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Optimization of Ethanol Purification by Adsorption Process Using Silica Gel with Response Surface Methodology

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ABSTRACT

Ethanol from the fermentation of sugar, cane, and biomass can be used as a renewable fuel, but it must be purified first with adsorption being an efficient method. This study aims to optimize ethanol purification using silica gel adsorption, investigating the effects of flow rates 0.1; 0.2; 0.3; 0.4; 0.5 ml/s and adsorbent weights 20, 30, 40, 50, 60 grams. Response Surface Methodology (RSM) with Central Composite Design (CCD) was used for optimization. The experimental results were input into Design-Expert 13 for RSM optimization using flow rate and adsorbent weight. The highest ethanol concentration of 99.8% was achieved at a flow rate of 0.1 ml/s and adsorbent weight of 60 grams. RSM optimization resulted in a concentration of 99.804% at a flow rate of 0.103 ml/s and silica gel weight of 59.818 grams. The results indicate that increasing adsorbent weight and decreasing flow rate significantly improve ethanol purity. Achieving 99.8% ethanol confirms the effectiveness of silica gel adsorption and supports the development of biofuels as renewable energy sources to reduce dependence on petroleum.

INTRODUCTION

Fuel consumption in Indonesia has been increasing over time. Therefore, alternative fuels or renewable energy sources are needed to replace petroleum, which is depleting. One possible substitute is ethanol. Ethanol can reduce dependence on imported foreign oil and help mitigate the effects of oil price fluctuations. The use of fuels blended with fuel-grade ethanol results in lower carbon emissions, thereby reducing environmental pollution. At a temperature of 60°C, ethanol has a heat of vaporization of 1177 kJ/kg, while gasoline has a heat of vaporization of 348 kJ/kg. Due to its 34.7% oxygen content, ethanol is considered more environmentally friendly compared to gasoline, which contains no oxygen (Zabed, 2017).

The purification of ethanol can be challenging due to the difficulty in separating hydrogen that is bound within its chemical structure. This is caused by the presence of an azeotropic point, which refers to a mixture of two or more components that has a constant boiling point (Sehwantoro, 2021). The existence of an azeotrope in the ethanol-water mixture makes simple distillation less effective for separation. This is due to the relatively close boiling points of ethanol and water, which are 78.3°C and 100°C, respectively. To separate ethanol from its mixture with water, methods such as extractive distillation or adsorption using porous media can be applied. Extractive distillation is a separation technique that utilizes a solvent (Wahyudi, 2017). Using extractive distillation, an ethanol concentration of 93.6322% was achieved (Bobsaid, 2023). However, this result indicates that extractive distillation is not highly effective in breaking the ethanol-water azeotropic point. Adsorptive distillation, on the other hand, is a process in which a fluid adheres to a solid or specific liquid called an adsorbent, involving physico-chemical bonding between the solute and the adsorbent (Purwitasari, 2022).

An adsorbent is a porous material capable of capturing substances through the adsorption process on the surface of its pores or at specific sites within the pores (Dwijayanti, 2023). When selecting an adsorbent, several criteria must be considered, including a large surface area, the presence of active pores, high purity, and chemical inertness toward the adsorbate (Atikah, 2019). One example of an adsorbent is silica gel, an inorganic solid material containing silanol (Si–OH) and siloxane (Si–O–Si) groups. The production process of silica gel consists of four main stages: first, the formation of sodium silicate through the reaction of silica from rice husk ash with sodium hydroxide; second, the fusion process at high temperatures; third, the reaction of sodium silicate with acid to form a hydrosol; and fourth, heating of the silica hydrogel to produce dry silica gel or sol-gel (Riza, 2022). In this process, the main raw material used is sodium silicate, which is obtained through the reaction between silica and sodium hydroxide as shown in the following reaction:

$$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)} \quad (1)$$

According to the Indonesian National Standard (SNI) No. 06-2477-1991, the standard for silica gel includes three main characteristics: a maximum moisture content of 15%, a maximum ash content of 10%, and an iodine adsorption capacity of at least 750 mg/g (Bramanta, 2023).

Adsorption offers several advantages, including low cost, operational simplicity, high efficiency, and the ability to regenerate the adsorbent (Riyanti, 2024). The purification of ethanol through adsorption using silica gel is categorized as solid-liquid adsorption. In solid-liquid adsorption, the solid adsorbent is silica gel, while the liquid adsorbate is the ethanol-water mixture. This process consists of three main stages. The first stage involves the mass transfer of the adsorbate by diffusion from the bulk fluid to the external surface of the solid. The second stage is the diffusion of the adsorbate into the pores of the adsorbent and the phase that has already adsorbed the substance. The final stage is the attachment of the solute to the active sites of the adsorbent, which can occur through physical or chemical mechanisms (Inglezakis, 2020).

This study aims to investigate the purification process of 96% ethanol by varying the flow rates at 0.1, 0.2, 0.3, 0.4, and 0.5 ml/s, and using silica gel weights of 20, 30, 40, 50, and 60 grams, with the goal of achieving fuel-grade ethanol at 99.8%. Additionally, the study seeks to analyze the effect of the amount of silica gel in the column and the flow rate on the increase in ethanol concentration, as well as to optimize ethanol concentration using the Response Surface Methodology (RSM).

This study aims to investigate the purification process of 96% ethanol using varying flow rates of 0.1, 0.2, 0.3, 0.4, and 0.5 ml/s and silica gel weights of 20, 30, 40, 50, and 60 grams, with the goal of achieving fuel-grade ethanol at 99.8%. In addition, the research aims to analyze the effects of silica gel quantity in the column and flow rate on ethanol concentration improvement, as well as to optimize ethanol purity using the Response Surface Methodology (RSM). Several previous studies have examined ethanol purification using extractive distillation methods; however, few have explored adsorption with silica gel as a more environmentally friendly alternative. Moreover, most existing research still relies on trial-and-error approaches or simple factorial designs, without applying statistical optimization methods capable of modeling complex interactions between variables. Therefore, RSM is employed in this study due to its ability to evaluate variable interactions and identify optimal conditions efficiently with fewer experimental runs compared to conventional methods.

RESEARCH METHODS

Materials and Equipment

The equipment used in this study consisted of a series of adsorption apparatuses, as shown in figure 1, including a three-neck flask, thermometer, stand, clamp, adsorption column, divider, condenser, valve, hose, erlenmeyer flask, and heating mantle. The primary materials used in this study were 96% ethanol and silica gel. The 96% ethanol was obtained from PT. Enero Mojokerto and was produced through a fermentation and distillation process using molasses as the raw material. Standard silica gel was purchased from CV. Nirwana Sains.

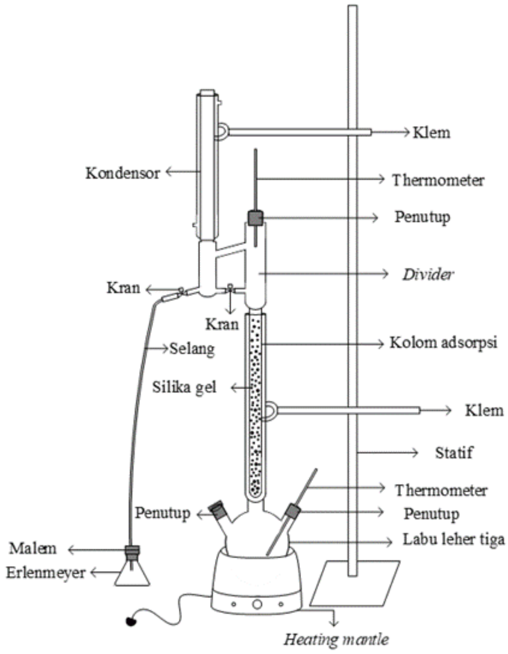

Figure 1. Adsorption Equipment Setup

Procedure

A total of 25 experimental runs were performed, each representing a distinct combination of flow rate and adsorbent weight, without replication. Model validation was conducted by comparing the predicted values obtained from the RSM optimization, which was based on 13 Central Composite Design (CCD) data points, with the corresponding actual experimental results. The model's accuracy was evaluated through the analysis of prediction errors and confidence intervals.

Adsorption Process

The adsorption process was carried out by evaporating 300 ml of ethanol at a temperature of approximately 78°C. The ethanol vapor passed through an adsorption column containing silica gel where water and impurities present in the vapor were adsorbed by the silica gel. Subsequently, the ethanol vapor was condensed into liquid form and collected in an erlenmeyer flask for analysis using an alcoholmeter.

Optimization Procedure (RSM)

The determination of optimal variables was conducted through optimization using Response Surface Methodology (RSM) with Central Composite Design (CCD) method, employing three center points. Based on the CCD analysis, a total of 11 experimental runs were required, involving two factors: ethanol flow rate (mL/s) and silica gel weight (grams) and one response variable, namely ethanol concentration (%). Experimental data optimization was performed using the Design-Expert 13 software to obtain the Analysis of Variance (ANOVA) results, response surface plots, contour plots, as well as the optimal conditions and their verification.

RESULTS AND DISCUSSION

Purification Results Using the Adsorption Process

The ethanol purification results from each adsorption variable were collected and measured for concentration using an alcoholmeter, as shown in Figure 2. The results from each experimental variable are presented in Figure 3 and 4 to illustrate their effects.

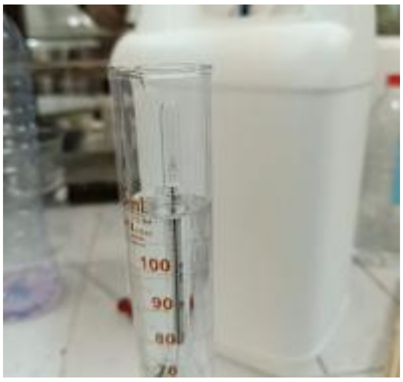

Figure 2. Analyzing ethanol concentration using an alcoholmeter.

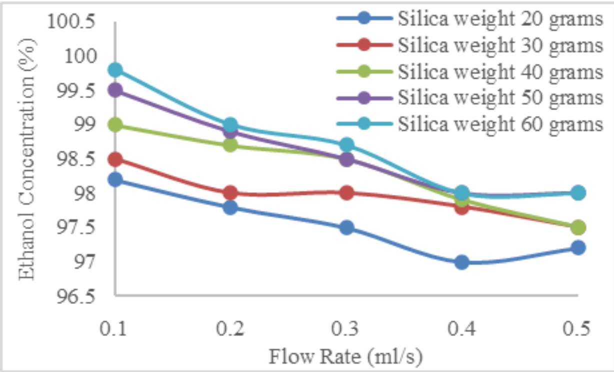

Figure 3. Graph of the Relationship Between Ethanol Flow Rates (mL/s) and Ethanol Concentrations (%) at Various Silica Gel Weights (grams)

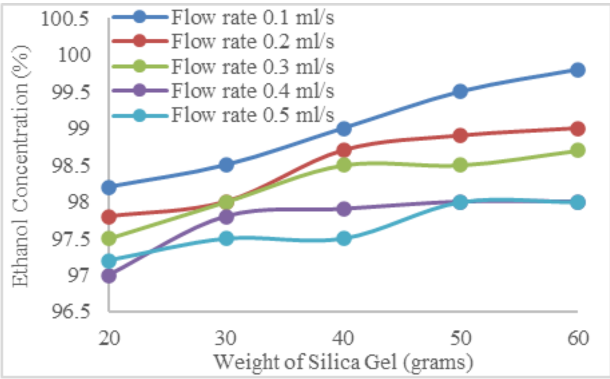

Figure 4. Graph of the Relationship Between Silica Gel Weights (grams) and Ethanol Concentrations (%) at Various Ethanol Flow Rates (mL/s)

Based on Figure 3, ethanol concentration is shown to be inversely proportional to the flow rate during the adsorption process, where a lower flow rate results in a higher ethanol concentration. This is due to the longer contact time between the ethanol–water mixture and the silica gel, which enhances the effectiveness of water adsorption (Amsar, 2016). Conversely, a higher flow rate shortens the contact time, leading to suboptimal water adsorption and reduced purification efficiency. However, at certain flow rate points, a constant or even increased ethanol concentration is observed. The constant concentration indicates that dynamic equilibrium has been reached, where the silica gel is operating at its optimal capacity. Meanwhile, the increase in ethanol concentration at higher flow rates is presumed to occur due to the rapid flow causing partial desorption of impurities previously adsorbed onto the surface of the silica gel.

Based on Figure 4, the ethanol concentration is directly proportional to the weight of silica gel during the adsorption process. However, there are points at which the ethanol concentration remains constant despite increasing the adsorbent weight. This plateau occurs when the silica gel reaches its maximum capacity to adsorb water from the ethanol, indicating it has reached saturation. The highest ethanol concentration was achieved when 60 grams of silica gel were used. The greater the amount of silica gel, the more water is adsorbed from the ethanol mixture, resulting in ethanol with lower water content and higher purity (Oktaviani, 2017).

These results are in line with the study by Ibrahim (2022), which reported that decreasing the flow rate in the adsorption process using silica gel significantly increases the efficiency of water removal from ethanol, especially in fixed-bed column systems. However, the maximum ethanol concentration achieved in this study 99.8% is higher than that reported in previous research, which only reached approximately 98.5%. This difference is likely due to the use of a larger amount of silica gel and more integrated control of operational parameters in this study. The greater the amount of silica gel used, the more water molecules from the ethanol–water mixture can be adsorbed, resulting in lower water content and higher ethanol purity in the final product. In addition, the study by González (2021) also supports the finding that increasing the adsorbent mass can improve ethanol purity, although the maximum concentration achieved in that study remained below the result of the present research. These findings indicate that the combination of a low flow rate and optimal adsorbent mass has a significant synergistic effect in achieving higher ethanol purity levels.

Optimization Results of Ethanol Purification Data Using Response Surface Methodology (RSM)

Based on the CCD analysis, a total of 11 experimental runs were required, involving two factors:

Table 1. Factor and Response Data for RSM Analysis (11 Data Points)

Run	Factor 1	Factor 2	Response
	Silica Gel Weight (gr)	Flow Rate (mL/s)	Ethanol Concentration (%)
1	40	0.3	98.5
2	20	0.3	97.5
3	60	0.5	98.0
4	40	0.5	97.5
5	60	0.1	99.8
6	20	0.5	97.9
7	40	0.1	99.0
8	40	0.3	98.5
9	60	0.3	98.7
10	40	0.3	98.5
11	20	0.1	98.2

The experimental data will be input into Design-Expert 13 to interpret the relationship between the variables ethanol flow rate and silica gel weight and the response, which is ethanol concentration. This will be analyzed using Analysis of Variance (ANOVA), as presented in Table 2 below.

Table 2. Analysis of Variance (ANOVA) Results

Source	df	F-value	P-value	
Model	3	25.48	0.0004	significant
A-Silica Gel Weight	1	25.98	0.0014	
B-Flow Rate	1	40.04	0.0004	
AB	1	10.43	0.0145	
Residual	7			
Lack of Fit	5			

In table 2, the P-value for silica gel weight was 0.0014 and for ethanol flow rate was 0.0004, both of which are less than 0.05 (5%), indicating that these variables have a statistically significant effect on the response. The F-value indicates that the model used can adequately explain the data variation, suggesting that the recommended regression model is significant (Tesfaw, 2021). The ANOVA determination was based on the equation provided by the software.

Table 3. Comparison of the Regression Models Recommended by the Software

Source	R^2	Adj. R^2	Pred. R^2	PRESS	
Linear	0.739	0.739	0.456	2.45	
2FI	**0.916**	**0.880**	**0.722**	**1.25**	**Suggested**
Quadratic	0.842	0.842	0.376	2.81	
Cubic	0.817	0.817	-5.962	31.35	**Aliased**

Based on Table 3, the recommended model is the Two-Factor Interaction (2FI) model, as the difference between the Adjusted R^2 and Predicted R^2 values is less than 0.2, and the standard deviation in the 2FI model is the smallest compared to the other models. The Predicted Error Sum of Squares (PRESS) value for the 2FI model is also the lowest among all models, indicating better predictive accuracy, as a lower PRESS value reflects improved prediction performance (Bas and Boyaci, 2017). The linear 2FI model equation obtained from the software analysis for each factor's effect on the response is as follows:

$$Y = 97.1811 + 0.0523X_1 + 0.7500X_2 - 0.0938X_1X_2 \quad (2)$$

Description:
Y = Ethanol concentration response
X_1 = Silica gel weight
X_2 = Flow rate

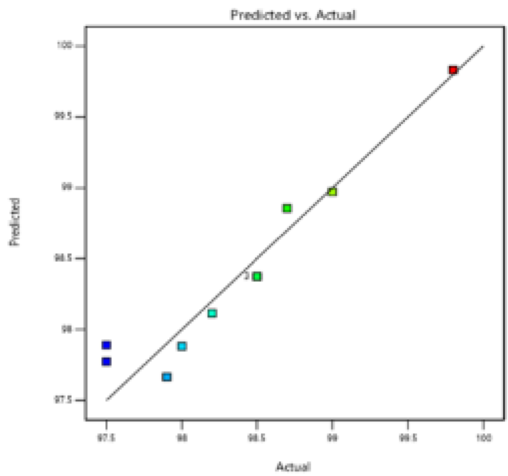

Figure 5. Comparison Chart of Ethanol Concentration Response: Predicted vs. Actual Values

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Figure 5 shows the relationship between actual and predicted values. The closer the actual data data points are to the line of normality, the better agreement between the actual data and the predicted results (Ramadhanti, 2024). The response surface plots illustrating the effects of silica gel weight and ethanol flow rate on ethanol concentration are presented in Figure 6 and 7.

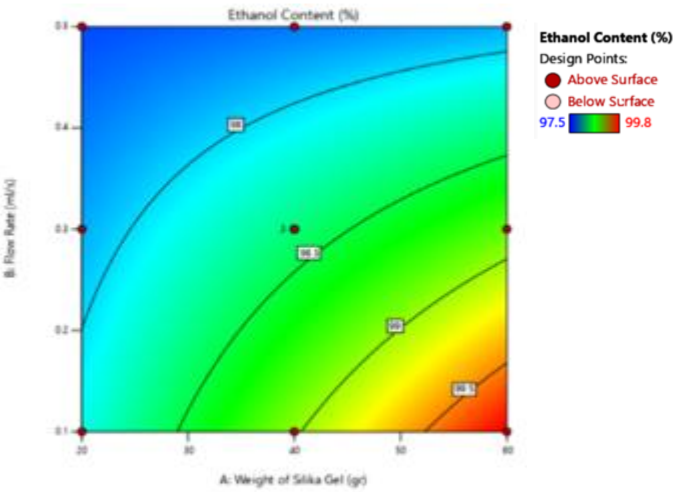

Figure 6. Contour Plot Showing the Effect of Silica Gel Weight (gr) and Flow Rate (mL/s) on Ethanol Concentration (%)

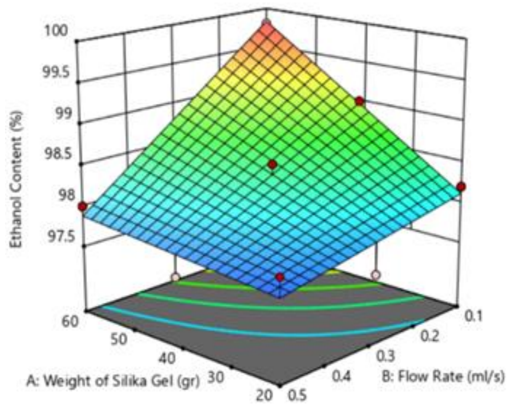

Figure 7. 3D surface Plot Showing the Effect of Silica Gel Weight (gr) and Flow Rate (mL/s) on Ethanol Concentration (%)

The contour plot represents the two-dimensional response based on the predictive model, while the 3D surface plot provides a three-dimensional visualization. The contour lines, composed of data points, indicate combinations of variables that yield the same ethanol concentration (Rahmawati, 2022). As shown in Figure 6 and 7, the highest ethanol concentration of 99.8% was achieved at silica gel weight of 60 grams and flow rate of 0.1 mL/s. The plots feature three color gradients, where deeper red indicates higher ethanol purity. Optimization was performed using software to maximize ethanol concentration, based on 11 experimental runs. The parameters used for determining optimal conditions are presented in Table 4.

Table 4. Factor and Response Constraints for Optimization

Name	Goal	Lower Limit	Upper Limit
A: Silica Gel Weight	is in range	20	60
B: Flow Rate	is in range	0.1	0.5
Ethanol Consentration	maximize	97.5	99.8

The optimum solution was derived from the combination of two factors: flow rate and silica gel weight that effectively enhanced ethanol concentration after purification. The optimal solution obtained from the data analysis is presented in Table 5.

Table 5. Optimization Results of Ethanol Concentration Response

Number	Silica Gel Weight (gr)	Flow Rate (mL/s)	Ethanol Concentration (%)	Desirability
1	59.818	0.103	99.808	1.000

Based on Table 5, the optimal results were achieved at a flow rate of 0.102 mL/s and a silica gel weight of 59.617 grams. The maximum ethanol concentration obtained was 99.804%, with a desirability value of 1. Desirability values range from 0.1 to 1, where higher values indicate a more accurate optimization outcome aligned with the desired response (Anwar, 2021). The optimization results were subsequently verified, as shown in Tables 6 and 7.

Table 6. Verification Results of The Confidence Interval

Analysis	95% CI low	95% CI high	95% TI low	95% TI high
Ethanol Concentration	99.364	100.252	98.3771	101.239

Table 7. Verification Results of The Prediction Interval

Solution 1 of 11 Response	n	SE Pred	95% PI low	95% PI high
Ethanol Concentration	1	0.2803	97.0421	98.3675

In Table 6, the Confidence Interval (CI) indicates a 95% level of confidence that the calculated Tolerance Interval (TI) correctly includes 99% of the response population. Coverage probability reflects the property of a long series of CI derived from a valid model (Greenland, 2016). In Table 7, the actual data fall within the range of the 95% lower and upper Prediction Intervals (PI), confirming that the optimum formulation with the highest desirability yields test results consistent with the software's predictions (Ambarwati, 2023).

CONCLUSION

This study demonstrated that silica gel adsorption is highly effective in increasing ethanol purity, with the highest measured concentration of 99.8% obtained using an alcoholmeter at a flow rate of 0.1 ml/s and 60 grams of adsorbent. Optimization using Response Surface Methodology (RSM) predicted a slightly higher ethanol concentration of 99.808% at a flow rate and adsorbent weight significantly affect the purification process. The application of silica gel provides an environmentally friendly and efficient alternative to conventional chemical separation methods, making it promising for industrial scale bioethanol purification. Future studies are recommended to evaluate the performance of alternative adsorbents such as zeolite or activated carbon, and to explore integration with other separation techniques to further improve process efficiency and reduce operational costs.

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