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Taguchi Experiment Design for DES K_2CO_3-Glycerol Performance in RBDPO Transesterification

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Abstract

Biodiesel production using novel glycerol and potassium carbonate-based catalysts has not been developed under the Taguchi technique. This study aims to determine the most influential parameter in biodiesel production from refined bleach-deodorized palm oil (RBDPO) using DES K2CO3-Glycerol as the novel catalyst. The raw material was subjected to transesterification at the desired reaction parameters estimated by the orthogonal 16-run (L16) approach with 2 levels and 4 factors of the Taguchi technique. Signal-to-noise ratio (SNR) and ANOVA were used to confirm the predicted value. From the results, the catalyst is the most influential variable in the TG value of biodiesel, placed in the first rank of the influence factor. Biodiesel production with a minimum total glycerol value (0.210%) using DES K2CO3-Glycerol as a catalyst is most optimally produced at 95 °C for 4 h and 400 rpm using 30 wt% methanol and 4 wt% catalysts achieved by the Taguchi technique. The biodiesel obtained from RBDPO complies with the required international standards.

Keywords:

Biodiesel; Deep Eutectic Solvent; Orthogonal Array; Taguchi Method; Optimization.

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1- Introduction

Crude oil energy sources will not be sufficient to meet human needs for long. Air pollution due to the use of fossil fuels, which leads to suspended particles and carbon dioxide emissions, is unavoidable. The immense contribution of these emissions has led to ozone layer depletion and global warming. Fatty acid methyl esters (FAME) or biodiesel are non-toxic, renewable, and biodegradable and are similar to conventionally obtained diesel. Biodiesel is produced through a transesterification reaction between triglyceride and primary alcohol in the presence of a catalyst [1–3]. Biodiesel can be used as a transportation fuel and can be mixed with diesel without engine modification. In 2019, the total biodiesel production in Indonesia reached 7380 million liters, equivalent to 16% of the global production [4], making Indonesia the third largest biodiesel producer globally.

In biodiesel production, catalysts play an essential role in increasing the conversion and yield of biodiesel. Thus, the production cost often becomes higher to supply the catalyst. The overall production cost can be reduced by optimizing the efficiency of the catalyst used [5]. The catalysts used in transesterification reactions are homogeneous or heterogeneous catalysts. Conventionally, homogeneous base catalysts such as NaOH, KOH, CH_3ONa, and CH_3OK have been used [6]. The common catalysts used in biodiesel production were potassium and sodium methylate. Unfortunately, Indonesia still imports this quite expensive catalyst [7].

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Opportunities for the availability of inexpensive and suitable catalysts come from catalysts based on deep eutectic solvents (DES) [8]. DES is an eutectic mixture obtained from the complexation of two to three pure components interacting through hydrogen bonds to form stable hydrogen bonds. Usually, the melting point of DES is lower than that of each of its pure components [9–11]. DES is made from a mixture of salts and other compounds that act as hydrogen donors, such as alcohols, acids, halides, and amines [12]. Deep eutectic solvents (DES) are formed from Brønsted or Lewis acids, and base mixtures can be made from renewable components. DES consists of plant metabolites (such as ammonium salts, sugars, and organic acids) and is also known as a natural eutectic solvent (NADES) [13]. DES has non-volatile properties and high thermal conductivity and stability [14]. DES can dissolve organic compounds, inorganic compounds, and even reagent mixtures [15]. Furthermore, DES can be applied as a catalyst and co-solvent in transesterification to reduce the saponification reaction and facilitate the separation and purification of glycerol and residual base catalysts in biodiesel products [16].

Researchers have investigated the development of DES. Manurung et al. [17] performed a biodiesel purification process from vitamin E using DES K_2CO_3-Glycerol. DES-based ChCl-Glycerol is also used as a co-solvent in biodiesel production using KOH-catalyzed and ethanol solvents [18]. Hayyan et al. [19] produced low-grade CPO-based biodiesel using a two-stage process using phosphonium-based (P-DES) and alkali-based eutectic solvents as catalysts. Shahbaz et al. [20] combined $DESNH_4$ and phosphonium-Glycerol in the biodiesel purification process resulting from the transesterification reaction. Low-cost DES as a new reaction medium for producing enzymatic biodiesel from waste oil was used by Merza et al. [21]. DES consisting of chlorine-chlorides and glycerol was tested, and its effectiveness was compared with 1-butyl-3-methylimidazolium hexafluorophosphate. DES is more often used as a solvent than as a catalyst in biodiesel production. Thus, the role of DES as a catalyst, especially DES K_2CO_3-Glycerol, needs to be investigated.

The Taguchi method is a statistical method developed by Genichi Taguchi to improve production quality and has recently been applied to engineering and biotechnology [22]. The Taguchi method contains system and parameter design and tolerance design procedures to achieve robust processes and results for the best product quality [23]. The effects of temperature, reaction time, catalyst concentration, and oil and methanol molar ratios can be investigated to obtain the maximum yield of methyl esters. The number of research runs required can be reduced with a matrix designed using the Taguchi method compared to other optimization techniques [24–26]. Regression model equations were used to check the relevance of the studied parameters, which were analyzed statistically to determine the relationship using analysis of variance (ANOVA) [27].

Research on the application of the Taguchi method to investigate parameters affecting biodiesel production has been carried out by several previous researchers. Karmakar et al. [28] carried out homogeneous and heterogeneous acid esterification of castor oil with a FAME yield of 90.83% with a homogeneous catalyst from castor oil using the Taguchi L16 orthogonal array approach. Karabas [29] optimized the reaction parameters for biodiesel production from crude kernel oil through the orthogonal arrangement of L9 by the Taguchi method, resulting in fewer experiments (< 81). The highest methyl ester yield was 90%, with 0.7 wt% KOH as the catalyst and a molar ratio of methanol to oil of 8:1 at 50 °C for 40 min. Saravanakumar et al. [30] used the Taguchi method to optimize Pongamia oil biodiesel production parameters. The maximum biodiesel yield obtained was 86% with a stirring speed of 550 rpm and 15 g of NaOH catalyst for 80 min. Kumar et al. [31] applied the Taguchi method to biodiesel production from safflower oil with an orthogonal L9 arrangement. The highest biodiesel yield obtained was 93.8% at 60 °C for 90 minutes with a molar ratio of methanol and oil of 4:1 and a catalyst of 1.5 wt%. The study revealed that the catalyst concentration contributed 51.1% as the parameter that most influenced the yield. Meanwhile, biodiesel production from Gadhave & Ragit [32] by optimizing parameters using the Taguchi method produces a yield of up to 93.20%. The methanol ratio is the most influential parameter of the biodiesel yield in this work.

Studies in the literature only focused on predicting the best parameters to produce high biodiesel yields, while total glycerol as a biodiesel quality parameter was not considered. To the best of our knowledge, no research has been reported regarding identifying significant parameters that affect the optimization of the transesterification of refined bleach-deodorized palm oil (RBDPO), a derivative fraction of purified palm oil, with a DES K_2CO_3-Glycerol catalyst using the Taguchi method approach. This research aims to determine the best parametric condition to obtain FAME with the lowest total glycerin value using the orthogonal array Taguchi method.

2- Materials and Method

2-1- Synthesis of DES K_2CO_3-Glycerol

The eutectic solution in K_2CO_3 – glycerol was synthesized from glycerol and K_2CO_3 with a molar ratio of 3.5: 1. K_2CO_3 salts and glycerol as hydrogen donors were mixed and stirred at 80 °C for 2 hours. The potassium glyceroxide–glycerol mixture prepared from DES was then heated at 120 to 180 °C. During this process, CO_2 and H_2O will be released. H_2O will be condensed, while CO_2 will be accommodated in the KOH solution. The presence of CO_2 in the KOH solution can prove the success of synthesizing potassium glyceroxide.

2-2- Transesterification of RBDPO

The transesterification process was performed in a three-neck flask connected to a condenser, which functions to reflux methanol back (batch process). To start the process, the RBDPO is preheated in a three-neck flask. Meanwhile, the DES K_2CO_3–Glycerol solution and the potassium glyceroxide-glycerol mixture were mixed into methanol (according to the orthogonal array). After the reaction temperatures reached 65 and 95 °C, a mixture of methanol and DES and a mixture of potassium glyceroxide–glycerol were fed into the three-neck flask. This reaction lasted 4 hours with constant stirring (450 and 600 rpm). The schematic diagram of lab-scale biodiesel production employed in this work is shown in Figure 1. It was adopted from the literature [33]. The resulting biodiesel and glycerol mixture was separated using a separatory funnel. The upper phase (biodiesel) will then be washed with distilled water until the water used for washing is clear. The biodiesel was heated for approximately 1 hour at 90 °C to evaporate the remaining water. The biodiesel quality was analyzed based on the acid number, methyl ester yield, free glycerol, total glycerol, and monoglyceride content. The transesterification process was carried out in one or two stages. Total glycerin (TG), as an indicator of biodiesel quality, which consists of free glycerol and mono-, di-, and tri-acylglycerol [34], was analyzed based on the solution test.

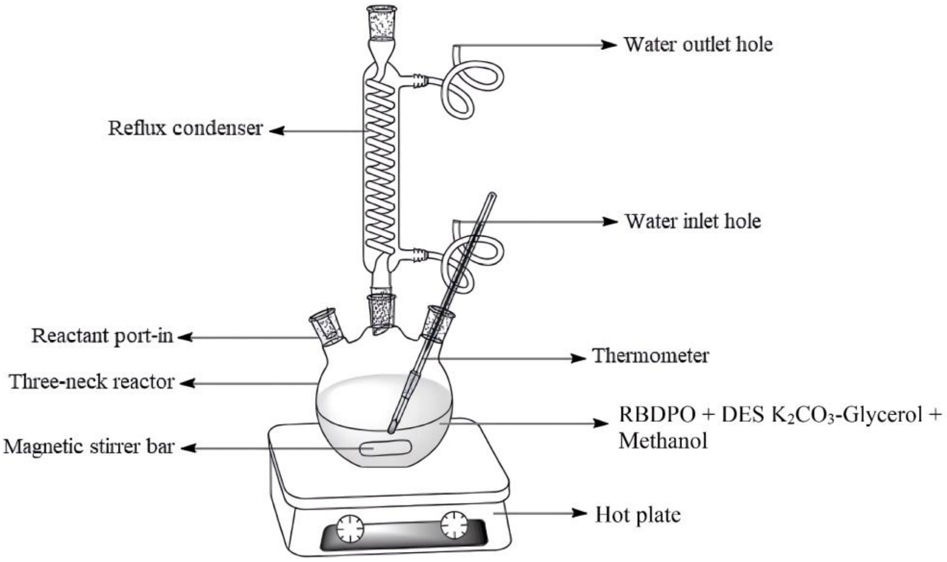

Figure 1. The biodiesel batch reactor setup

2-3- Experimental Design using the Taguchi Method

The Taguchi experimental design provides an efficient and systematic approach to determining optimal conditions [35]. The Taguchi method was assessed using the L9 orthogonal array exploited with the help of MINITAB software. This study used a Taguchi experimental design to determine the right and optimal composition by varying the control factor parameters in biodiesel production from RBDPO with variations in temperature, time, catalyst, and methanol ratio. The Taguchi method begins with designing the parameters for making biodiesel using a factorial orthogonal array design with 2 levels and 4 factors ($L2^4$).

The Taguchi approach used orthogonal arrays and variance analysis (ANOVA) for the experimental study. Using ANOVA, the influence of factors can be estimated, and with an orthogonal arrangement, a minimum number of experiments is required. In this method, the variability of the parameters is expressed by the signal-to-noise ratio (S/N), which represents the ratio of the desired result (signal) to the unwanted result (noise) [36]. This statistical method, the S/N ratio, measures the decrease in quality characteristics from the desired value. The maximum S/N ratio is optimal because the variability is inversely proportional to the S/N ratio. The Taguchi method has been used to design suitable experiments and quality control strategies under optimized conditions. The Taguchi experimental design is used to obtain information such as the main effect of the design parameters from a minimal number of experiments [37].

The advantage of the experimental design is that the number of trials is significantly reduced. Important decision variables that control and improve product or process performance can be identified. Optimal parameter settings can be found through this method. Qualitative estimates of the parameters can be made, and experimental errors can be estimated. Inferences regarding the influence of parameters on process characteristics can also be measured [35].

2-3-1- Determination of Control Factor and Noise

Factors in a product or process can be classified into control factors and noise factors. Control factors are process or design parameters within the manufacturer's control during production. The noise factor is a process or design parameter that is difficult or expensive to control during manufacturing. However, the noise factor may significantly impact the variation in quality characteristics [38]. The selection of factors for the experiment is based on Table 1.

Table 1. Control factor

Symbol	Control factor	Level 1	Level 2
A	Mixing speed (rpm)	450	650
B	Reaction time (h)	2	4
C	Methanol (wt%)	20	30
D	Catalyst (wt%)	4	6

2-3-2- Determination of Control Factor and Noise

An orthogonal array is a fractional factorial design. Orthogonal matrices are very efficient in obtaining relatively small amounts of data and can translate meaningful and clear conclusions. An orthogonal matrix determines the number of factors, or degrees of freedom (db), and the minimum level required by the inner array [39]. Then, choosing an orthogonal matrix that fits the experiment such that the degrees of freedom in the standard orthogonal matrix must be greater than or equal to the calculation of the degrees of freedom in the experiment, then use $L_{16}(2^4)$, where L is the Latin square design, 16 represents the number of rows or experiments, 2 states the number of levels, and 4 states the number of columns or factors. This study had 16 experiments, 4 factors, and 2 levels. The selected orthogonal arrays are summarized in Table 2.

Table 2. Taguchi experimental design with orthogonal array $L_{16}(2^4)$

Run	A	B	C	D
1	-	-	-	-
2	-	-	-	+
3	-	-	+	-
4	-	-	+	+
5	-	+	-	-
6	-	+	-	+
7	-	+	+	-
8	+	+	+	+
9	+	-	-	-
10	+	-	-	+
11	+	-	+	-
12	+	-	+	+
13	+	+	-	-
14	+	+	-	+
15	+	+	+	-
16	+	+	+	+

Furthermore, the Signal Noise Ratio (S/N) is determined to identify the factors influencing the experimental results. ANOVA is carried out to determine the effect on each input parameter from the experimental results to interpret the experimental data [40]. ANOVA is the second statistical approach used. ANOVA does not consider differences in variance between populations but differences in means. Generally, the principle of ANOVA lies in dividing the total variation in the factorial component relative to the equation or regression model and the residual components. The first component was tested concerning the second [41]. The factorial and residual components are mathematically represented by the mean square. Ultimately, the interest in using ANOVA is to aid in testing the absolute effect of the variation of a given factor [35]. In the analysis of variance, only one hypothesis is used (a two-way hypothesis) to determine whether there is a difference in the mean and not to determine which one is different [42].

3- Results and Discussion

In this analysis, Minitab 2020 software is used to calculate the Signal Noise Ratio SNR value and analyze the ANOVA variance to determine the quality of a section when the input variable changes [43]. First, this analysis assesses the randomization of factor values using an orthogonal matrix L_{16} (2^4) so that there are 16 experiments with 2 levels, and the average value is used as the initial value for each response. Then, after the randomization factor has been determined, it is continued by entering the lowest value of the sink mark and shrinkage. Then the Taguchi design analysis is carried out to determine the value of more minor is better or can be categorized as the smaller the quality characteristic value, the better by producing a signal-to-noise ratio (SNR) graph. After the Taguchi design analysis, ANOVA analysis is carried out by creating a table of variance analysis results with limits for F-value and P-value in the statistical table.

3-1- Determination of Signal-to-Noise Ratio (SNR)

This analysis is carried out to determine the quality characteristics using Smaller Is Better because it indicates that the smaller the parameter size, the better the quality. On the other hand, if using Large the Better, it will suggest that the larger the parameter, the better the quality will be, and the Nominal the Better, which means that the quality will be said to be good if it is close to the nominal (target) [44]. After selecting the orthogonal matrix and placing the factors in the matrix, the next step is to conduct an experiment based on the matrix. An outer array is performed to obtain a more accurate estimate of a factor's effect. The data obtained from the results of biodiesel testing on total glycerin are shown in Table 3.

Table 3. **Results for total glycerin (TG)**

Test matrix	Mixing speed (rpm)	Reaction time (h)	Methanol (wt%)	Catalyst (wt%)	TG Avg (%)
	A	B	C	D	
1	450	2	20	4	0.690
2	450	2	20	6	0.720
3	450	2	30	4	0.440
4	450	2	30	6	0.876
5	450	4	20	4	0.640
6	450	4	20	6	0.760
7	450	4	30	4	0.210
8	450	4	30	6	0.734
9	650	2	20	4	0.639
10	650	2	20	6	0.692
11	650	2	30	4	0.690
12	650	2	30	6	0.720
13	650	4	20	4	0.640
14	650	4	20	6	0.760
15	650	4	30	4	0.640
16	650	4	30	6	0.596

Furthermore, the level of process parameters was sorted by the most influential and presented in the main effect graph. The data from Table 4 was then interpreted into the main effect graph, as shown in Figure 2.

Table 4. **The influence of factors and levels on the average value of total glycerin**

Factor	Level		Deviation	Rank
	1	2		
A	0.6338	0.6721	0.0384	4
B	0.6834	0.6225	0.0609	3
C	0.6926	0.6132	0.0794	2
D	0.5736	0.7322	0.1586	1

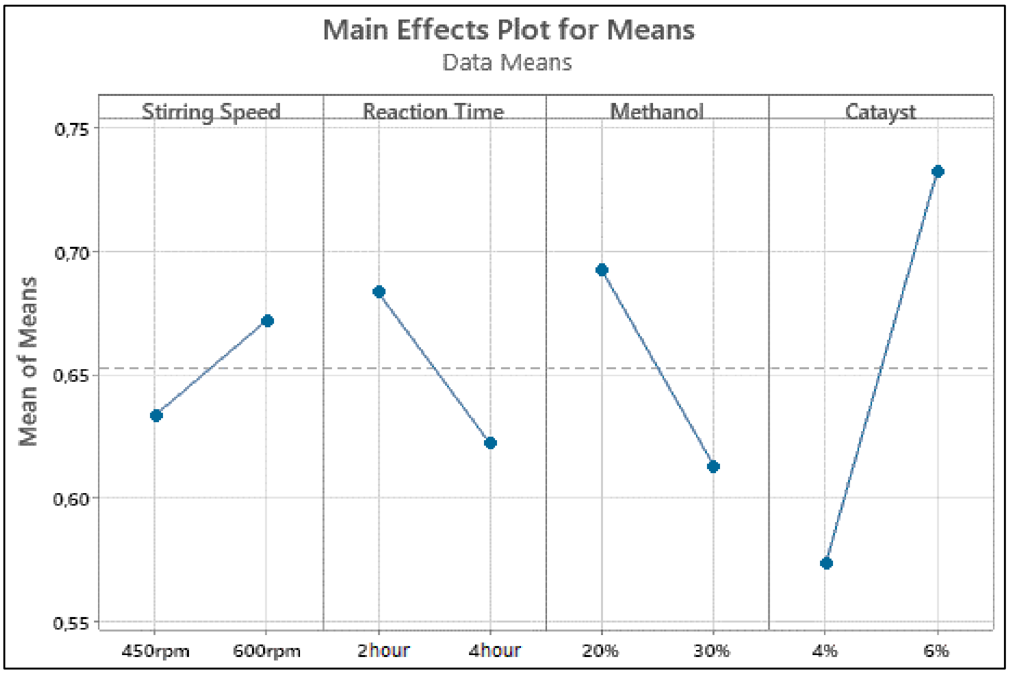

Figure 2. The influence of factors and levels on the average value of total glycerin

Figure 2 shows the order of the average value of the effect of each factor, from the largest to the smallest, on the total quality of glycerin. The average effect shows that the effect of the catalyst on total glycerin is of the first order. In other literature, the catalyst also ranks first as the most influential parameter in biodiesel production [45]. The effect of methanol on total glycerin is in the second order, and the effect of reaction time on total glycerin is in the third order. The influence of stirring speed is in the fourth. For product quality parameters, an analysis is carried out using the SNR method. The smaller is better, or smaller the response value, the better. The target value of the smaller is better. SNR has a non-negative value, and the value consists of zero to infinity (∞) where the desired defect value is zero. In this stage, although smaller is better, the ratio is defined in such a way that it can be continually transformed into a quality character: the bigger, the better. Therefore, the most optimal level is the level with the highest average SNR of each parameter [46].

The calculation of the effect of the SNR on the factors aims to reduce the average value of the largest influence by the average value of the smallest effect. From the influence of the SNR of each factor, the order of the influence of each factor can be seen in Figure 3.

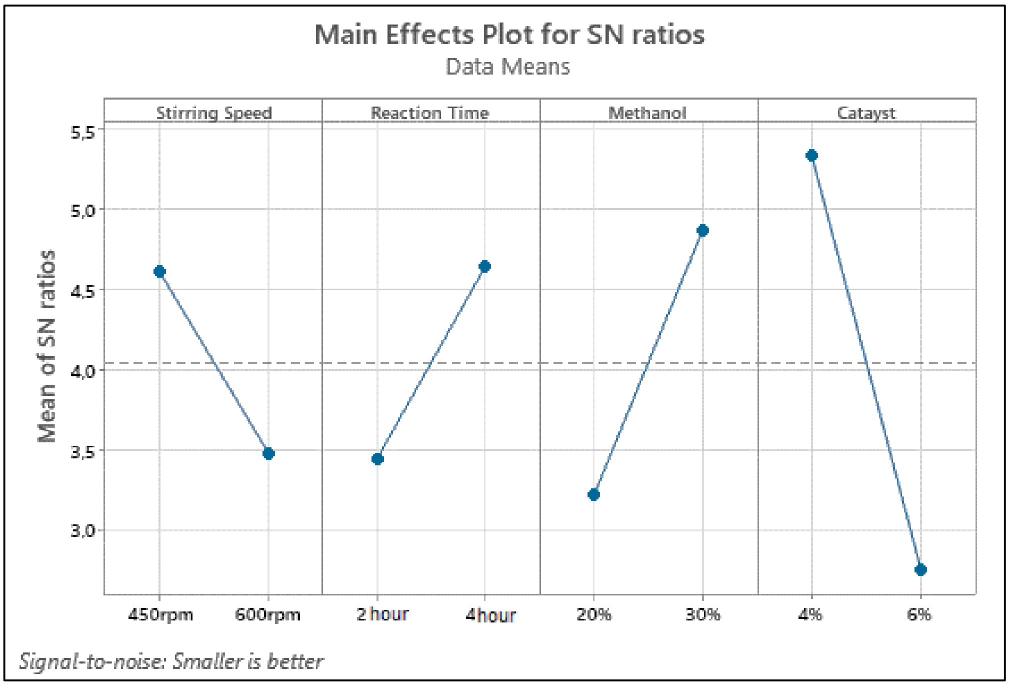

Figure 3. The effect of signal noise to ratio smaller is better

Table 5 shows the influence of SNR from level randomization on each process parameter by looking at the highest noise value. In the SNR response, the most optimum level was the catalyst at level 1 of 4%, methanol at level 1 of 30%, reaction time at level 2 of 4 hours, and stirring speed at level 1 of 450 rpm. From the results of the signal noise to smaller is better ratio, it can be concluded that the quality can be said to be optimal with the need for a level of influence with a minimum value. With this SNR analysis, it is known that the level of the process parameter has the most influence on the experimental results. So that the optimum proposed design for producing biodiesel DES K_2CO_3-Glycerol in the RBD-PO transesterification is obtained, namely A1B2C2D1. The TG concentration was affected by the catalyst concentration increasing from 4 wt% to 6 wt% as the number of active sites increased in this range. As can be observed in Figure 3, enhancing the catalyst weight will increase TG content. It will decrease biodiesel yield along with the increase in catalyst [47]. The high amount of catalyst impairs biodiesel production, which makes the reaction reversible [48]. Optimization of methanol weight to oil is also an important parameter. The TG content in biodiesel decreases with the increase of the methanol-to-oil ratio because of the improvisation of the transesterification reaction. Nevertheless, the biodiesel content will decrease with a further increase in methanol because the glycerol dissolves in excess methanol. Due to the polar nature of the hydroxyl groups in methanol, glycerol is challenging to separate. This result is somewhat different from other studies that obtained the methanol to oil molar ratio in the first rank as the most influential variable [49, 50]. To analyze and describe all variations in the parts studied, it is necessary to classify the experimental results according to the source of interpretation using the analysis of variance method.

Table 5. The effect of signal noise to ratio smaller is better

Factor	Level		Deviation	Rank
	1	2		
A	4.607	3.474	1.133	4
B	3.440	4.642	1.201	3
C	3.211	4.871	1.661	2
D	5.331	2.750	2.581	1

3-2-Analysis of Variance (ANOVA)

In the following analysis, an analysis of variance (ANOVA) was carried out to determine the effect of each factor on the resulting TG value. ANOVA is used as an analytical tool to test research hypotheses that assess whether there is a difference in means between groups. The final result of the ANOVA analysis is the value of the F-test or F-count. This F-value will be compared with the value in the F-table. In ANOVA, the so-called factors are independent variables. The independent variable is measured on a nonmetric or categorical scale, while the dependent variable is measured at least on an interval scale. The results of the analysis of variance from this study are summarized in Table 6. The ANOVA results were analyzed with the assumption that H_0: had no effect, accepted if, p-value > 0.05, and H_a: had to influence, accepted if, p-value < 0.05.

Table 6. The result of ANOVA

Source	DF	Adj. SS	Adj. MS	F-value	P-value
Mixing speed	1	0.005891	0.005891	0.34	0.572
Reaction time	1	0.014823	0.014823	0.85	0.376
Methanol	1	0.025202	0.025202	1.45	0.254
Catalyst	1	0.100648	0.100648	5.78	0.035
Error	11	0.191432	0.017403		
Total	15	0.337995			
S	0.131920				
R-sq	43.36%				
R-sq (adj)	22.77%				

Based on the results of the ANOVA test, the probability value (P-value) or significance of each test factor is obtained. The P-value for the stirring speed factor, reaction time, and methanol shows an average P-value greater than 0.05, so the assumption of H_0 is accepted, meaning that the three factors do not significantly influence the TG value.

In comparison, the probability value (P-value) for the catalyst factor shows a value of 0.035, which is smaller than 0.05, so the assumption of H_a is accepted, which means that the catalyst factor has a significant influence on the TG value.

In ANOVA, hypothesis testing is carried out using F-test statistics. Suppose the F-count is greater than the F-table. In that case, it is concluded that the simultaneous hypothesis accepts Ha, meaning a set of independent variables is proven to have a significant effect on the dependent variable. On the other hand, if the F-count is less than the F-table, then a set of independent variables is not proven to have a significant effect on the dependent variable or accept H_0. The F-table is calculated through Equations 1 and 2.

$$df1 = k - 1 \tag{1}$$

$$df2 = n - k \tag{2}$$

where k is the number of variables (independent + bound) and n = is the number of observations/samples forming the regression. The research shows that k = 4 and n = 16, so df1 and df2 can be determined from Equations 3 and 4.

$$df1 = k - 1 = 4 - 1 = 3 \tag{3}$$

$$df2 = n - k = 16 - 4 = 12 \tag{4}$$

If the test is carried out at 5%, then the F table value is 3.49. At N1 = 3 and N2 = 12, the F distribution percentage point for probability was 0.05. The results of the ANOVA in Table 5 show the factors of stirring speed, reaction time, and methanol produce a calculated F-value less than the F-table, so these factors are not proven to have a significant effect on the TG value or the assumption Ho is accepted. In comparison, the catalyst factor shows a calculated F-value of 5.78, which is greater than the table F-value. The assumption that H_a is accepted or that the catalyst factor is proven to have a significant effect on the TG value. Because this study uses more than two independent variables or more than two factors, it is better to use an adjusted R square than an R square to find out the influence of the test factor on the TG value. The adjusted R square value of 22.77% (Table 6) means that the research factor variable influences 22.77% of the TG value.

4- Conclusion

Taguchi's experimental design revealed that selecting the optimal factor level is essential to maximizing biodiesel production with a low total glycerol concentration. The optimal parameter in biodiesel production from RBDPO using DES K_2CO_3-Glycerol catalyst was successfully determined using the L16 Taguchi technique to minimize the number of experiments. The lowest value of total glycerol in biodiesel was 0.210%. The optimal operating conditions to produce biodiesel are 30 wt% methanol and a catalyst loading of 4 wt% at 95 °C for a reaction time of 4 h with the mixing of 450 rpm. According to the ANOVA results, the research factor variables have an influence of 22.77% on the total glycerol value of biodiesel products.

5- Declarations

5-1- Author Contributions

Conceptualization, S.A. and W.A.; methodology, W.A.; software, W.A.; validation, L.N.K., S.A., and W.A.; formal analysis, F.H.; investigation, C.R.; resources, F.H.; data curation, C.R.; writing—original draft preparation, W.A.; writing—review and editing, C.R.; visualization, W.A.; supervision, S.A.; project administration, L.N.K.; funding acquisition, S.A. All authors have read and agreed to the published version of the manuscript.

5-2- Data Availability Statement

The data presented in this study are available in the article.

5-3- Funding and Acknowledgements

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5-4- Institutional Review Board Statement

Not applicable.

5-5- Informed Consent Statement

Not applicable.

5-6- Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancies have been completely observed by the authors.

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