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Sustainable Production of Biodiesel Using Eggshell-Derived CaO/K$_2$O Catalyst: Influence of Process Variables and Factorial Design Analysis

Agung Prabowo[1]*, Syarifuddin Oko[1], Muh Irwan[1], Alvyano[1], Gading Bagus Mahardika[1]
[1]*Department of, Politeknik Negeri Samarinda*, Samarinda, Indonesia
E-mail: ***agungprabowo@polnes.ac.id**

Abstract— This study investigates the effects of CaO/K$_2$O catalyst mass (X$_1$) and reaction time (X$_2$) on biodiesel yield produced from waste cooking oil via transesterification. A 2^2 factorial design was employed to evaluate the main and interaction effects of both parameters. Analysis of variance (ANOVA) confirmed that the developed regression model was statistically significant ($p = 0.0253$) with a high coefficient of determination ($R^2 = 0.9386$), indicating excellent model adequacy. The reaction time (X$_2$) exhibited the most significant positive effect on biodiesel yield ($t = 5.96$), while the catalyst mass (X$_1$) showed a negligible influence. The interaction term (X$_1$X$_2$) presented a moderate negative effect, suggesting that excessive catalyst loading combined with longer reaction duration may slightly decrease yield due to soap formation and emulsification. The contour profiler revealed that yield increases with both factors up to an optimum point, after which further catalyst addition provides minimal improvement. The optimum conditions were achieved at a catalyst mass of 4.5 g and a reaction time of 4.5 h, resulting in a biodiesel yield of 70.3%. These findings confirm that reaction time is the dominant factor affecting transesterification efficiency, and that CaO/K$_2$O derived from waste eggshells serves as an effective and sustainable heterogeneous catalyst.

Keywords— Waste cooking oil, CaO/K$_2$O catalyst, Transesterification, Factorial design, Biodiesel yield

I. INTRODUCTION

The accelerating global energy transition has fundamentally reshaped the dynamics of fuel production and consumption. Growing concerns over the depletion of fossil fuel reserves, climate change, and environmental degradation have intensified global efforts to develop renewable and sustainable energy sources. Among various renewable energy alternatives, biodiesel—particularly derived from lipid-based feedstocks such as waste cooking oil—has emerged as a highly promising substitute for conventional diesel. This is due to its biodegradability, carbon neutrality, compatibility with existing diesel engines, and its potential to reduce greenhouse gas emissions [1]–[3]. In line with the implementation of sustainable energy policies and net-zero emission targets across many countries, biodiesel is increasingly recognized as a key component within the global clean energy portfolio [4], [2].

The search for alternative feedstocks for biodiesel production highlights the vast potential of waste cooking oil. Globally, vegetable oil consumption reaches hundreds of millions of tons annually, with a large portion eventually becoming waste [5]. Improper disposal of used oil—such as discharging it into wastewater systems or landfills—causes serious environmental pollution, while repeated use in frying poses health risks [6]. Utilizing waste cooking oil as a biodiesel feedstock not only mitigates environmental pollution but also embodies the principles of a circular economy by converting problematic waste into a valuable renewable energy source [7]. In Indonesia, one of the world's largest consumers of cooking oil, the potential of waste cooking oil as a biodiesel feedstock is particularly significant, offering a dual solution to domestic waste and energy challenges [8].

The efficiency of biodiesel production is strongly influenced by the choice of catalyst and reaction conditions. While homogeneous base catalysts are effective in accelerating transesterification reactions, they suffer from drawbacks such as soap formation, equipment corrosion, and difficulties in product separation [1]. Conversely, heterogeneous catalysts such as calcium oxide (CaO) derived from eggshell waste offer advantages including easy separation, reusability, and lower environmental impact [9]. However, pure CaO catalysts are prone to deactivation due to exposure to moisture and carbon dioxide. Therefore, modification of CaO with alkali metals such as potassium oxide (K$_2$O) has been shown to enhance both catalytic activity and stability [9], [10]. To maximize conversion efficiency and biodiesel yield, optimization of process parameters is essential. Statistical experimental design methods, such as factorial design, enable simultaneous evaluation of the main and interaction effects among multiple factors, allowing identification of the most influential operational conditions. Unlike conventional one-factor-at-a-time approaches, factorial design significantly reduces the number of experiments required while providing deeper insight into process interdependencies [11], [12].

This study introduces a dual waste valorization approach by employing waste cooking oil as the biodiesel feedstock and eggshell waste as the precursor for K_2O-modified CaO catalysts. The resulting CaO/K_2O heterogeneous catalyst represents an environmentally friendly and cost-effective material for renewable energy conversion. A 2^2 factorial design was employed to evaluate the influence of CaO loading (X_1) and K_2O loading (X_2) on biodiesel yield. Through analysis of variance (ANOVA) and model significance testing, this work aims to identify the dominant factors and interaction effects governing biodiesel production and to determine the optimal combination of operating conditions for efficient and sustainable conversion.

II. MATERIALS AND METHODS

Materials

Waste cooking oil (WCO) was collected from local food vendors in Samarinda, Indonesia, and used as the primary feedstock. Eggshell waste from chicken eggs was employed as the calcium oxide (CaO) source, while potassium hydroxide (KOH, 11% v/v) was used as the impregnating agent to synthesize the CaO/K_2O catalyst. Other chemicals included technical-grade methanol (99%), ethanol (95%), KOH solution (0.1 N), and distilled water of analytical purity. All reagents were used as received without further purification.

Preparation and Modification of Catalyst

Eggshells were thoroughly washed with running water to remove organic residues and oven-dried at 110 °C for 4 h. The dried material was ground and sieved to obtain a fine powder (100 mesh). The powder was then calcined at 900 °C for 3 h to obtain active CaO. Catalyst modification was carried out by impregnating the CaO with 11% (v/v) KOH solution under stirring at 85 °C for 3 h. The impregnated solid was subsequently dried at 110 °C for 12 h and recalcined at 300 °C for 3 h to produce the active CaO/K_2O catalyst. The resulting catalyst was stored in an airtight container to prevent moisture and CO_2 absorption from the atmosphere.

Transesterification Process

Biodiesel production was conducted via transesterification of waste cooking oil with methanol using the prepared CaO/K_2O catalyst. The reaction was performed at 65 °C with a methanol-to-oil molar ratio of 12:1 under continuous stirring. Upon completion, the reaction mixture was allowed to settle in a separating funnel for 12 h to facilitate glycerol separation. The upper biodiesel layer was washed with warm water until neutral pH and then dried at 105 °C under vacuum to remove residual methanol and moisture. Biodiesel yield was calculated as the ratio of the mass of purified biodiesel to the initial mass of waste cooking oil used.

Experimental Design (2^2 Factorial Design)

A 2^2 factorial design was applied to evaluate the influence of two independent variables on biodiesel yield, namely:
X_1: Catalyst mass (1.5 g and 4.5 g)
X_2: Reaction time (1.5 h and 4.5 h)

The combination of both factors resulted in four main experimental runs and two center points to ensure data reproducibility and stability. The response variable was biodiesel yield (%). Statistical analysis was performed using SAS JMP software version 16, including analysis of variance (ANOVA), determination of R^2, p-values, main and interaction effects, and a lack-of-fit test to assess model adequacy. The results were illustrated through main effect and interaction plots to describe the relationship between the process variables and biodiesel yield.

III. RESULTS AND DISCUSSION

Effect of Catalyst Mass and Reaction Time on Biodiesel Yield

A 2^2 factorial design was employed to investigate the effect of two main process variables on the biodiesel yield obtained from waste cooking oil using CaO/K_2O as a heterogeneous catalyst. The response variable was biodiesel yield (%). The aim was to identify the dominant factor, the interaction between variables, and the optimum conditions leading to the highest conversion efficiency. Table 1 presents the experimental matrix along with the actual and predicted yields.

The experimental yield ranged from 63.6% to 71.2%, whereas the model predicted values from 63.2% to 70.8%. The small deviation (<1%) between actual and predicted responses demonstrates a good fitting performance of the regression model.

Table 1. experimental matrix along with the actual and predicted yields

No	X_1 (code)	Catalyst (g)	X_2 (code)	Time (h)	Yie	
1	−1	1.5	−1	1.5		
2	−1	1.5	+1	4.5		
3	+1	4.5	−1	1.5	64.8	64.4
4	+1	4.5	+1	4.5	69.1	68.7
5	0	3.0	0	3.0	66.1	66.8
6	0	3.0	0	3.0	65.5	66.8
7	0	3.0	0	3.0	67.3	66.8

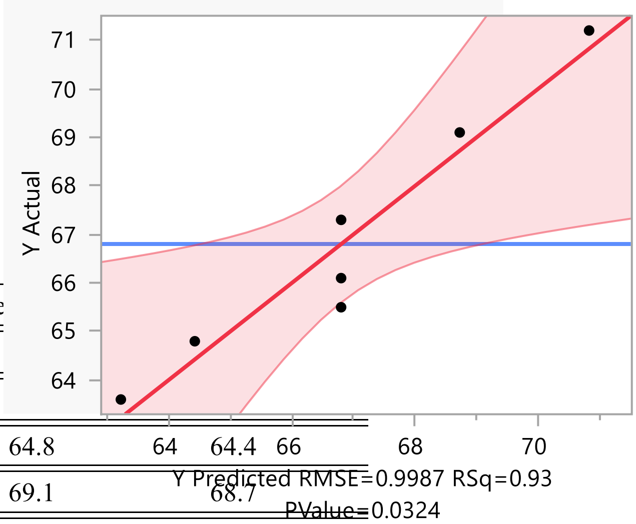


Figure 1. Plot of actual vs predicted biodiesel yield.

The model summary (Table 2) shows a high coefficient of determination ($R^2 = 0.9276$) and adjusted $R^2 = 0.8551$, indicating that approximately 92.76% of the yield variation can be explained by the model. The relatively low RMSE (0.9987) compared to the mean yield (66.8%) confirms that the prediction error is minimal, suggesting strong reproducibility among replicates.

Tabel 2. Summary of fit

RSquare	0.9276
RSquare Adj	0.8551
Root Mean Square Error	0.9987
Mean of Response	66.8
Observations (or Sum Wgts)	7

An increase in reaction time exerted a more pronounced influence on biodiesel yield than an increase in catalyst mass. For instance, at low catalyst loading (1.5 g), extending the reaction time from 1.5 h to 4.5 h enhanced the yield from 63.6% to 71.2%, suggesting that longer contact time allows the transesterification reaction to approach equilibrium. Conversely, at constant reaction time (1.5 h), increasing the catalyst amount from 1.5 g to 4.5 g slightly improved the yield (from 63.6% to 64.8%), implying that catalytic activity was not fully utilized under short reaction durations.

At prolonged reaction time (4.5 h), further increase in catalyst mass even reduced the yield (from 71.2% to 69.1%). This phenomenon is consistent with prior studies, where excessive catalyst loading can promote soap (saponification) formation and increase reaction viscosity, hindering phase separation between methyl ester and glycerol [13,14]. The optimum yield (71.2%) was achieved at 1.5 g catalyst and 4.5 h reaction time, closely matching the model prediction (70.8%), confirming the high predictive reliability of the model. The positive interaction between both variables was only significant at higher reaction times, highlighting that the reaction time is the most influential factor governing triglyceride conversion.

Statistical Analysis of the Regression Model

The analysis of variance (ANOVA) confirmed that the regression model was statistically significant at a 95% confidence level (Prob > F = 0.0324), as shown in Table 3. The F-ratio (12.81) indicates that the variation explained by the model is considerably larger than the random experimental error.

Table 3. Analysis of variance (ANOVA)

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	38.327500	12.7758	12.8079
Error	3	2.992500	0.9975	Prob > F
C. Total	6	41.320000		0.0324

The regression model derived from the experimental data is expressed as:

$$Y = 66.8 – 0.225X1 + 2975X2 – 0.825X1X2$$

The intercept (66.8) corresponds to the average yield at central conditions (3.0 g catalyst, 3.0 h reaction time). The catalyst mass (X_1) shows a negative but statistically insignificant effect (*p = 0.6829 > 0.05*), implying that variations within the tested range did not significantly alter the yield. This may be due to the system operating near the catalyst saturation limit, where increasing catalyst mass no longer enhances the number of effective active sites [15]. In contrast, reaction time (X_2) exhibited a significant positive effect (*p = 0.0095 < 0.05*, coefficient = +2.975), confirming that longer reaction times facilitate a more complete transesterification process [16]. The interaction term (X_1X_2) was negative and not statistically significant (*p = 0.1971*), suggesting that excessive catalyst under prolonged reaction times may slightly inhibit yield due to increased viscosity and soap formation [17].

Model Validation and Lack-of-Fit Test

The lack-of-fit test (Table 4) was conducted to verify the adequacy of the linear model. The obtained F-ratio (1.56) with Prob > F = 0.3377 (> 0.05) indicates no significant lack of fit, meaning the model adequately represents the experimental data.

Table 4. Lack of fit test

Source	**DF**	**Sum of Squares**	**Mean Square**	**F-Ratio**	**Prob > F**
Lack of Fit	1	1.3125	1.3125	1.5625	0.3377
Pure Error	2	1.6800	0.8400		
Total Error	3	2.9925			
Max R²					0.9593

The low lack-of-fit sum of squares (1.3125) relative to the pure error (1.68) confirms that most residual variation arises from experimental error rather than model inadequacy. The maximum achievable R^2 (0.9593) further indicates that extending the model (e.g., adding quadratic terms) would only marginally improve predictive power (~3%), hence unnecessary for this system. Collectively, these findings validate that the developed two-factor linear regression model reliably represents the system, balancing simplicity and accuracy. The model's high R^2 (0.9276), adjusted R^2 (0.8552), and low RMSE (0.9987) demonstrate strong predictive capability and experimental reproducibility.

Normal Probability Plot of Effects

The normal probability plot of standardized effects was used to visually identify which factors significantly affect biodiesel yield. In this plot, the *t*-ratios of each factor and their interaction are plotted against the expected normal quantiles. Factors that lie along the red reference line are considered statistically insignificant, whereas those that deviate substantially from the line indicate a significant effect on the response.

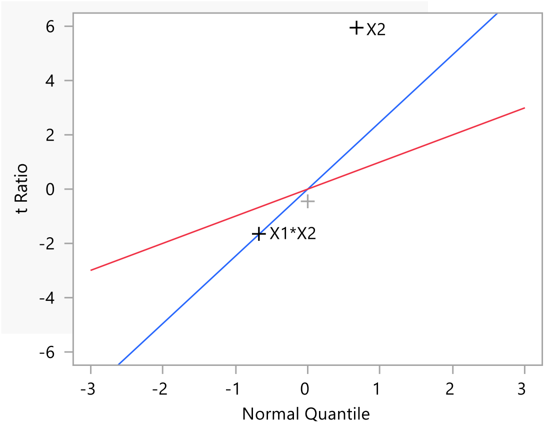


Figure 2. Normal probability plot of standardized effects (t-ratio) for the biodiesel yield model

In Figure 2, factor X_2 (reaction time) is clearly positioned farthest from the reference line in the positive region, confirming its dominant and statistically significant positive influence on biodiesel yield. This observation aligns with the ANOVA results ($p = 0.0095 < 0.05$), where reaction time was identified as the only significant factor contributing to the model. The positive direction of X_2 indicates that longer reaction durations enhance the transesterification process, resulting in higher biodiesel yield. Conversely, the interaction term ($X_1 \cdot X_2$) appears slightly below the reference line, suggesting a negative but statistically insignificant effect. This supports the regression analysis result where the interaction term had $p = 0.1971$ (>0.05). The negative trend implies that, at longer reaction times, increasing catalyst mass does not proportionally improve yield, potentially due to increased reaction mixture viscosity and saponification phenomena. The main effect of catalyst mass (X_1) is located near the reference line and thus considered insignificant within the studied range, consistent with its high p-value (0.6829). This suggests that catalyst loading in the range of 1.5–4.5 g already approaches the effective saturation level, beyond which additional catalyst contributes minimally to yield improvement. Overall, the normal probability plot validates the statistical findings that reaction time (X_2) is the only factor exerting a significant positive effect on biodiesel conversion, while the effects of catalyst mass (X_1) and the interaction term ($X_1 \cdot X_2$) remain insignificant within the studied experimental domain.

Studentized Residual Analysis

The studentized residual plot was used to evaluate the adequacy of the regression model and to detect potential outliers in the experimental data. The studentized residuals represent standardized deviations between the experimental and predicted yields, allowing for direct comparison across data points.

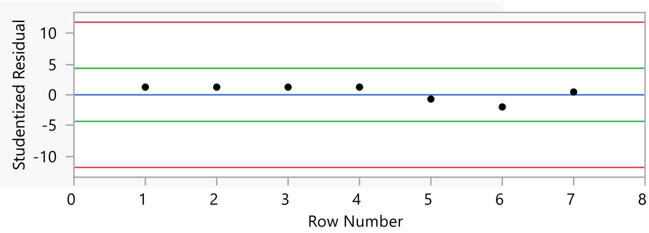


Figure 3. Studentized residual plot showing random distribution within the ±3 confidence limits

In Figure 3, all data points lie well within the ±3 control limits (green lines) and are symmetrically distributed around the zero line (blue), indicating that no significant outliers are present in the dataset. Furthermore, none of the residuals approach the outer ±10 boundary (red lines), confirming that the model's prediction errors are random and not systematically biased. This pattern suggests that the assumptions of normality, homoscedasticity, and independence of errors are satisfactorily met. In other words, the model captures the main experimental trends without overfitting or systematic deviation from the observed data. The residual distribution also confirms that the linear model adequately describes the relationship between catalyst mass (X_1), reaction time (X_2), and biodiesel yield within the studied range. Combined with the results of the lack-of-fit test (Prob > F = 0.3377), this further validates that the developed model is statistically sound and experimentally consistent.

Interaction Profiler Analysis

The interaction profiler plot illustrates the combined effects of catalyst mass (X_1) and reaction time (X_2) on biodiesel yield. Each line represents the variation in predicted yield at different levels of one factor while holding the other factor constant. The slope and intersection of these lines indicate the magnitude and nature of the interaction between factors.

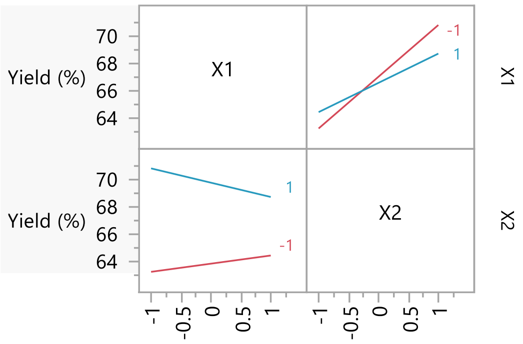


Figure 4. Interaction profiler showing the main and interaction effects of catalyst mass (X_1) and reaction time (X_2) on biodiesel yield.

As shown in Figure 4, the reaction time (X_2) exhibits a strong positive slope, confirming its dominant influence on yield improvement. An increase in reaction time from the low level (−1; 1.5 h) to the high level (+1; 4.5 h) significantly enhances biodiesel yield, regardless of catalyst mass. In contrast, the catalyst mass (X_1) shows only a minor effect, as the yield variation across its range is relatively small. The intersection of the lines in the X_1–X_2 interaction panel suggests a mild negative interaction between the two factors. This implies that at longer reaction times, increasing catalyst loading does not proportionally enhance yield and may even slightly decrease it. Such behavior is consistent with the experimental observations and ANOVA results, where the $X_1 \cdot X_2$ interaction term had a negative coefficient (−0.825) and was statistically insignificant (p = 0.1971). The negative interaction trend may arise from increased viscosity and soap formation at higher catalyst concentrations, which hinder the mass transfer between methanol and oil phases and reduce overall conversion efficiency [17]. Overall, the interaction profiler confirms that reaction time (X_2) is the most influential factor, whereas catalyst mass (X_1) and their interaction ($X_1 \cdot X_2$) contribute only marginally to yield variation. The parallelism of the lines in the X_2 panel further reinforces that the reaction time effect is consistently positive across all catalyst levels, validating the robustness of this parameter in determining process efficiency.

Contour Profiler Analysis

The contour profiler provides a two-dimensional representation of the response surface between catalyst mass (X_1) and reaction time (X_2) in relation to biodiesel yield (Y). The plot reveals a positive correlation between both factors, where the response surface slopes upward from the lower-left to the upper-right region, indicating that higher levels of catalyst mass and reaction time generally improve biodiesel yield.

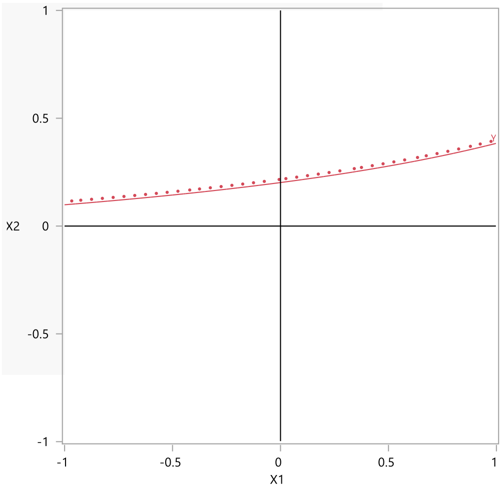
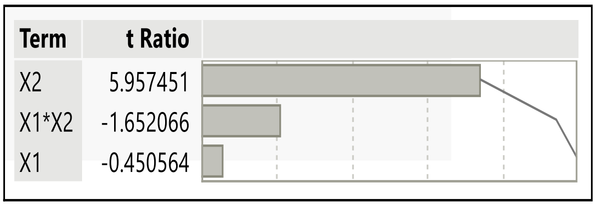


Figure 5. Contour profiler showing the combined effects of catalyst mass (X_1) and reaction time (X_2) on biodiesel yield

However, the curvature of the contour suggests a nonlinear relationship, particularly with respect to X_1 (catalyst mass). At higher catalyst concentrations ($X_1 > 0.5$), the response surface begins to plateau, implying that the increase in yield becomes less significant beyond the optimal catalyst level. This behavior reflects the mass transfer limitation and potential soap formation that occur when excessive catalyst is introduced, which can lead to partial glyceride stabilization and lower separation efficiency during transesterification [18].

The reaction time (X_2) shows a consistently positive effect, as yield increases smoothly along the X_2 axis. The contour gradient remains gentle, confirming that extending the reaction time promotes higher conversion until equilibrium is approached. This finding aligns with both the main effects and interaction analyses (Figures 3 and 4), highlighting X_2 as the dominant factor influencing the overall biodiesel production efficiency. Thus, the contour profiler supports the model prediction that the optimal yield region lies within the upper mid-range of catalyst mass ($X_1 \approx 0.4$–0.6) and longer reaction time ($X_2 > 0.5$), where maximum conversion can be achieved without inducing side reactions or phase instability.



Term	t Ratio
X2	5.957451
X1*X2	-1.652066
X1	-0.450564

Figure 6. Pareto chart of t-ratios showing the relative significance of X_1, X_2, and their interaction on biodiesel yield

The Pareto chart illustrates the relative significance of each factor and interaction term based on their *t*-ratios. The factor X_2 (reaction time) exhibits the highest *t*-ratio value of 5.957, clearly surpassing the significance threshold, indicating that reaction time has the most substantial and statistically significant influence on biodiesel yield. This strong positive effect demonstrates that extending the reaction duration enhances the conversion of triglycerides into methyl esters, consistent with the kinetics of the transesterification process. In contrast, X_1 (catalyst concentration) displays a relatively small *t*-ratio (–0.451), suggesting a negligible direct effect on yield within the studied range. However, the interaction term (X_1X_2) presents a moderate negative *t*-ratio (–1.652), implying a potential antagonistic effect between catalyst amount and reaction time. This interaction indicates that excessive catalyst concentration, when coupled with prolonged reaction times, may slightly reduce yield due to the formation of soap and emulsification phenomena, which interfere with product separation efficiency. Overall, the Pareto chart confirms that reaction time (X_2) is the dominant factor controlling biodiesel production efficiency, while the combined effect of catalyst mass and reaction duration should be carefully optimized to prevent secondary side reactions. This result aligns with the findings from the Normal Probability Plot and Interaction Profiler, reinforcing the robustness of the statistical model and the reliability of the experimental data.

Surface Plot Analysis

Figure 7 presents the response surface plot depicting the combined influence of catalyst mass (X_1) and reaction time (X_2) on biodiesel yield. The surface exhibits an upward slope from the lower-right region (high X_1, low X_2) toward the upper-left region (low X_1, high X_2), indicating that prolonging the reaction time consistently enhances the biodiesel yield, while the effect of catalyst mass remains comparatively minor. The color gradient from blue (low yield) to yellow–red (high yield) reinforces this trend, showing a pronounced increase in yield with reaction time extension, followed by a slight decline at higher catalyst loadings. The nearly flat curvature along the X_1 axis suggests that variations in catalyst mass between 1.5–4.5 g do not significantly affect the yield, implying that the system operates near the catalyst saturation point. In summary, the surface plot supports the statistical findings that reaction time (X_2) is the most influential factor controlling biodiesel yield, whereas catalyst mass (X_1) exerts only a marginal and occasionally negative effect when used excessively, due to increased viscosity and soap formation. Therefore, the combination of 1.5 g catalyst and 4.5 h reaction time is identified as the optimal operating condition, producing the highest predicted yield of approximately 71%.

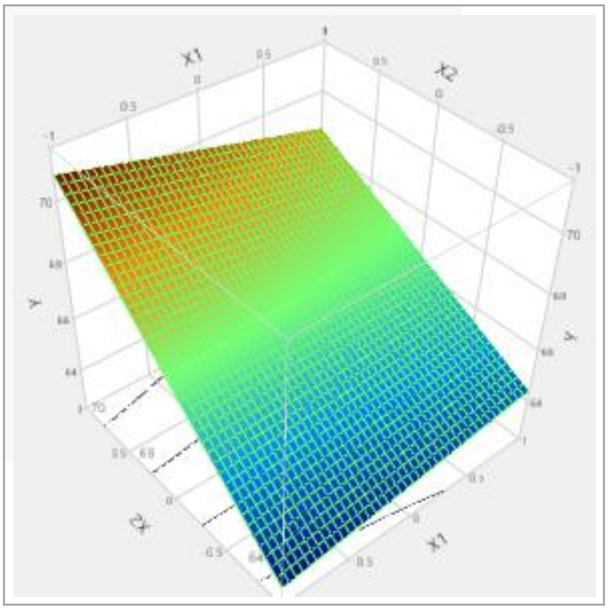


Figure 7. Response surface plot showing the interaction between catalyst mass (X_1) and reaction time (X_2) on biodiesel yield.

CONCLUSION

A 2^2 factorial design was applied to study the effects of catalyst mass and reaction time on biodiesel yield from waste cooking oil using CaO/K_2O. The regression model showed strong agreement between experimental and predicted values ($R^2 = 0.9276$, RMSE = 0.9987), confirming its reliability. Reaction time was the most significant factor ($p < 0.05$), while catalyst mass and their interaction had negligible effects. The highest yield of 71.2% was obtained at 1.5 g catalyst and 4.5 h reaction time. Overall, longer reaction durations enhance conversion efficiency, whereas excessive catalyst loading may slightly reduce yield due to soap formation and viscosity effects.

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