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Original Research Article

Response Surface Methodology for Optimization of Free Fatty Acid Glycerolysis in Crude Palm Oil Using Crude Glycerol

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ABSTRACT

The free fatty acid (FFA) content in crude palm oil (CPO) must stay within acceptable limits to meet product quality standards. As a result, managing and reducing FFA levels is crucial for preserving CPO quality. This study aimed to evaluate the impact of glycerolysis on reducing FFA levels in CPO under Response Surface Methodology (RSM). Four key operational parameters affecting FFA reduction were examined: reaction temperature (65–85°C), stirring speed (150–250 rpm), molar ratio of glycerol to FFA (3:1–5:1), and catalyst loading (0.5–1 wt.%). A statistical analysis employing analysis of variance (ANOVA) was conducted to evaluate the importance of each factor. The optimal conditions for glycerolysis, which successfully reduced the FFA content in CPO from 6.15% to 0.24%, were identified as a reaction temperature of 75.17°C, stirring speed of 235.06 rpm, glycerol to FFA molar ratio of 3.57:1, and catalyst loading of 0.98 wt.%, with a reaction duration of 90 minutes. The statistical models demonstrated high significance, with a p-value <0.0001 and a coefficient of determination (R^2) of 0.95. The reliability of these models was further validated through experimental trials under the optimal conditions, yielding an actual FFA content of 0.27%, confirming the model's predictive accuracy. This study highlights the utilization of crude glycerol for the reduction of FFA levels through glycerolysis reactions. The results of this study are expected to serve as an alternative to the esterification processes that have been applied in the industry to lower FFA levels in palm oil.

Keywords: Crude palm oil, Free fatty acid, Glycerolysis, Optimization, Response surface methodology

Introduction

Biodiesel has garnered significant attention as a sustainable alternative to fossil fuels due to its renewability, biodegradability, and lower environmental impact. Derived primarily from triglycerides in vegetable oils and animal fats, biodiesel can be seamlessly integrated into existing diesel engines without major modifications.[1] Among the various feedstocks available, palm oil has emerged as a leading source due to its high oil yield per hectare and cost-effectiveness compared to other vegetable oils such as soybean, sunflower, and rapeseed. Crude palm oil (CPO) is obtained through an extraction process from oil palm fruits, which involves stages such as steaming, pressing, and refining to produce oil rich in triglycerides. However, the presence of high free fatty acid (FFA) content in CPO presents a significant challenge for biodiesel production, as it interferes with the transesterification process, leading to soap formation and reduced biodiesel yield.[2]

Traditional biodiesel production relies on transesterification, a chemical reaction between triglycerides and simple alcohols (e.g., methanol or ethanol) in the presence of a catalyst, typically alkaline-based. However, high FFA content in feedstocks (>0.5%) triggers unwanted saponification reactions, complicating biodiesel separation and purification.[3]

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To mitigate this issue, esterification and glycerolysis have been explored as pre-treatment methods. Esterification utilizes acid catalysts and excess alcohol to convert FFAs into esters, but it requires high temperatures and generates water as a by-product, which can inhibit the reaction and necessitate additional separation steps.[4] In contrast, glycerolysis offers an alternative by directly converting FFAs back into mono-, di-, and triacylglycerols using glycerol, thus avoiding alcohol-related drawbacks and reducing acidity without significant side reactions.[5] Moreover, glycerolysis can utilize crude glycerol, a low-cost by-product of biodiesel production, making it an economically viable and sustainable process.[6] Glycerolysis reactions can be catalyzed using homogeneous and heterogeneous catalysts, including alkaline catalysts such as potassium hydroxide (KOH), sodium hydroxide (NaOH), and sodium silicate (Na_2SiO_3), as well as metal oxides like calcium oxide (CaO).[7-10] Alkaline catalysts are advantageous due to their high reaction rates and non-corrosive properties, but they require precise control to avoid soap formation.[11] Meanwhile, metal oxide catalysts offer reusability and greater stability but often require longer reaction times and higher temperatures.[12] The selection of an appropriate catalyst is crucial for ensuring an efficient and cost-effective glycerolysis process.

Despite its advantages, glycerolysis remains underexplored, particularly concerning its optimal reaction parameters for effective FFA reduction in CPO. Additionally, the direct utilization of crude glycerol without purification has not been reported. Previous studies have primarily focused on heterogeneous catalysts such as calcium oxide (CaO) derived from eggshells and low-temperature glycerolysis of jatropha oil.[13,14] Meanwhile, Binhayeeding et al.,[15] utilized crude glycerol, a biodiesel by-product, along with commercial lipase as a catalyst for glyceride production. However, limited research has systematically optimized the

critical operational variables— glycerol to FFA molar ratio, temperature, catalyst loading, and stirring speed—using advanced statistical approaches such as response surface methodology (RSM). RSM is a statistical and mathematical tool used to model and optimize complex processes by analyzing the effects of multiple independent variables simultaneously.[16] Central Composite Design (CCD) Method under under RSM is a structured method used for designing and enhancing processes. It aids in determining the significance of various factors that affect a given process.[17,18] This method helps determine the optimal conditions for maximizing FFA reduction while minimizing resource consumption and processing costs, making it highly suitable for refining biodiesel pre-treatment techniques. Furthermore, RSM allows for the evaluation of interaction effects between variables, thereby enhancing predictive accuracy and experimental efficiency. Additionally, studies assessing the economic feasibility and industrial scalability of glycerolysis remain scarce. Given these gaps, this study aims to optimize the glycerolysis process for FFA reduction in CPO by employing RSM. By systematically investigating key reaction parameters, this research seeks to enhance process efficiency and establish glycerolysis as a practical pre-treatment strategy for high-FFA feedstocks in biodiesel production. The findings are expected to contribute to the development of more sustainable and cost-effective biodiesel manufacturing techniques.

Materials and Methods

Materials

CPO was sourced from a local palm oil mill in Nagan Raya, Aceh, Indonesia (around 5° N, 96° E). The samples were gathered during the period of October–November 2022. Crude glycerol, a by-product of biodiesel transesterification process, was used as the glycerolysis agent. **Table 1** provides an overview of the physicochemical characteristics of CPO and the crude glycerol utilized in the study. Potassium hydroxide (99%) and analytical-grade phosphoric acid (85%) were purchased from Merck (Germany).

Experimental Design

A three-level, four-factor central composite design (CCD) within the response surface methodology (RSM) framework was implemented using Design Expert 13.0 (Stat-Ease Inc., Minneapolis). Key variables in the glycerolysis process, such as catalyst loading, stirring speed, glycerol to oil molar ratio, and reaction temperature play a crucial role in minimizing FFA content.[16] A total of 30 randomized experiments were conducted to evaluate FFA content across varying conditions, including reaction temperature (A: 65–85°C), stirring speed (B: 150–250 rpm), molar ratio of glycerol to FFA (C: 3:1–5:1), and catalyst loading (D: 0.5–1 wt.% of oil). The experimental design for glycerolysis in high-FFA CPO is summarized in **Table 2**. A total of 30 runs were conducted, and the actual and predicted values for FFA reduction are shown in **Table 3**.

Glycerolysis Procedure

Glycerolysis was performed following the procedure outlined by Suriaini et al.[7] Initially, CPO was degummed using phosphoric acid solution (1.5% v/v) to remove phospholipids. The glycerolysis reaction was conducted in a three-neck round-bottom flask, fitted with a condenser, magnetic stirrer, and thermometer. The crude glycerol and potassium hydroxide catalyst were mixed and stirred to form a potassium glyceroxide solution. The preheated CPO was then combined with the glycerol solution, and the reaction mixture was maintained at the desired temperature and stirring speed for 90 minutes. After completion, samples were collected for FFA analysis.

Analysis of the samples

The glycerolysis reaction was assessed by measuring the FFA levels across different experimental conditions. The FFA content in the glycerolysis product was determined using the acid-base titration method, as described in Equation (1).[5]

$$\text{FFA}\ (\%) = \frac{\text{m}\times\text{N}\times\text{Mr}}{\text{V}\times 1000} \times 100 \qquad (1)$$

where m represents oil mass (g), N denotes KOH concentration as the titrant (N), Mr is the relative molecular mass of palmitic acid (256 g/mol), and V refers to titrant volume (mL).

Statistical Analysis

The response surface regression model used a second-order polynomial equation of Eq. (2) to describe the relationship between independent variables and FFA reduction.

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

where Y represents the predicted response (%FFA), n is the number of variables examined and optimized during the experiment, β_0 is a constant, β_i, β_{ii}, β_{ij} are the coefficients of regression, and X_i, X_j are the uncoded independent variables. Analysis of variance (ANOVA) was performed to determine the statistical significance of the independent variables and their interactions. The model's goodness-of-fit was evaluated through the coefficient of determination (R^2), adjusted R^2, and predictive R^2 values. Additionally, experimental validation was carried out under optimized conditions to confirm the model's accuracy.

Results and Discussion

Optimization of the glycerolysis process

The optimization parameters were systematically adjusted to minimize the FFA content in CPO. The quadratic model was selected due to its ability to capture complex interactions between variables. Equation (3) represents the glycerolysis reaction model for FFA reduction derived from actual experimental data.

$$\begin{aligned} FFA\ (\%) = {} & 1.15 - 0.27A - 0.12B - 0.39C - 0.10D + 0.11AB + \\ & 0.19AC - 0.06AD + 0.11BC + 0.04BD + 0.17CD + \\ & 1.20A^2 - 0.57B^2 - 0.17C^2 - 0.59D^2 \end{aligned} \qquad (3)$$

The ANOVA results and the model's fit to the quadratic equation are summarized in **Table 4**, detailing the impact of each variable and their interactions on the response. The model's *F*-value of 21.75 confirms its statistical significance. With a *p*-value of less than 0.05, the model is deemed highly reliable. The analysis indicates that parameters A, B, C, D, along with interaction terms AB, AC, BC, CD, and quadratic terms A^2, B^2, D^2, are statistically significant. In contrast, the terms AD, BD, and C^2 were found to be insignificant in influencing the response. The regression coefficient (R^2) of the model is 0.9531 (**Table 5**), indicating a strong fit. The high predicted R^2 value (0.8244) suggests that the model exhibits high precision. As R^2 approaches unity, the model more accurately represents the experimental data. The adjusted coefficient of determination (Adj. R^2) is also robust at 0.9092, further confirming the model's reliability. The proximity of Adj. R^2 to the predicted R^2 value (a difference of not more than 0.2 is recommended) reinforces its predictive capability.[19] Additionally, the low coefficient of variation (CV) confirms the consistency and dependability of the model's results.

Effect of process parameters on the FFA reduction

Three-dimensional contour plots were utilized to visualize and assess the combined effects of various parameters on FFA reduction (**Figures 1**–**3**). **Figure 1(a)** illustrates the interaction between temperature and stirring speed in reducing FFA levels in CPO, with a molar ratio of glycerol to FFA of 4:1 and catalyst loading of 0.75 wt.%. Temperature proved to be a key factor in enhancing the glycerolysis reaction rate. Higher temperatures improved mass transfer and increased the solubility of glycerol in CPO, enhancing the conversion of FFAs. Elevated temperatures also impact the physical characteristics of the reaction mixture, particularly interfacial tension and viscosity, which play a crucial role in facilitating reactant mixing.[20] However, excessive temperatures (>75°C) resulted in marginal gains and increased the risk of undesirable thermal degradation. The interaction between temperature and stirring speed significantly influenced FFA reduction. At 65°C, increasing the stirring speed from 150 to 250 rpm caused a slight reduction in FFA content (from 2.28% to 1.83%) due to enhanced contact between the reactants.[7] A similar result trend was also reported by Hiranlordsanti et al.[21] Reducing FFA levels further may be possible by increasing the stirring speed beyond 250 rpm. However, the impact of higher stirring speeds could not be evaluated due to constraints in the experimental setup.

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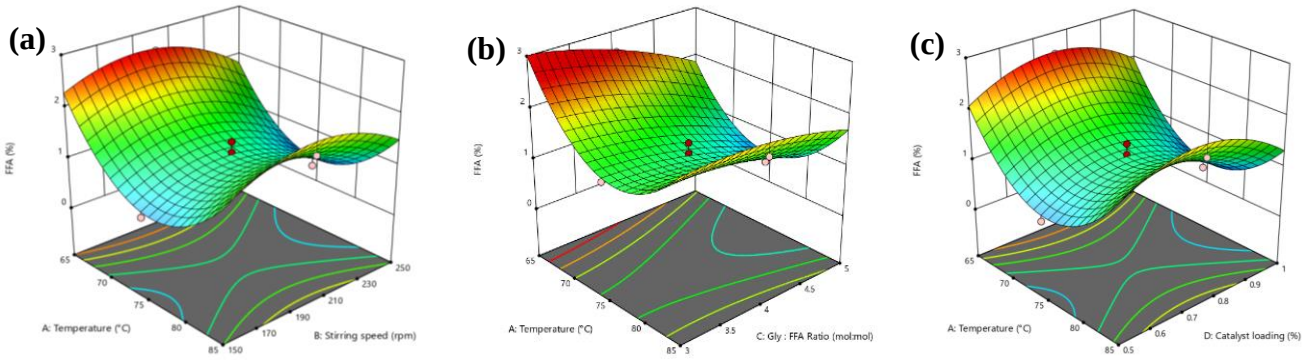

Figure 1: The temperature's correlation with other variables on the FFA of CPO (a) with stirring speed, (b) with glycerol to FFA molar ratio, and (c) with catalyst loading.

Table 1: Physicochemical characteristics of CPO and crude glycerol

Parameters	CPO	Crude glycerol
Visual appearance	Reddish to bright yellow	Light brownish yellow
Odor	Faint, fresh, and slightly green scent	Odorless, mild pleasant scent
Density (g/mL)	0.906	0.948
FFA (%)	6.150	0.720
Total glycerol (%)	-	73.290

Table 2: Parameters for glycerolysis process of high FFA in CPO.

Parameter	Symbols	Levels		
		-1	0	1
Temperature (°C)	A	65	75	85
Stirring speed (rpm)	B	150	200	250
Glycerol to FFA molar ratio (-)	C	3	4	5
Catalyst loading (wt.%)	D	0.5	0.75	1

Table 3: Actual and predicted results of CCD for reducing FFA level

Std.	Temperature (°C)	Stirring speed (rpm)	Glycerol to FFA molar ratio (-)	Catalyst loading (wt.%)	FFA (%)	
					Actual results	Predicted results
1	65	150	3	0.50	2.42	2.45
2	85	150	3	0.50	1.40	1.44
3	65	250	3	0.50	1.65	1.71
4	85	250	3	0.50	1.29	1.12
5	65	150	5	0.50	0.79	0.76
6	85	150	5	0.50	0.59	0.50
7	65	250	5	0.50	0.52	0.45
8	85	250	5	0.50	0.51	0.61
9	65	150	3	1.00	2.05	1.94
10	85	150	3	1.00	0.70	0.71
11	65	250	3	1.00	1.34	1.36
12	85	250	3	1.00	0.53	0.55
13	65	150	5	1.00	0.81	0.91

Std.	Temperature (°C)	Stirring speed (rpm)	Glycerol to FFA molar ratio (-)	Catalyst loading (wt.%)	FFA (%)	
					Actual results	Predicted results
14	85	150	5	1.00	0.51	0.44
15	65	250	5	1.00	0.81	0.76
16	85	250	5	1.00	0.80	0.70
17	65	200	4	0.75	2.58	2.62
18	85	200	4	0.75	1.83	2.08
19	75	150	4	0.75	0.59	0.70
20	75	250	4	0.75	0.28	0.46
21	75	200	3	0.75	1.29	1.37
22	75	200	5	0.75	0.38	0.60
23	75	200	4	0.50	0.54	0.66
24	75	200	4	1.00	0.28	0.45
25	75	200	4	0.75	1.19	1.15
26	75	200	4	0.75	1.40	1.15
27	75	200	4	0.75	1.40	1.15
28	75	200	4	0.75	1.40	1.15
29	75	200	4	0.75	1.19	1.15
30	75	200	4	0.75	1.19	1.15

Table 4: ANOVA and estimated coefficients of the fitted quadratic model.

Factor	Sum of squares	df	Mean square	FFA after glycerolysis (%)			
				Coefficient estimate	*F*-value	*p*-value	
Intercept	10.23	14	0.7308	1.15	21.75	< 0.0001	Significant
A-Temperature	1.29	1	1.29	-0.27	38.26	< 0.0001	
B-Stirring speed	0.2521	1	0.2521	-0.12	7.50	0.0152	
C-Glycerol to FFA molar ratio	2.68	1	2.68	-0.39	79.87	< 0.0001	
D-Catalyst loading	0.1964	1	0.1964	-0.10	5.84	0.0288	
AB	0.1764	1	0.1764	0.11	5.25	0.0368	
AC	0.57	1	0.57	0.19	16.97	0.0009	
AD	0.0484	1	0.0484	-0.06	1.44	0.2486	
BC	0.1806	1	0.1806	0.11	5.38	0.0349	
BD	0.0256	1	0.0256	0.04	0.76	0.3965	
CD	0.4422	1	0.4422	0.17	13.16	0.0025	
A^2	3.75	1	3.75	1.20	111.67	< 0.0001	
B^2	0.832	1	0.832	-0.57	24.76	0.0002	
C^2	0.072	1	0.072	-0.17	2.14	0.1639	
D^2	0.907	1	0.907	-0.59	27.00	0.0001	
Residual	0.5039	15	0.0336				
Lack of Fit	0.4378	10	0.0438		3.31	0.0994	Not significant

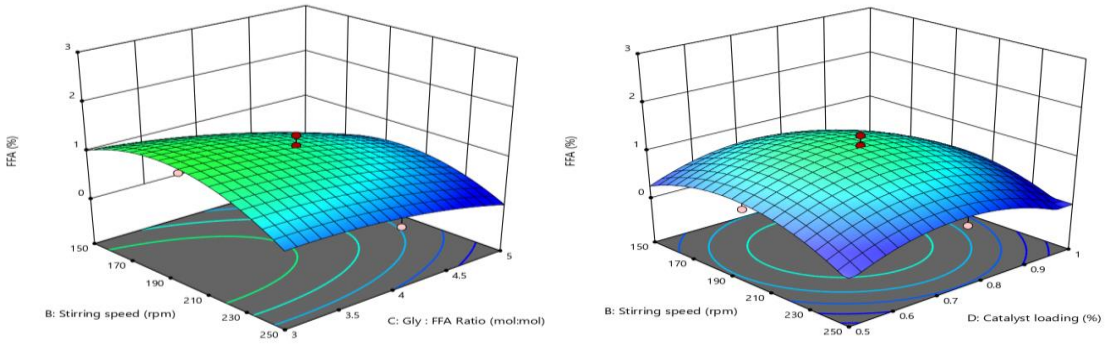

Figure 2. Stirring speed's correlation with other variables on the FFA of CPO (a) with glycerol to FFA molar ratio, (b) with catalyst loading.

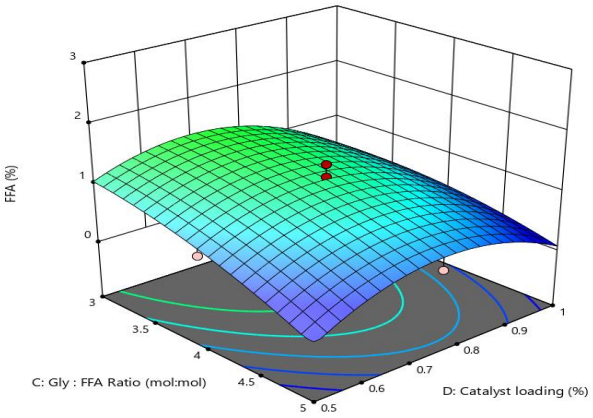

Figure 3: Glycerol to FFA molar ratio's correlation with catalyst loading on the FFA.

Figure 1(b) illustrates the combined influence of temperature and the glycerol to FFA molar ratio on FFA content in CPO at a stirring speed of 200 rpm and catalyst loading of 0.75% (w/w). Raising the glycerol to FFA molar ratio from 3:1 to 5:1 at 65°C led to a reduction in FFA content from 3.02 to 1.88%. As the glycerolysis reaction between glycerol and FFA is reversible, an excess glycerol amount (5:1 ratio) is necessary to shift the reaction equilibrium toward the formation of mono-, di-, and triglycerides.[4] Further increasing the temperature to 75°C resulted in a significant FFA reduction to 0.38%. However, beyond this temperature, no substantial improvement was observed, with the estimated FFA content at 85°C being 1.70%. Generally, higher temperatures improve mass transfer and enhance glycerol solubility in oil, facilitating the conversion process. However, excessive heat may cause undesirable flavor and color changes in the product.[7] Therefore, the optimal conditions for achieving the lowest FFA content were determined at a glycerol to FFA molar ratio of 5:1 and a temperature of 75°C.

Table 5: Analysis of regression coefficients

Fit statistics	Value
R^2	0.9531
Adj. R^2	0.9092
Prediction R^2	0.8244
Adeq. Precision	16.843
Coefficient of variance	17.05%

Figure 1(c) illustrates the combined effect of catalyst loading and temperature on FFA reduction in CPO at a 200 rpm of stirring speed and a glycerol to FFA molar ratio of 4:1. At a lower temperature of 65°C, increasing the catalyst loading from 0.5 to 1% had a minimal impact, as FFA content was only slightly reduced from 2.08% to 1.98%. However, higher catalyst loading enhances the reaction efficiency by generating a greater number of glyceroxide radicals.[22] However, excessive catalyst concentrations can raise the oil's viscosity, ultimately reducing the overall yield.[23] Proper selection of catalyst concentration is essential in glycerolysis, as the right balance between catalyst loading and operating conditions can maximize reaction efficiency and overall system performance.[8] However, raising the temperature to 75°C while maintaining a 1% catalyst loading significantly reduced the FFA content to 0.45%. Conversely, further increasing the temperature to 85°C led to a less pronounced reduction in FFA levels. These findings indicate that temperature is a key factor influencing the glycerolysis process, as the combined effect of temperature and catalyst loading improves the distribution of the catalyst's ionic liquid within the oil phase, thereby enhancing its catalytic efficiency.[24]

Figure 2(a) depicts the combined effect of glycerol to FFA molar ratio and stirring speed on FFA content in CPO. It shows that increasing the glycerol to FFA molar ratio to 5:1 at a stirring speed of 250 rpm reduced the FFA content to 0.38%. This highlights the importance of optimizing glycerol levels to enhance the reaction efficiency. However, the decrease in FFA content becomes less significant when the molar ratio is increased from 3:1 to 5:1 across all stirring speeds. This trend suggests that the intensity of mixing plays a crucial role in ensuring sufficient reactant interaction at an optimal stirring speed.[25] Similarly, Figure 2(b) illustrates the effect of stirring speed and catalyst loading on FFA reduction at a constant glycerol to FFA molar ratio of 4:1 and a temperature of 75°C. The results indicate that at a stirring speed of 250 rpm and a catalyst loading of 0.75 wt.%, the FFA content in CPO decreased to 0.28%. This finding reinforces the necessity of optimizing both stirring speed and catalyst concentration to achieve maximum FFA reduction.

Figure 3 presents the combined effect of catalyst loading and glycerol to FFA molar ratio on FFA content in CPO at a a stirring speed of 250 rpm and temperature of 75°C and. The results indicate that the lowest FFA content was achieved at a molar ratio of glycerol to FFA 5:1 and a catalyst loading of 1 wt.%. While increasing the glycerol to FFA molar ratio continued to reduce FFA levels, the rate of reduction became less significant. The figure also highlights the impact of catalyst loading, showing that increasing it from 0.5% to 1% led to a greater reduction in FFA content while keeping the stirring speed constant. Specifically, the FFA level was 0.54% at a catalyst loading of 0.5 wt.%, which further decreased to 0.28% when the catalyst loading was increased to 1 wt.%. These findings emphasize the importance of optimizing both catalyst concentration and glycerol to FFA molar ratio to achieve maximum FFA /reduction in the glycerolysis process.

Model validation

To validate the predictive accuracy of the optimized model, experimental confirmation was performed under the predicted optimal conditions: 75.17°C, 235.06 rpm stirring speed, molar ratio of glycerol to FFA 3.57:1, and 0.98 wt.% catalyst loading. The resulting FFA content of 0.27% closely matched the predicted value of 0.24%, reinforcing the reliability of the RSM approach.

Compared to previous studies, the optimized conditions in this research achieved a greater reduction in FFA content.[10,13] This demonstrates the advantage of employing RSM for process optimization, allowing for a systematic approach to refining biodiesel pre-treatment strategies.

Conclusion

This study optimized the glycerolysis process to reduce free fatty acid (FFA) content in crude palm oil (CPO) using Response Surface Methodology (RSM). Temperature, stirring speed, molar ratio of glycerol to FFA, and catalyst loading were key factors affecting FFA reduction. The optimized conditions—75.17°C, 235.06 rpm, 3.57:1 glycerol to FFA molar ratio, and 0.98 wt.% catalyst—achieved an FFA content of 0.24%, validated experimentally at 0.27%. These findings confirm RSM as an effective tool for optimizing biodiesel pre-treatment. The direct utilization of crude glycerol in reducing FFA in palm oil offers a cost-effective approach for refining high-FFA feedstocks, benefiting biodiesel industries. Future research should focus on industrial-scale applications, process stability, and alternative catalysts to further improve glycerolysis efficiency.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article are original and that any liability for claims relating to the content of this article will be borne by them.

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