2, it is known that the temperature will start to reach equilibrium at the 4th to 5th minute. At 250 W power, the

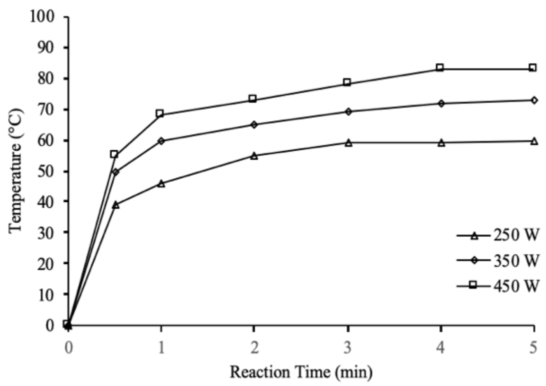

Fig 2. Microwave temperature profile

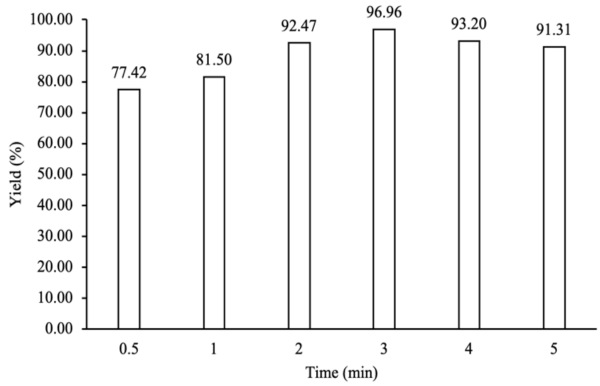

Fig 3. Reaction time effect on isopropyl ester yields with 0.2 wt.% NaOH catalyst and microwave power 350 W

temperature reaches equilibrium at around 60°C. At 350 W power, the temperature reaches equilibrium at around 73°C and 450 W reaches equilibrium at around 83°C. Microwave can be used to heat a mixture of oil and alcohol to a temperature above the boiling point of the solvent in a faster time rather than the conventional heating (Rohman and Fatmawati 2016).

3.2 The effects of reaction time

Experiment was conducted at 350 W microwave power with a NaOH catalyst concentration of 0.2 wt.%. As shown in Figure 3, the resulting yields were 77.42%, 81.5%, 92.46%, 96.96%, 93.20%, and 91.31% for reaction times of 0.5, 1, 2, 3, 4, 5 minutes respectively. This demonstrates that the reaction time variable can have an impact on the yield of isopropyl ester produced. The time required for transesterification holds a significance role in ensuring that the reaction can run perfectly and produce high yields. Longer reaction time will make the continuous electromagnetic field from the microwave interact with dipole molecules and ions which causes excitation and produces heat rapidly (Manco *et al.* 2011) so it will make the reaction temperature higher.

Elongating the time of reaction will also make the biodiesel yield higher because it gives more time for alcohol to convert triglycerides into alkyl esters. However, if the reaction continues, there will be a decrease in yield which can be seen from Figure 3, when the time enters the fourth minute. The yield will decrease and reach constant after a reaction time of around 3 minutes because it is possible that the reaction has reached equilibrium. If transesterification is carried out for a longer time, it will likely cause soap formation or a saponification reaction since it has passed equilibrium condition. Otherwise, if the transesterification is done in a shorter duration, there is a chance that monoglyceride and diglyceride production will increase and ester production will decrease due to incomplete transesterification reaction (Zhang *et al.* 2010). Therefore, in order to provide the higher yield, the proper reaction time must be determined (Kusuma *et al.* 2018).

3.3 The effects of catalyst concentration

According to the catalyst's ability to fasten the reaction, the use of a catalyst will give an influence on the transesterification reaction. To examine the catalyst effect, the experiment was conducted for 2 minutes with microwave power of 350 W. The resulting yields were 96.96%, 96.74%, and 93.38% when the catalyst used was 0.2, 0.6, and 1 wt.% respectively. From Figure 4, it is shown that the more of the % catalyst used, the lower the yield of isopropyl ester produced, but this does not significantly affect the yield obtained (there is a tendency to produce yields that are quite close to the same). This shows that the use of

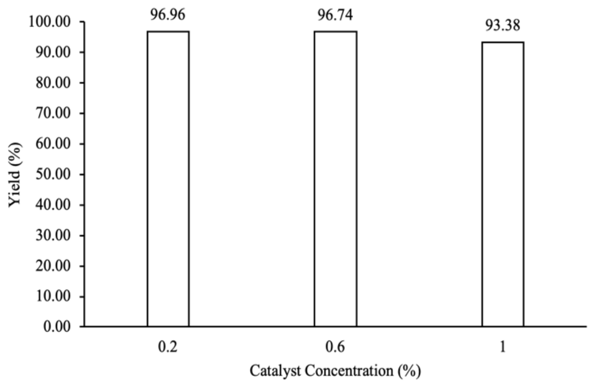

Fig 4. Catalyst concentration effect on isopropyl ester yields with microwave power 350 W and reaction time 2 minutes

microwaves can suppress the catalyst needed in the transesterification reaction. This is consistent with studies from Suryanto *et al.* (2015), which indicates that there will be a propensity for the biodiesel yield to create the same value of yield if the concentration of the catalyst higher than 0.2–0.3 wt.%. In addition, excessive amount of alkaline catalyst will cause an increase in emulsion formation, increasing biodiesel viscosity which can lead to gel formation which will hinder the separation of glycerol and cause dilution of the biodiesel and lower the amount of product that is produced (Suryanto *et al.* 2015; El-Gendy *et al.* 2015).

3.4 The effects of microwave power

To find out microwave power influence, experiment was done under the operating conditions of 0.2 wt.% NaOH catalyst and 2 minutes time of reaction. Figure 5 illustrates that microwave power influences the yield of isopropyl ester. The yields produced were 64.96%, 96.74%, and 99.34% for power 250, 350, 450 W. This shows that an increase in the microwave power used can cause an increase in yield. A higher yield on the product can be achieved by choosing the appropriate microwave power. Higher microwave power will also give a higher temperature where temperature can affect the reaction rate (Suryanto *et al.* 2015). The temperature profile in Figure 5 shows that when the reaction time is 2 minutes, at each power the temperature does not exceed 75°C which is still quite far from the boiling point of isopropyl alcohol (boiling point 82.5°C) so that in this reaction, the temperature still does not cause alcohol evaporates in the reflux condenser, which will not shift the reaction equilibrium to the reactants.

However, excessively high microwave power levels can also cause the saponification reaction rate which increase triglyceride to soap production process. The biodiesel formation reaction is reversible, while the saponification reaction is irreversible. The reversible biodiesel reaction will create a reaction equilibrium. Soap formation potential arises when the reaction equilibrium shifts towards the reactants, leading to triglycerides reacting with the remaining alkaline catalyst to generate soap (Rohman and Fatmawati 2016).

3.5 Box-behnken design (BBD) optimization analysis of isopropyl ester production

The BBD method employs three independent parameters: reaction time (A), catalyst concentration (B), and microwave power (C). Initially, the highest and lowest point on each variable were selected as the two primary points. For the time variable, the highest point chosen was 1 minute and the lowest point was 0 minute, as the reaction shows a noticeable response within this timeframe. 15 trials were done in total to discover the interaction of biodiesel production utilizing the BBD method. The optimization simulation resulted in the biodiesel (isopropyl ester) yield equation shown in Equation 3.

$$Yield = -160.1 + 150.7A + 93.4B + 0.723C - 143.8A^2 - 20.9B^2 - 0.000800C^2 + 16.1AB + 0.1905AC - 0.179BC \quad (3)$$

The actual equation will be derived as a second-degree polynomial. Equation 3 can be utilized to estimate the response value in the form of isopropyl ester yield for the levels of each independent factor. Equation 3 can also be applied for predicting the model fit compared with the experimental data, as indicated by the lack of fit value, as shown in Table 2. From the Equation 3, each coefficient indicates the changes affected by each independent variable when studied under constant, linear, quadratic or interaction conditions. A high coefficient value indicates the significance of the parameter and its impact on the average change in biodiesel yield.

An analysis of variance (ANOVA) was done to assess each variable influence in affecting the yield of isopropyl ester. The actual data from each parameter utilized can be evaluated using the statistical model in Table 2, Table 3, and Figure 6.

The linear model indicates that the yield of isopropyl ester is significantly affected by two parameters: reaction time (A) and microwave power (C), as proved by the p-value < 0.05.

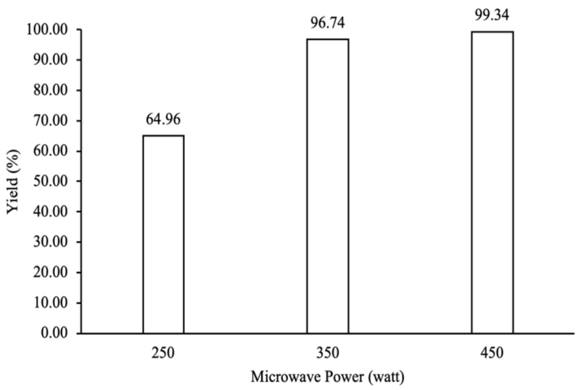

Fig 5. Microwave power effect on biodiesel yield with catalyst concentration 0.2 wt.% and reaction time 2 minutes

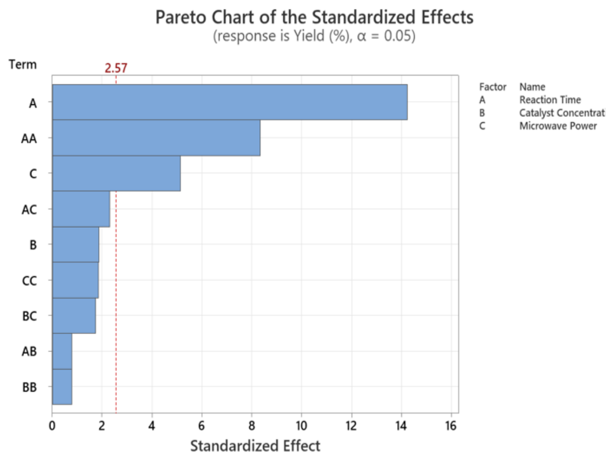

Fig 6. Pareto chart for biodiesel optimization

Table 2
Analysis of variance (ANOVA) isopropyl ester optimization

Source	Degree of freedom	Adj sum square	Adj mean square	F-value	P-value	
Model	9	21410.5	2378.9	34.73	0.001	*
Linear	3	15927.4	5309.1	77.51	0.000	*
A: Reaction time	1	13877.8	12877.8	202.59	0.000	*
B: Catalyst concentration	1	237.6	237.6	3.47	0.122	**
C: Microwave power	1	1812.0	1812.0	26.45	0.004	*
Square	3	4872.6	1624.2	23.71	0.002	*
A^2	1	4770.8	4770.8	69.65	0.000	*
B^2	1	41.3	41.3	0.60	0.472	**
C^2	1	236.1	236.1	3.45	0.123	**
2-Way interaction	3	610.4	203.5	2.97	0.136	**
AB	1	41.6	41.6	0.61	0.471	**
AC	1	362.9	362.9	5.30	0.070	**
BC	1	205.9	205.9	3.01	0.143	**
Error	5	342.5	68.5			
Lack of fit	3	327.1	109.0	14.12	0.067	**
Pure error	2	15.4	7.7			
Total	14	21753.0				

* = significant
** = insignificant

Table 3
ANOVA fitting statistics for isopropyl ester optimization

Fit Statistics	Result
R^2	0.984
Adjusted R^2	0.956
Predicted R^2	0.758
Adeq Precision	8.276

However, catalyst concentration (B) did not show a significant effect, as it has a p-value > 0.05. Additionally, the quadratic model and 2-way interaction models were also used to examine the interaction of each parameter. It is known that quadratic reaction time (A^2) is the sole factor that has a substantial influence on isopropyl ester yield (p-value < 0.05). This demonstrates that A, C, A^2 are the primary factors in the transesterification process that influence isopropyl ester yield, as shown in Table 2. It is also proven in the Figure 6 which indicates that the other factors do not provide a minimum value for the standardization effect in Pareto chart, so it is considered as insignificant factors (Ahmad *et al.* 2015; Bahadi *et al.* 2019). Furthermore, the lack of fit p-value is known to be 0.067, showing that the model is considered suitable as it is not significantly affected by pure error (Kashyap *et al.* 2018; Bahadi *et al.* 2019).

From Table 3, the R^2 value of 0.984 demonstrates that the model can be effectively used and explains 98.4% of the variability of the response. This high value of R^2 implies a strong relation between actual experiment and predictive simulation, indicating the model's accuracy in estimating biodiesel yield is high. Additionally, the adjusted R^2 value is 0.956, which does not differ significantly from the predicted R^2 (0.758) by more than 0.2. Therefore, the model used is considered feasible and sufficient. Furthermore, the value of adeq precision obtained is 8.276, showing that the model is accurate and beneficial for navigating the design area (Singh *et al.* 2009; Kolakoti *et al.* 2022; Mugagga *et al.* 2023).

Overall, it is recognized that the model utilized in optimizing biodiesel (isopropyl ester) using the BBD approach is adequate and reliable, as evident from Tables 2 and 3, as well as Figure 6. These findings demonstrate the capability of this approach in predicting and optimizing biodiesel production.

3.5.1 Interaction parameter and contour plot RSM

In this study, the primary impacts and interactions of each parameter on the biodiesel (isopropyl ester) yield produced were examined using contour and surface plots. The equation of regression is graphically illustrated by contour and surface plots, which display two factors while maintaining the other variables constant. The plot in the quadratic model given by

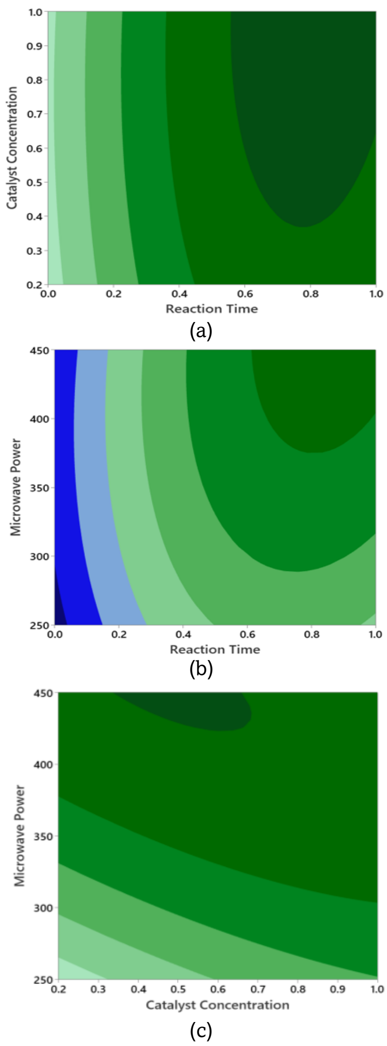

Fig 7. Contour plot of isopropyl ester production showing the effect of interaction between: reaction time and catalyst concentration (a); reaction time and microwave power (b); catalyst concentration and microwave power (c)

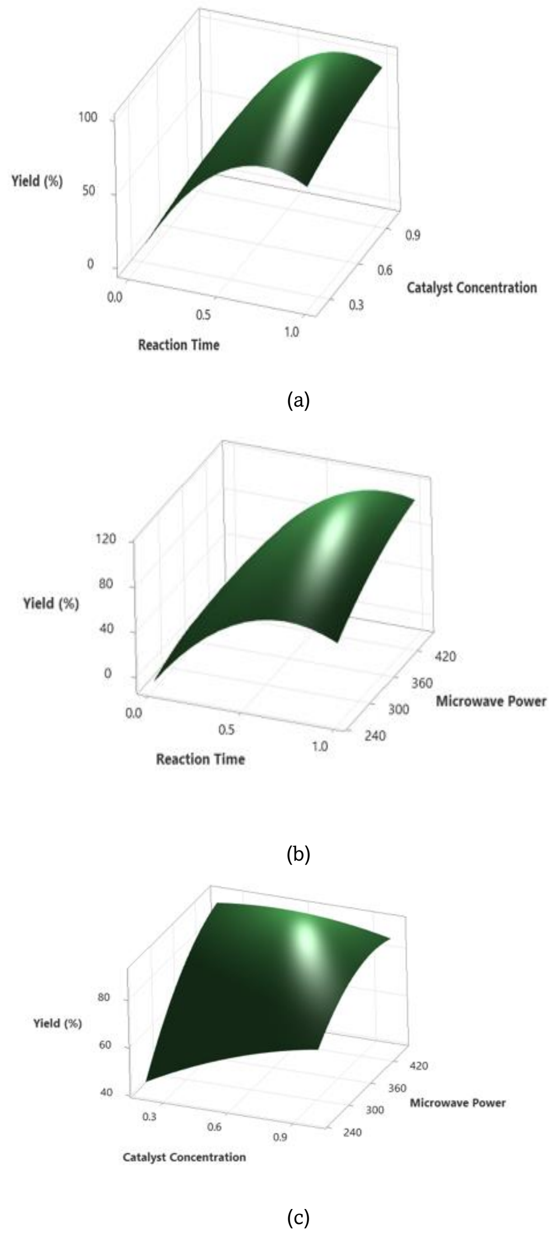

Fig 8. Surface plot of isopropyl ester production showing the effect of interaction between: reaction time and catalyst concentration (a); reaction time and microwave power (b); catalyst concentration and microwave power (c)

equation (3) is obtained by using regression. In this plot, the parameters that are held constant are reaction time 0.5 minutes, catalyst concentration 0.6 wt.%, and microwave power 350 W. The interaction of variables on yield are presented in Figure 7 and 8. The geometry design of contour and surface plots illustrates the combined impact of the independent factors on the response parameters. These plots serve as predictive tools, indicating the relevance of the combined interaction between factors and highlighting the optimum response through color degradation. In optimizing the reaction conditions, highest yield value can be obtained when the reaction is conducted at the plot which gives the darkest color on the contour plot and the lightest color on the surface plot. Furthermore, the contour plot's geometry provides the level of interaction and actuality between the independent variables. Circular contours indicate less interaction among the operational variables, while elliptical contours suggest a cumulative combination of the procedural variables, indicating a complete reciprocal interaction between all variables (Elgaindi *et al.* 2022).

Biodiesel production parameters interact in complex ways to influence biodiesel yield, as discussed in the biodiesel parametric study (Part 3.2 – 3.4). Overall, the interactions between each parameter, including reaction time, catalyst concentration, and microwave power, are crucial in determining the efficiency and yield of biodiesel production. Optimizing these parameters necessitates employing a statistical approach, as response surface methodology, to achieve the desired biodiesel yield while minimizing energy consumption and production costs.

3.5.2 Model adequacy

Model validation involves verifying the model's accuracy by comparing its predictions with actual experimental data. A reliable and highly precise mathematical model should provide a close approximation to real-world processes. The findings from the model suitability investigation are presented in Figure 9.

The transesterification process's design and residual effects are presented in Figure 9. Figure 9(a) shows the % normal versus residual probability plot which is an evaluation

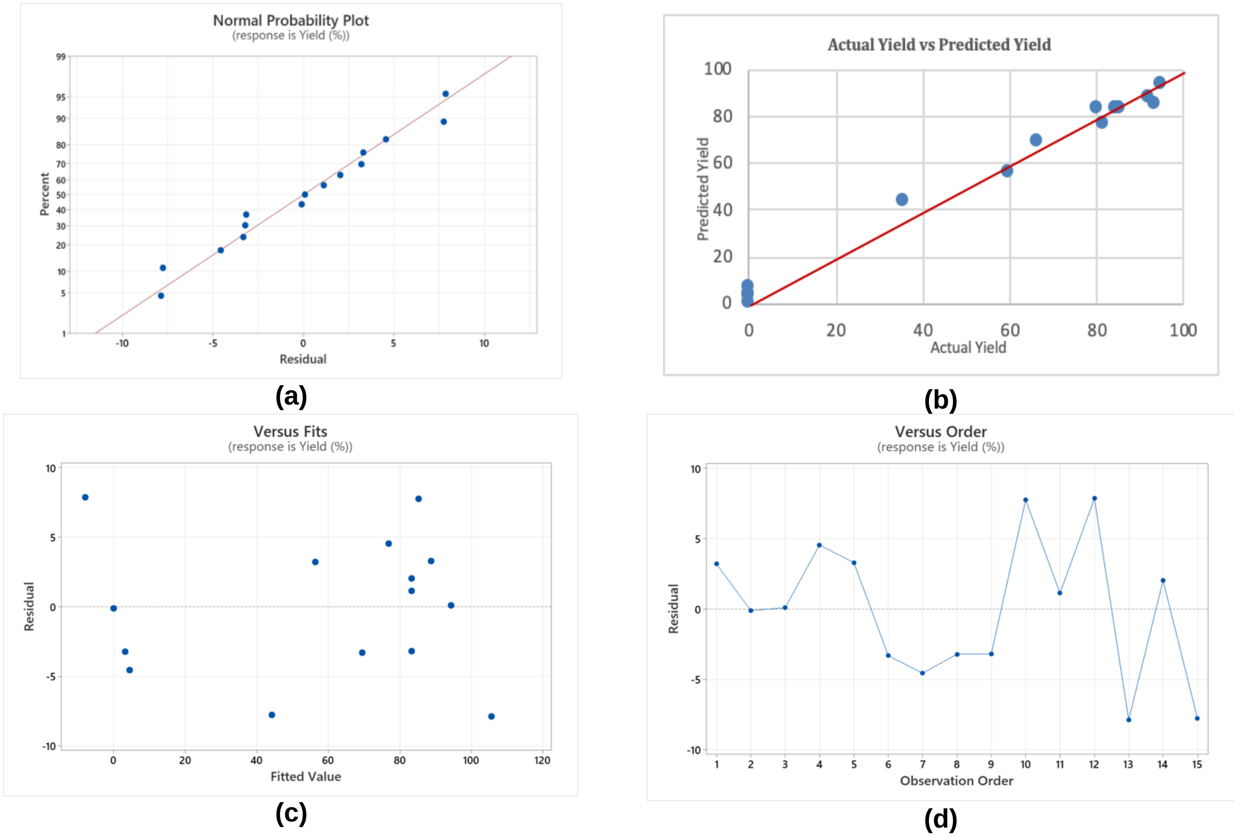

Fig 9. Model adequacy graph: normal probability plot (a); predicted yield vs actual yield (b); versus fits (residual vs fitted value) (c); versus order (residual vs observation order) (d)

instrument for examining and interpreting the assumptions of a normally distributed response system. This is shown by the values of the experiment which proportionally lie on a straight line (majority of the points are around the straight line) so there are not any response alterations, variance variations, or issues with normality. As a result, the graph is deemed adequate. Meanwhile, Figure 9(b) shows yield of actual condition versus the predicted yield. It is clear from the graphic that the points are uniformly spaced out near a straight line, demonstrating a strong correlation and indicating that the outcomes are adequate. Furthermore, Figure 9(c) shows the relationship between predicted versus studied residues for the transesterification process. It is visible that the variance does not change for any response value. This is implied by the points distribution that spread out randomly throughout the boundary around 0 to ± 10. Therefore, this model is suitable and there is no need to change the response variable while using this model in this research. Meanwhile, the observation order vs residual plot provides information for analyzing the model's adequacy (Figure 9 (d)). As can be seen in the Fig 9(d), every experimental data point remains within the range of 0 to ±10. This implies that the model is not affected by unexpected errors. Based on the favorable graph results in Figure 9, it is possibly inferred that the BBD model used is appropriate for characterizing and optimizing the coconut oil transesterification using microwave (Ansori and Mahfud 2021).

3.5.3 Box-behnken design (BBD) optimization

Optimization was done to examine the optimum value of the reaction condition on biodiesel (isopropyl ester) yield using Minitab software. The results obtained indicate that a yield up to 99.89% can be achieved by operating at one minute reaction time, 0.2 wt.% NaOH catalyst concentration, and a microwave power of 443.9 W.

These findings indicate that the optimized reaction conditions achieved through the BBD approach offer a significant advancement in biodiesel production efficiency and sustainability. The model demonstrates high accuracy in predicting and optimizing biodiesel yield, as evidenced by the strong correlation between actual experiments and predictive simulations. These optimized conditions not only enhance biodiesel production yield but also contribute to reducing energy consumption and production costs. Ultimately, the application of these optimized conditions holds great promise for the development of sustainable and improved biodiesel production processes, paving the way for a greener and more environmentally friendly energy future.

3.6 Analysis of biodiesel (isopropyl ester)

In this study, biodiesel (isopropyl ester) was produced from transesterification process of coconut oil, isopropyl alcohol, and NaOH base catalyst using a microwave. The resulting product is in the form of isopropyl ester with a clear color and slightly brown glycerol by-product. The composition of the isopropyl ester product resulting from Gas Chromatography with Flame Ionization Detector (GC-FID) investigation is shown in Table 4 and Figure 10. It is known in Table 4 that the GC-FID results contain an isopropyl laurate component of 47.42%. This is in accordance with the main content of coconut oil which is lauric acid.

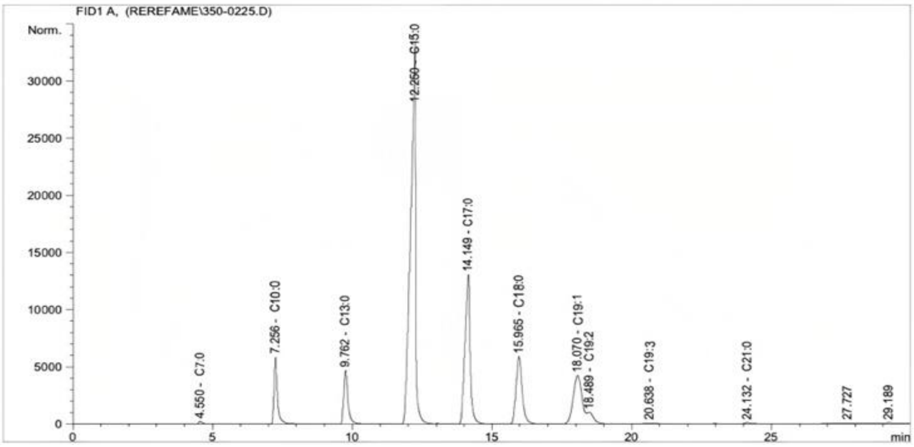

Fig 10. GC-FID result of isopropyl ester obtained

3.7 Product comparison to Indonesian National Standard (SNI) of biodiesel

Character analysis of the isopropyl ester product obtained was conducted and compared to biodiesel Indonesian National Standard (SNI 7182:2015) to prove that the experiment product is suitable for use. From Table 5 it is known that the density of isopropyl ester obtained at a temperature of 40°C was 0.87 g/cm^3 which satisfied the SNI-2015 standard. The resulting kinematic viscosity was 3.99 cSt where the SNI-2015 standard was 2.3-6 cSt. The flash point of the product produced is 172.5°C, which exceeds the SNI-2015 standard minimum. It is also known that using alcohol in the form of isopropanol will raise the biodiesel flash point produced and enhanced the product quality, this is proven by the results of previous research data by Suryanto *et al.* (2015) who conducted research with alcohol in the form of methanol which only gave a flash point value of 125°C that is still below the SNI-2015 standard. The results shown that product obtained satisfied Indonesian National Standard and can be considered a substitute or additive for diesel fuel.

Table 4
Composition of isopropyl ester obtained

Retention time	% Area	Name
4.55	0.22%	Isopropyl Butanoate
7.26	5.53%	Isopropyl Caprylic
9.76	5.46%	Isopropyl Capric
12.25	47.42%	Isopropyl Laurate
14.15	18.95%	Isopropyl Myristate
15.97	10.02%	Isopropyl Pentadecanoate
18.07	10.31%	Isopropyl Palmitate
18.49	1.85%	Isopropyl Palmitate
20.64	0.15%	Isopropyl Palmitate
24.13	0.10%	Isopropyl Stearate
Total	100.00%	

Table 5
Product comparison to SNI

Parameter	Unit	Product	Standard
			SNI 7182:2015
Density (40°C)	g/cm^3	0.87	0.85 – 0.89
Kinematic Viscosity (40°C)	cSt	3.99	2.3 – 6.0
Flash Point	°C	172.5	Min. 130

4. Conclusion

The use of microwave technology can be utilized to speed up the transesterification reaction time when compared to conventional methods. From this study, reaction time and microwave power known to have an influential effect to the biodiesel (isopropyl ester) yield and are directly proportional to the yield obtained up to the equilibrium point, however, the % catalyst give an insignificant influence on the isopropyl ester yield. The highest yield of isopropyl ester produced was 99.5% at 3 minutes reaction, 0.2 wt.% NaOH catalyst, and microwave power 450 watt. In addition, Response Surface Methodology (RSM) using Box-Behnken Design (BBD) was done to enhance biodiesel (isopropyl ester) production and has demonstrated the suitability and effectiveness of the model used according to the outcomes of analysis of variance (ANOVA). Based on BBD optimization simulation, the optimal condition in producing biodiesel (isopropyl ester) from coconut oil using microwave is 1 minute reaction time, 0.2 wt.% NaOH catalyst, and 443.9 W microwave power, which will maximize the yield up to 99.89%. It was also found that biodiesel obtained satisfied Indonesian National Standard (SNI) specifications and could be used as an additive or replacement for diesel fuel.

This research highlights the potential of microwave assisted transesterification and the effectiveness of this innovative approach, contributing to the development of isopropyl ester production with enhanced quality. Additionally, regarding the insignificant influence of catalyst concentration on biodiesel production, the possibility of investigating various percentages of catalyst concentration or lower percentages of catalyst concentration has to be considered for further research.

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