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Purification Optimization of Biodiesel Derived from Glyceroxide-Catalyzed Transesterification

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ABSTRACT. The washing stage is a critical step in biodiesel purification because it determines the total glycerol and water contents, which directly affect final fuel quality. This study aims to optimize the washing conditions of biodiesel produced from palm oil transesterification using sodium glyceroxide catalyst and to compare its characteristics with biodiesel produced using NaOH catalyst. Optimization was carried out using the Response Surface Methodology (RSM) with a with a Central Composite Design (CCD), in which the washing water to biodiesel ratio and washing temperature were used as independent variables, while total glycerol content and biodiesel water content were used as response variables. ANOVA analysis indicated that the quadratic model was statistically significant for both responses ($p<0.05$), with the washing water to biodiesel ratio being the most dominant factor. The surface and contour plots revealed non-linear behavior at each optimization point. The optimum washing condition were achieved at a water to biodiesel ratio of 1:1.9 and a washing temperature of 59ºC. The washing results demonstrated that biodiesel produced using sodium glyceroxide catalyst had lower total glycerol content and water content compared to biodiesel using NaOH catalyst, with reductions of 14.28% and 16.30%, respectively. These findings indicate that the use of sodium glyceroxide catalyst produces biodiesel that is easier to purify through the washing process and has better overall quality compared to biodiesel produced with NaOH catalyst.

1. INTRODUCTION

Fuel plays a vital role in supporting human activities, yet the heavy reliance on fossil fuels poses a significant challenge the limited availability of petroleum reserves, which are projected to last only about 11 more years[1]. Ensuring long-term energy security in Indonesia, therefore, requires strategic efforts to prevent fuel shortages and price instability. Among the various solutions, renewable alternative fuels have gained strong attention as a sustainable option.

Biodiesel is a renewable and environmentally friendly fuel produced through the transesterification of triglycerides with alcohol, typically in the presence of a catalyst. Catalysts are essential for accelerating reactions under mild conditions (e.g., at low temperatures and atmospheric pressure)[2]. Common catalysts are strong bases derived from first-row metals, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH)[3]. However, these hydroxide catalysts can promote side reactions, such as saponification, particularly because water is formed when hydroxides react with methanol during alkoxide formation[4]. The water produced in this reaction complicates biodiesel separation in the final stages, particularly during purification (washing)[5].

Sodium glyceroxide has emerged as a promising alternative catalyst because it can be synthesized from glycerol, a major by-product of biodiesel production, thereby supporting circular and sustainable processing. Sodium is also more economical than potassium, and glycerol is considered more environmentally benign compared to methanol derived from non-renewable natural gas sources[6]. These advantages position sodium glyceroxide as an innovative, more sustainable catalyst for biodiesel production.

Crude biodiesel must be purified to meet quality specifications SNI 7182:2024[7]. Purification is essential to remove contaminants that may cause operational issues[8]. For example, residual glycerol can lead to injector fouling[9], whereas excess alcohol reduces the flash point and increases corrosion risk for metals such as aluminum

and zinc[10]. Biodiesel purification can generally be performed using two main methods, wet washing and dry washing. Wet washing uses water as a cleaning agent to remove impurities, such as glycerol, residual alcohol, and soap, from biodiesel[11]. This method is simple and effective but requires large volumes of water and generates wastewater if not properly managed. In contrast, dry washing uses adsorbents such as magnesol, silica gel, or ion-exchange resins to remove impurities without water, but it is more costly and less efficient in achieving the same purity as wet washing. Therefore, optimization of the wet washing process remains essential to improve biodiesel quality while minimizing water use and waste generation.

Water washing remains one of the most commonly applied purification methods due to its ability to remove residual alcohol, catalyst, water, and glycerol[12]. However, washing conditions strongly influence the final biodiesel quality and product yield, and studies in the past five years highlight the need for optimized washing strategies to ensure consistent fuel quality.

While previous research has investigated various washing techniques and purification aids, only a limited number of studies have focused on optimizing washing conditions specifically for biodiesel produced using sodium glyceroxide catalysts. Moreover, the combined effects of water-to-biodiesel ratio and washing temperature on critical quality parameters such as total glycerol and water content remain underexplored. Therefore, this research aims to optimize biodiesel washing conditions using Response Surface Methodology (RSM) and to compare the purification performance of sodium glyceroxide and NaOH catalysts under optimal conditions. The objectives of this study are to determine the optimal washing parameters and to evaluate the resulting biodiesel quality based on total glycerol and water content.

2. MATERIALS AND METHODS

2.1 Materials

The materials used for sodium glyceroxide catalyst synthesis included sodium hydroxide (NaOH, Merck, ≥99%) and glycerol (Merck, ≥99.5%). Biodiesel was produced through the transesterification of palm oil using methanol (Merck, ≥99.8%) as the alcohol and sodium glyceroxide as the catalyst. Purification of the crude biodiesel was performed by water washing with distilled water until the wash water reached neutral pH. Total glycerol analysis employed periodic acid ($HIO_4.2H_2O$, Merck, ≥99%), glacial acetic acid, potassium dichromate ($K_2Cr_2O_7$), hydrochloric acid (HCl), starch indicator, sodium thiosulfate ($Na_2S_2O_3.5H_2O$), potassium iodide (KI), chloroform ($CHCl_3$), potassium hydroxide (KOH), and 95% ethanol, all of analytical grade.

2.2 Equipment

The main equipment used in this study included a 500 mL three-neck flask, a reflux condenser, a magnetic stirrer with heating, a thermometer, and a separatory funnel. Total glycerol was analyzed using iodometric titration equipment, while water content was measured with a Karl Fischer Titrator (Mettler Toledo DL39).

2.3 Methods

2.3.1 Preparation of Sodium Glyceroxide Catalyst

Sodium glyceroxide catalyst was synthesized by reacting NaOH and glycerol at a molar ratio of 3:1 and a temperature of 120–145°C [13]. After the reaction was completed, the product was allowed to cool for several minutes, covered with plastic wrap, and stored for use in the transesterification process on the following day.

2.3.2 Transesterification Process

The transesterification process was carried out using a methanol-to-oil molar ratio of 9:1, 0.6 wt% sodium glyceroxide catalyst, a reaction temperature of 60°C, and a reaction time of 60 minutes. After completion, the mixture was allowed to settle for phase separation. The upper layer containing crude biodiesel was collected for the washing stage, while the lower layer consisted of glycerol. The same procedure was performed using NaOH as a comparison catalyst.

2.3.3 Washing Process

Crude biodiesel was washed using water at various water-to-biodiesel ratios (1:1, 1:1.5, and 1:2 v/w) and washing temperatures (50, 60, and 70°C). After mixing, the mixture was allowed to settle until two layers were formed: an upper layer containing methyl esters and a lower layer containing water, glycerol, and impurities. The lower layer was discarded, while the upper layer was collected and washed again with fresh water under the same

ratio and temperature conditions. Washing was repeated until the wash water clear and neutral pH. To improve phase separation, the washed biodiesel was subsequently centrifuged.

In this study, optimization focused on two variables: water-to-biodiesel ratio and washing temperature, because both have been reported in previous studies to be the most influential factors affecting the removal efficiency of glycerol and water. Other variables, such as washing time, number of washing cycles, agitation intensity, and centrifugation speed, were kept constant based on preliminary laboratory trials that identified their minor contribution within the studied range. This approach was selected to minimize experimental complexity and to ensure clearer interpretation of variable interactions in the RSM model.

Table 1. Experimental Variables for Optimization of the Washing Process

Run Order	Water to Biodiesel Ratio	Washing Temperature (°C)
1	1:1	50
2	1:2	50
3	1:1	70
4	1:2	70
5	1:0.8	60
6	1:2.2	60
7	1:1.5	46
8	1:1.5	74
9	1:1.5	60
10	1:1.5	60
11	1:1.5	60
12	1:1.5	60
13	1:1.5	60

2.3.4 Analysis of Product Biodiesel

The quality of the washed biodiesel was evaluated based on two key parameters in accordance with the Indonesian National Standard (SNI 7182, 2024), total glycerol content (≤ 0.02 wt%) and water content (≤ 0.05%). Water content was determined using Karl Fischer titration[14]. Total glycerol content was measured using iodometric titration[15]. The calculation of total glycerol content was performed using Equation 1.

$$\text{Glisol}_{\text{tot}} = \frac{2{,}302 \times (\text{B}-\text{C}) \times \text{N}}{\text{W}} \quad (1)$$

Where *B* refers to the volume of sodium thiosulfate solution used in the blank titration (mL), while *C* is the volume of sodium thiosulfate required for the sample titration (mL). *N* represents the exact normality of the sodium thiosulfate solution, and *m* denotes the mass of the biodiesel sample (g).

3. RESULTS AND DISCUSSION

3.1 Analysis of Variance (ANOVA)

Model evaluation was performed through analysis of variance (ANOVA) to determine the significance of the independent variables on the responses. Table 2 presents the F-values and P-values obtained from the analysis using Minitab software.

Based on Table 2, the ANOVA results indicate that the developed quadratic model is statistically significant for both observed responses. The model p-values were 0.020 for total glycerol and 0.001 for water content, both below the 95% confidence level ($p < 0.05$), indicating that the model adequately represents the relationship between the water-to-biodiesel ratio (A) and washing temperature (B) in the responses. For total glycerol, the linear effect was significant ($p = 0.021$), with A being the dominant factor ($p = 0.008$), whereas B showed no significant linear effect ($p = 0.372$). The quadratic effect was also significant ($p = 0.020$), particularly for A^2 ($p = 0.007$), suggesting a non-linear relationship, whereas the interaction term A×B was not significant ($p = 0.447$), indicating that the factors act independently. For water content, both linear ($p = 0.026$) and quadratic ($p = 0.000$) effects were significant, with A being the most influential factor ($p = 0.009$), whereas B had no significant linear effect ($p = 0.892$). The quadratic effects of A^2 ($p = 0.000$) and B^2 ($p = 0.001$) indicated the presence of optimum

points for both factors, while the interaction term (A×B) was not significant (p = 0.258), confirming the independent action of the factors.

Table 2. ANOVA Results for Total Glycerol and Water Content Responses

Source	Total Glycerol		Water Content	
	F-Value	P-Value	F-Value	P-Value
Model	5,81	0,020	18,22	0,001
Linear	7,08	0,021	6,45	0,026
A	13,24	0,008	12,88	0,009
B	0,91	0,372	0,02	0,892
Square	7,13	0,020	38,33	0,000
A*A	13,92	0,007	59,11	0,000
B*B	1,14	0,322	26,59	0,001
2-Way Interaction	0,65	0,447	1,52	0,258
A*B	0,65	0,447	1,52	0,258

Note: A = Water-to-Biodiesel Ratio, B = Washing Temperature (°C)

The model's ability to explain the variability of the responses was evaluated using the coefficient of determination (R^2). For water content, R^2 = 92.86%, indicating that the model accounts for most of the observed variation; for total glycerol, R^2 = 80.59%, indicating satisfactory data explanation. To further illustrate the relative contributions of each factor, Pareto charts were constructed (Figures 1 and 2). These charts display the standardized effects of each factor and interaction, with a vertical line indicating the 95% significance threshold. For total glycerol, A^2 and A were dominant, B was retained in the model to maintain the structure of the response surface and support multi-response optimization. For water content, A^2, B^2, and A were dominant, reflecting the non-linear relationship and the presence of optimum points. Although the linear effect of B was insignificant, it was retained because the quadratic effect (B^2) was significant, which is important for shaping the response surface and identifying optimal washing conditions.

Overall, the ANOVA and Pareto chart analyses indicate that the water-to-biodiesel ratio is the primary factor controlling both total glycerol and water content, while washing temperature contributes as a secondary factor through its quadratic effect, supporting the determination of optimum process conditions.

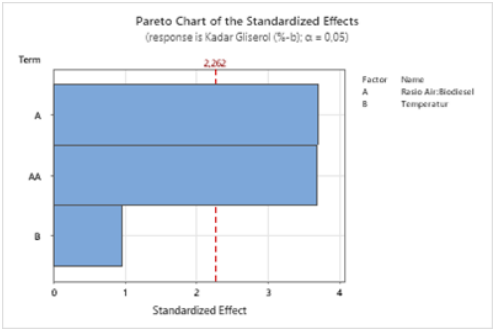

Figure 1. Pareto chart of total glycerol content in biodiesel.

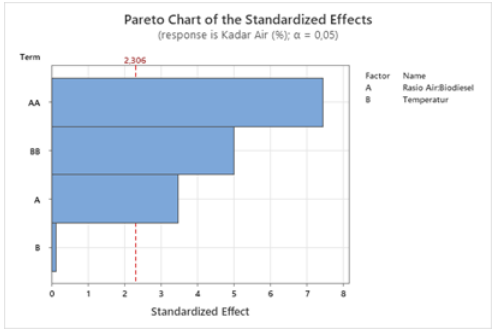

Figure 2. Pareto chart of water content in biodiesel.

3.2 Response Surface Methodology (RSM) Analysis

The optimization of the biodiesel washing process was conducted using Response Surface Methodology (RSM) with a Central Composite Design (CCD). This method was used to model the relationship between two key process variables, water to biodiesel ratio and washing temperature, and two quality responses, total glycerol content (wt%) and water content (%). The CCD approach was selected due to its flexibility in exploring variable interactions beyond the center point without exceeding practical operating limits[16]. Using axial points positioned outside the factor boundaries ($\alpha > 1$) enables the model to capture quadratic effects more accurately by expanding the range of variation in the experimental data. The CCD results showed that total glycerol content ranged from 0.133 to 0.180%, wh water content ed 0.135 to 0.154%. These variations indicate that changes in water ratio and

washing temperature influence the efficiency of glycerol and water removal from biodiesel.

Visualization of the Response Surface Methodology (RSM) in Figures 3 and 4 shows the relationship between water to biodiesel ratio and washing temperature on total glycerol and water content. Both responses quadratic surfaces with clear minim, indicating optimal washing conditions. The optimum conditions were achieved at a water-to-biodiesel ratio of 1:1.9 and a temperature of 59 ºC, resulting in a total glycerol content of 0.154% and a water content of 0.144%. Increasing the water to biodiesel ratio initially reduced the glycerol content due to more efficient glycerol removal; however, at higher ratios, the glycerol content increased again. This phenomenon is attributed to emulsion formation, which traps part of the glycerol in the biodiesel phase, observed visually as a turbid interfacial layer and slower phase separation. In addition, imperfect phase separation may contribute to the in in glycerol. The effect of washing temperature exhibited a similar quadratic trend. Raising the temperature to 59 °C improved mass transfer and reduced glycerol content, owing to decreased viscosity and enhanced contact between biodiesel and water. In contrast, temperatures above 65 °C decreased washing efficiency, likely due to partial water evaporation and the potential for reverse reactions between methyl esters and glycerol[10].

A similar trend was observed for water content. Increasing the water ratio and washing temperature up to 59 ºC decreased the water content by enhancing the solvent capacity of water and accelerating phase separation. However, at higher temperatures or with insufficient water, phase separation was incomplete, resulting in residual water in the biodiesel. Although the optimization successfully reduced both glycerol and water contents, the water content of the resulting biodiesel still reached 0.144%, indicating that most impurities had been removed, but the level is still slightly above the maximum limit of 0.05% specified in SNI 7182:2024. This suggests that, although the washing effectively lowers glycerol and water contents, an additional drying step is still required for the biodiesel to fully meet the quality standard.

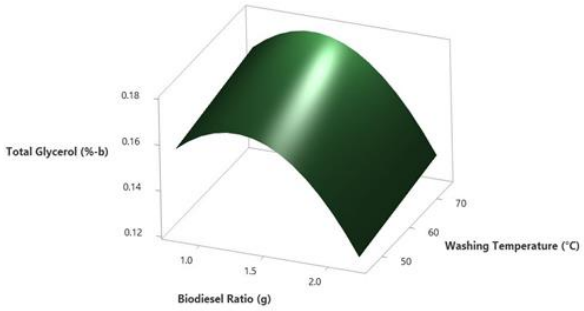

Figure 3. Surface and contour plots of biodiesel total glycerol content as a function of the water to biodiesel ratio and washing temperature

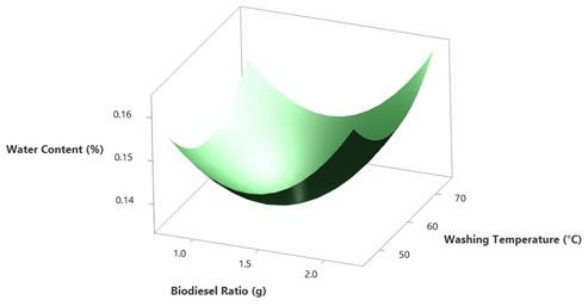

Figure 4. Surface and contour plots of biodiesel water content as a function of the water to biodiesel ratio and washing temperature

3.3 Comparison of the Performance of Sodium Glyceroxide and NaOH Catalysts

The comparison of catalyst performance was conducted to assess the effectiveness of sodium glyceroxide relative to NaOH in producing high-quality biodiesel in accordance with the quality requirements specified in SNI 7182:2024, EN 14214, and ASTM D6751, particularly with respect to total glycerol, water content, and methyl ester content. The transesterification reactions using both catalysts were performed under identical operating conditions. The resulting biodiesel was subsequently purified by washing under the optimal conditions determined by RSM, namely a water-to-biodiesel ratio of 1:1.9 and a temperature of 59 °C. The results indicate that sodium glyceroxide produced biodiesel of superior quality compared to NaOH, as presented in Table 3.

Table 3. Comparison of Biodiesel Produced Using Sodium Glyceroxide and NaOH Catalysts

Parameter	Catalysts		SNI	EN	ASTM
	Sodium Glyceroxide	NaOH	7182:2024	14214	D6751
Total glycerol (%-b)	0.144	0.168	0.24	0.25	0.24
Water content (%)	0.154	0.184	0.05	0.05	0.05
Methyl ester content (%-b)	99.506	99.417	96.50	96.50	-

As shown in Table 3, biodiesel washed using the sodium glyceroxide catalyst exhibited a water content of 0.144% and a total glycerol content of 0.154%, whereas biodiesel produced using the NaOH catalyst showed higher values of 0.168% and 0.184%, respectively. These results indicate that the use of sodium glyceroxide reduced the water content by 14.28% and the total glycerol content by 16.30% compared to NaOH under identical process conditions. In addition, the methyl ester content of biodiesel produced with sodium glyceroxide reached 99.506%, which is slightly higher than that obtained with NaOH 99.417%, indicating a more complete conversion of triglycerides.

The reduction in water and total glycerol contents observed for biodiesel produced using sodium glyceroxide can be explained from a chemical perspective. Sodium glyceroxide is formed by reacting glycerol with sodium hydroxide, yielding a more stable and selective –ONa active group toward triglycerides. Due to its stronger basicity and more homogeneous nature, the transesterification reaction proceeds more efficiently, resulting in lower soap formation compared to the use of pure NaOH, as also reported in previous studies. Lower soap content facilitates the washing process by reducing emulsion formation, thereby enhancing biodiesel purification efficiency. Moreover, biodiesel produced using sodium glyceroxide exhibits easier glycerol phase separation, as residual glycerol is largely bound in the form of glyceroxide salts that are insoluble in the methyl ester phase. This behavior directly contributes to the lower total glycerol content observed in the final biodiesel, consistent with findings reported in the literature on glyceroxide-based catalysts.

4. CONCLUSION

This study confirms that the washing process plays a crucial role in improving biodiesel quality by reducing total glycerol and water content. The application of RSM using a CCD design successfully modeled the effects of the water to biodiesel ratio and washing temperature, and identified the optimum process conditions. The water-to-biodiesel ratio was found to be the most influential factor, while washing temperature showed significant effects through its quadratic component. The optimum conditions were 1:1.9 and 59 ^{o}C, producing biodiesel with a total glycerol content of 0.154% and a water content of 0.144%.

A comparison of catalyst performance indicates that biodiesel produced using sodium glyceroxide exhibits better final quality after washing than biodiesel produced using NaOH, as reflected by lower total glycerol and water contents under identical washing conditions. This behavior is attributed to lower soap formation and a reduced tendency to form emulsion during the washing stage, thereby facilitating more effective impurity removal. These results demonstrate that sodium glyceroxide improves the washability of biodiesel rather than acting directly as a purification agent. Overall, sodium glyceroxide shows strong potential as a more efficient and sustainable catalyst for biodiesel production. Future work may focus on process scale-up, economic feasibility analysis, and integration of sodium glyceroxide into sustainable biodiesel production systems.

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