

Optimization of Poly Aluminum Chloride Liquid Dosage in Clean Water Treatment Using Box-Behnken Design

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Abstract

Various human activities that are increasing will have an impact on the decline in the quality of raw water, a key source of clean water production. To achieve optimal results in the clean water treatment process, a suitable formulation is required for the addition of liquid PAC, which acts as a coagulant to reduce the turbidity level of raw water. Currently, the formulation of liquid PAC dosage in raw water treatment has an accuracy level of less than 70%. Therefore, a method is needed to improve the accuracy level of PAC dosage. Box-Behnken Design (BBD) is one of the RSM design methods, which can analyze problems whose responses are influenced by several variables, where the interaction can be used to predict the optimal formula. This study aims to determine the optimal dosage of liquid PAC required for clean water treatment using the Box-Behnken Design. Before the optimization process is carried out, it is necessary to select parameters that have a significant effect. The methodology in this study is divided into two main stages. The first stage involves parameter selection using Design-Expert Two-Level Factorial, with five parameters tested: liquid PAC dosage, turbidity, pH, stirring speed, and stirring time. The second stage involves optimizing parameters that have a significant effect using Design-Expert Box-Behnken. The results obtained from the parameter selection process indicate that turbidity and liquid PAC dosage have a significant effect. Optimization is carried out by entering the turbidity parameters and liquid PAC dosage as independent variables, with the response being the final turbidity. The results show that in water with turbidities of 20 NTU, 60 NTU, and 100 NTU, the optimum liquid PAC concentration is 50 ppm, with percentage reductions in turbidity of 95.72%, 97.13%, and 97.37%, respectively. BBD shows that there is a significant parameter interaction between the turbidity parameter and the liquid PAC dosage, while there is no significant interaction between the turbidity parameter and pH, and the pH parameter and the liquid PAC dosage.

Keywords: Box-Behnken Design; Design-Expert; optimization; Poly Aluminium Chloride; turbidity

1 Introduction

Human activities, from primary to secondary needs, require water. In everyday life, it is used as a necessity in both households and industries, thereby increasing the need for clean water. However, the increasing and diverse human activities will have an impact on the decline in water quality. Domestic and industrial activities that produce pollution cause a decrease in water quality values [1]. To overcome this problem, various efforts have been made to improve water quality, ensuring that clean water is suitable for use. One of these efforts is the existence of Clean Water Treatment Plants.

Clean Water Treatment Plants in Indonesia generally currently apply conventional water treatment technology, where water undergoes coagulation, flocculation, sedimentation, filtration,

and disinfection. The coagulation process begins with the addition of chemical coagulants, namely Poly Aluminum Chloride (PAC), Aluminum Sulfate, and alum, which function to reduce pollutants and decrease water turbidity. One of the coagulants commonly used in Clean Water Treatment Plants is PAC (Poly Aluminum Chloride) liquid.

PAC or Poly Aluminum Chloride is an inorganic coagulant composed of macromolecular polymers with several advantages, namely having a strong adsorption rate, high floc formation rate even in low doses, fast sedimentation rate, is a water clarifying agent with high efficiency, fast in the process, safe and effective in use at low concentrations. As an alternative to solid alum, PAC offers superior performance in the coagulation process. PAC can quickly collect colloidal particles

in water, regardless of the turbidity level, resulting in denser and more easily separable deposits. Additionally, PAC produces less aluminum residue in the treated water, resulting in improved water quality [2]. However, the use of coagulants in this process must be precise, not too little or too much. This is due to increased turbidity, alkalinity, and organic content that require adjustments to higher coagulant doses. Therefore, to achieve better results, a correct formulation is necessary when using PAC.

Generally, the addition of PAC dosage in water treatment uses the Jar-Test method, as has been done by several researchers, including the optimization of Aluminum Sulfate and PAC coagulant dosage at PDAM Tirta Musi Palembang using the Jar-Test method [3]; optimization of PAC addition and stirring time on raw water quality at PPSDM Migas Cepu using the Jar-Test method [1]; and optimization of the use of Alum and Poly Aluminum Chloride (PAC) coagulant dosage in coal liquid waste management using the Jar-Test method [2].

Based on research conducted, the parameters that have a significant relationship with the PAC dosage are TSS and pH. The addition of PAC dosage reduces the TSS value by 98.88% and the pH value by 16.41% [2]. A significant relationship exists between the addition of PAC dose and turbidity, indicating that increasing the PAC dose reduces the turbidity value in water by 93.99% [3]. PAC is efficient in lowering TDS values by 53.59-55.5% [4]. This Jar-Test method is also carried out in Clean Water Installations on a laboratory scale; however, the Jar-Test method has an accuracy level of less than 70%.

In the dose optimization process, the optimal formula is determined by evaluating data from the prepared formulations. In the optimization process, the most optimal combination of product or process characteristics will be sought under certain conditions [5]. Design-Expert is a software that uses statistical methods to assist in the implementation of experimental designs in determining dose optimization with a predetermined formulation. However, before the optimization process is carried out, selecting the parameters is necessary. In this study, parameter selection was carried out using Design-Expert Two-Level Factorial to identify parameters that significantly influence the dose of coagulant added. After obtaining significant parameters, optimization was carried out using Design-Expert Box-Behnken Design.

Box-Behnken Design (BBD) is a Response Surface Methodology (RSM) design method that analyzes problems whose responses are influenced by various variables, where the interaction of each variable determines the interaction between factors. This interaction is then used to predict the optimal

formula with mathematical calculations [6]. The mathematical model produced by the RSM method determines the relationship between independent variables (factors) and dependent variables (responses) [7].

Box-Behnken design is a type of experimental design used to model quadratic (second-order) relationships. This design is based on a factorial design with three levels (-1, 0, +1), where the middle level (0) is used to generate quadratic model terms and analyze second-order interaction effects among the factors [7]. The Box-Behnken Design (BBD) method is used to optimize three independent variables. BBD is more efficient because it requires fewer trials or runs. Despite the smaller number of trials, BBD is still able to accurately predict the optimum value for both linear and quadratic relationships [8]. BBD is widely used to model or optimize responses influenced by one or more other quantitative factors [9]. The BBD method ensures that not all factors are set at high levels simultaneously, and this method identifies significant interaction effects in each trial [10].

In this study, the optimization of the liquid PAC dose in the coagulation process of clean water treatment will be conducted using the Design-Expert Box-Behnken method. The interaction between the optimal dose of liquid PAC and the parameters of pH, turbidity, stirring speed, and stirring time will be investigated.

2 Method

2.1 Material

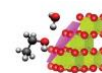
The research materials used include water, distilled water, mud, and PAC liquid (p.a Merck).

2.2 Instrumentation

The tools used in this study included Design-Expert v.13 software, a Portable pH meter (Mettler Toledo Seven2Go), a Portable Turbidimeter (HACH 2100Q), and other glassware available in the analytical chemistry laboratory.

2.3 Procedure

The raw water sample was then placed in a 1,000 ml beaker, and a 50 ppm PAC solution was added. The mixture was stirred at 120 rpm for 1 minute. During the flocculation process, the stirring speed was reduced to 50 rpm for 5 minutes. The stirring speed was reduced to 20 rpm for 5 minutes. Then, the stirring speed was reduced to 0 rpm, and the mixture was left to settle for 15 minutes, allowing the flocs to settle to the bottom of the beaker. This procedure was repeated for samples with the addition of PAC solution concentrations of 60 ppm, 70 ppm, 80 ppm, 90 ppm, 100 ppm, 110 ppm, and 120 ppm, respectively.



2.3.1 Parameter Selection Using Design-Expert Two-Level Factorial

Parameter selection was carried out using the Design-Expert version 13 application with the Two-Level Factorial method. The parameters tested for their effect on the optimization process were the PAC liquid dosage, turbidity, pH, stirring speed, and stirring time, as evaluated in **Table 1**. These parameters were then processed using Design-Expert Two-Level Factorial to obtain standards and experiments by the design results presented in **Table 2**.

2.3.2 Liquid PAC Optimization Using Box-Behnken Design

PAC liquid optimization was performed using the Design-Expert version 13 application with the BBD method. After obtaining the significantly influential parameters, the data were processed into BBD. The significantly influential parameters, namely PAC liquid dosage, turbidity, and pH, were evaluated as shown in **Table 3**. The data were then further tested using ANOVA to determine the significance of the resulting model.

Table 1 Variable values entered the Two- Level Factorial

Independent Variables	Code	Unit	Level	
			Low	High
PAC liquid dosage	A	ppm	0	100
Turbidity	B	NTU	3	100
pH	C	-	7	8
Stirring Speed	D	rpm	20	120
Stirring Time	E	minute	5	30

3 Result and Discussion

3.1 Parameter Selection Using Design-Expert Two-Level Factorial

The parameter selection process, which significantly influences dose optimization, begins by entering five parameters into the Design-Expert Two-Level Factorial. The five parameters are listed in **Table 4**. Specifically, the PAC liquid dose, turbidity, pH, stirring speed, and stirring time were set at their minimum and maximum values for each parameter.

The responses in **Table 4** are presented in the form of water turbidity following the Jar Test. The responses were then calculated to determine the percentage reduction in turbidity levels, allowing for comparison of the standard experiments to identify the one with the highest percentage reduction. The responses obtained in the form of final turbidity were then entered into the design results of the parameter selection process and processed into a Pareto diagram, as shown in **Fig. 1**.

The Pareto diagram in **Fig. 1** shows that parameter B, namely turbidity, has a highly significant effect on the optimization of the PAC liquid dose, with the height of parameter B bar exceeding the Bonferroni limit line. Parameter A, namely the PAC liquid dose, has a significant effect, indicated by the height of the bar exceeding the t-value limit line. The height of the bar exceeding the Bonferroni limit line suggests that the factor has a significant and absolute influence. In contrast, the height of the bar exceeding the t-value limit line indicates that the factor has a significant impact [11]. Parameters pH, stirring speed, and stirring time do not have a significant influence on the optimization of the liquid PAC dose, as indicated by the height of the bar for each parameter in the Pareto diagram not exceeding the t-value limit line.

The turbidity parameter in the Pareto diagram is shown in orange, indicating a positive effect. In contrast, the PAC liquid dosage in the Pareto diagram is shown in blue, indicating a negative impact. This suggests that when the positive effect is at a higher level, the dosage optimization process is more effective. The dose will increase if the adverse effects are at a higher level; then, the dose optimization process will decrease [12]. It can be concluded that the higher the turbidity, the higher the process dose optimization will be, while the higher the dose of PAC liquid, the lower the dose optimization process will be.

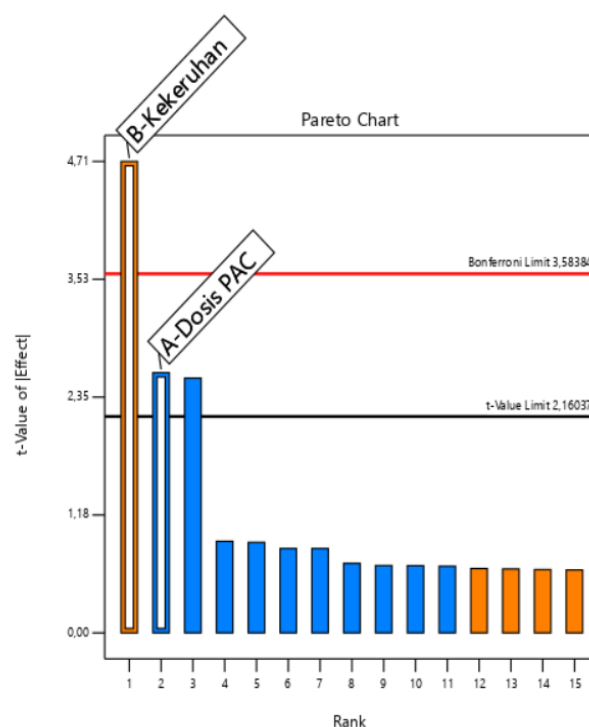


Figure 1. Pareto diagram of parameter selection with Two-Level Factorial

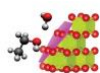


Table 2 Response data specified in the selection of PAC liquid dose determination parameters.

Std	Run	A	B	C	D	E
		PAC liquid dosage (ppm)	Turbidity (NTU)	pH	Stirring Speed (rpm)	Stirring Time (minute)
11	1	0	100	7	120	30
3	2	0	100	7	20	5
7	3	0	100	8	20	30
1	4	0	3	7	20	30
2	5	100	3	7	20	5
16	6	100	100	8	120	30
8	7	100	100	8	20	5
9	8	0	3	7	120	5
4	9	100	100	7	20	30
12	10	100	100	7	120	5
14	11	100	3	8	120	5
13	12	0	3	8	120	30
15	13	0	100	8	120	5
5	14	0	3	8	20	5
6	15	100	3	8	20	30
10	16	100	3	7	120	30

Table 3 The values of the variables entered into the Box-Behnken

Independent Variables	Code	Unit	Level		
			Low (-1)	Mid (0)	High (+1)
PAC liquid dosage	A	ppm	10	50	120
pH	B		7	7.5	8
Response Variable		Limitation			
Final Turbidity Value		NTU	Minimized		

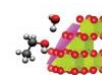
Table 4 Response data obtained in the selection of parameters for determining the dose of PAC liquid.

Std	Run	A	B	C	D	E	Response	Percentage of Turbidity Reduction
		PAC liquid dosage (ppm)	Turbidity (NTU)	pH	Stirring Speed (rpm)	Stirring Time (minute)		
11	1	0	102	7	120	30	101	0.98%
3	2	0	101	7	20	5	100	0.99%
7	3	0	101	8	20	30	100	0.99%
1	4	0	3.05	7	20	30	2.86	6.23%
2	5	100	3.41	7	20	5	3.37	1.17%
16	6	100	104	8	120	30	7.31	92.97%
8	7	100	102	8	20	5	90.6	11.18%
9	8	0	2.78	7	120	5	2.70	2.88%
4	9	100	97.2	7	20	30	8.24	91.52%
12	10	100	104	7	120	5	20.4	80.38%
14	11	100	3.15	8	120	5	3.12	0.95%
13	12	0	3.22	8	120	30	3.20	0.62%
15	13	0	98.6	8	120	5	97.6	1.01%
5	14	0	2.93	8	20	5	2.78	5.12%
6	15	100	3.14	8	20	30	1.71	45.54%
10	16	100	3.29	7	120	30	0.80	75.68%

3.2 Liquid PAC Optimization Using Box-Behnken Design

The results of the parameter selection process, using the Two-Level Factorial method, indicate that

the parameters with significant influence are turbidity and PAC liquid dosage. The selected parameters become independent variables in the optimization process. The pH parameter in the



optimization process is not included as an independent variable, but rather as a standard for clean water, which is specified in PERMENKES No. 2 of 2023, with a range of 6.5–8.5.

3.3 Effect of PAC Liquid Dosage on Dose Optimization Process in Clean Water Treatment

Optimization of the liquid PAC dose was performed 17 times, as outlined in **Table 5**. The sample turbidity was varied to represent the fluctuating raw water turbidity value with the sample pH measured in actual values in the pH range of 7–8, the turbidity was set at 20 NTU, 60 NTU and 100 NTU, and the liquid PAC dose was set in stages starting from 10, 35, 50, 85, and 120 ppm, as listed in **Table 5**.

Table 5. Optimization of PAC liquid dose with BBD

Std	Run	A		C
		Turbidity (NTU)	PAC liquid dosage (ppm)	
6	1	101	85	7
10	2	59.7	120	7
2	3	101	50	7.5
8	4	101	10	8
4	5	101	120	7.5
3	6	21.5	120	7.5
5	7	21.5	85	7
16	8	101	35	7.5
12	9	59.6	10	8
15	10	59.6	35	7.5
17	11	19.3	10	7.5
1	12	21.5	50	7.5
13	13	60	85	7.5
9	14	19.3	35	7
11	15	60	50	8
14	16	57.9	85	7.5
7	17	21.5	85	8

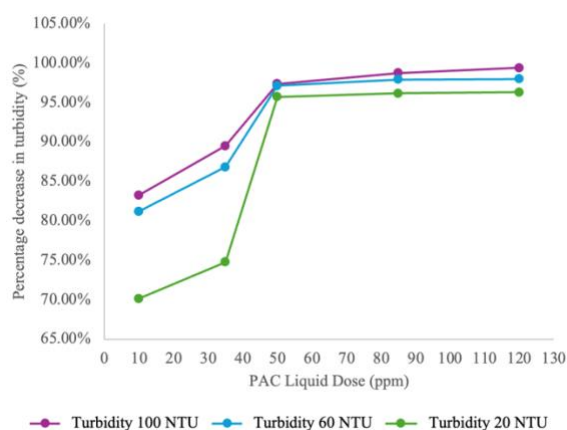


Figure 2. Curve of percentage decrease in turbidity against PAC liquid dose at turbidity of 20 NTU, 60 NTU, and 100 NTU.

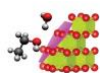
The response recorded in each experiment was the final turbidity. The calculated response was then the percent decrease in turbidity value, indicating the optimal dose, as shown in **Table 6**. Next, a curve of the percent decrease in turbidity against the dose of PAC liquid was created. The curve of the percent decrease in turbidity against the dose of PAC liquid was plotted separately for each turbidity value to determine the optimal dose range at each turbidity level, as shown in **Fig. 2**.

Table 6 shows the percentage decrease in turbidity. The higher the PAC dose, the greater the percentage decrease in turbidity, with the final pH value in each sample ranging from 6.5 to 8.5. The optimum liquid PAC dose, at a concentration of 50 ppm, with a sample turbidity of 20 NTU, achieved a turbidity reduction rate of 95.72%. For a sample turbidity of 60 NTU, the turbidity reduction rate was 97.13%, and for a sample turbidity of 100 NTU, the turbidity reduction rate was 97.37%. The addition of higher concentrations of PAC liquid, specifically 85 and 120 ppm, results in a percentage decrease in turbidity that is almost constant or not significantly different from the addition of a 50 ppm concentration. Therefore, it can be concluded that the optimum dose is between 50 ppm and 120 ppm. Increasing PAC doses does not significantly decrease the percentage of turbidity. This occurs because increasing the PAC dose has a negative effect; if the negative effect is at a higher level, it will reduce the dose optimization process [12].

3.4 Data Distribution Model Graph

The data that has been processed in BBD will produce a model graph that shows the model from the processed data has good accuracy and suitability with the design predictions, the model graph resulting from the design is in the form of a standard probability plot against the residual, a residual plot against the prediction and a prediction plot against the actual response to the optimization of the liquid PAC dose, shown in **Fig. 3**.

Fig. 3a shows a normal probability distribution of the residuals. The distribution of data approaching the regression line indicates that the data has a good fit, where each data point is along the straight regression line. Data points closer to the regression line indicate a good fit, assuming a normal distribution [11]. **Fig. 3b** shows a plot of residual values against predictions. The data in the graph appears to be randomly distributed and does not exceed the upper and lower limits, confirming the model's accuracy. Data that falls within the upper and lower limits indicates that the model's accuracy can be confirmed [13]. **Fig. 3c** shows the predicted value against the actual; the data in the graph is spread close to the reference line, indicating that the



model has good accuracy. Data that is well distributed, approaching the reference line, is assumed to indicate that the model has high accuracy [13].

Table 6 Percentage variation of turbidity reduction at turbidity values of 20 NTU, 60 NTU, and 100 NTU.

Turbidity (NTU)	pH	actual pH	Actual Turbidity (NTU)	PAC Dose (ppm)	Final turbidity	final pH	Percentage reduction in turbidity (%)
20	7.5	7.3	19.3	10	5.76	7.2	70.16%
	7	7.3	19.3	35	4.87	7.2	74.77%
	7.5	7.4	21.5	50	0.92	7.2	95.72%
	7	7.4	21.5	85	0.82	7	96.19%
	7.5	7.4	21.5	120	0.79	6.8	96.33%
60	8	7.3	60	10	11.2	7.1	81.21%
	7.5	7.3	60	35	7.84	7.1	86.85%
	8	7.3	60	50	1.72	7.1	97.13%
	7.5	7.3	60	85	1.25	6.9	97.92%
	7	7.3	60	120	1.19	6.8	98.01%
100	8	7.3	100	10	16.9	7.1	83.27%
	7.5	7.3	100	35	10.6	7	89.50%
	7.5	7.5	100	50	2.66	7.2	97.37%
	7	7.5	100	85	1.25	7.4	98.76%
	7.5	7.5	100	120	0.59	7	99.42%

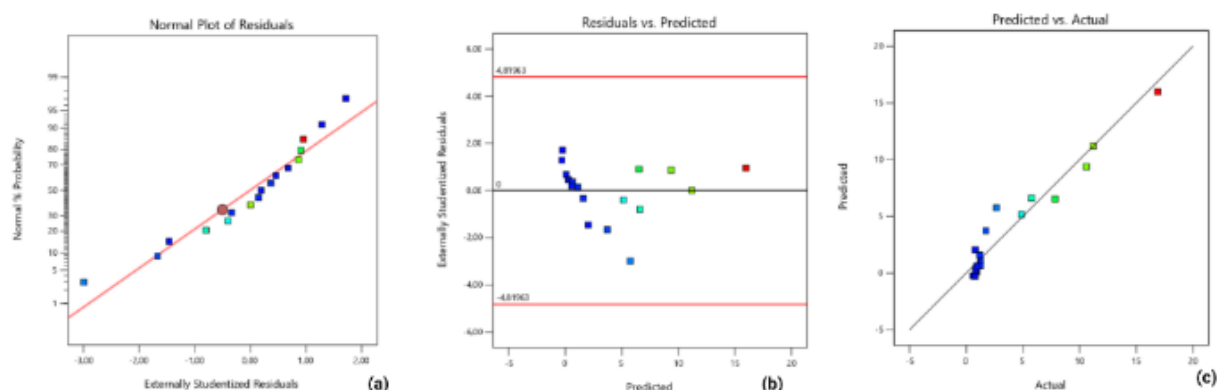


Figure 3 Diagnostic plot values of (a) normal probability against residual, (b) residual against prediction, (c) prediction against actual of the response to PAC liquid dose optimization.

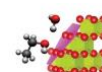
3.5 Interaction Between Each Parameter

The Box-Behnken design can illustrate the interaction between two parameters in a contour plot and a 3D surface. This plot illustrates the effect of the interaction between two parameters in the process of optimizing the liquid PAC dose.

Fig. 4 shows the 3D surface plot and contour of the interaction between two parameters with other parameters held constant [14]. It can be seen from each interaction color on the contour and 3D surface that the color indicates that the interaction occurs at a minimum value [11]. The elliptical contour on the graph indicates an interaction between independent variables [15].

Fig. 4a depicts the interaction between the PAC dose and liquid with turbidity on the final

turbidity value. The 3D surface shows an interaction at optimum conditions, where the percentage of turbidity reduction does not differ significantly at a dose concentration of 50 ppm to 120 ppm. **Fig. 4b** illustrates the interaction between pH and turbidity on the final turbidity value. The turbidity parameter does not exhibit a significant interaction with pH, as evidenced by the lack of change in the 3D surface, which is also reflected in the p-value of both parameters, which is greater than 0.05 (**Table 7**). **Fig. 4c** shows the interaction between the pH parameter and the PAC dose on the final turbidity value. The PAC dose parameter with pH did not exhibit a significant interaction, as indicated by the addition of the PAC dose not significantly altering the pH



on the 3D surface. This is demonstrated in **Table 7**, where both parameters have a p-value greater than 0.05.

3.6 ANOVA Statistical Model

ANOVA of the model will indicate whether the model is significant or not. The significance of the model is seen from the p-value in the ANOVA. In **Table 7**, the p-value of 0.0020, which is less than 0.05, indicates that the model is significant. Adequate precision measures the signal-to-noise ratio; in this study, the ratio was 11.571, a ratio greater than 4, indicating that the model is acceptable and valid [16].

To test the interaction of each parameter, a hypothesis test is used. If the p-value is > 0.05 , then H_0 is accepted and H_1 is rejected, whereas if the p-value < 0.05 , then H_0 is rejected and H_1 is accepted, with the assumption that each hypothesis is:

H_0 : both parameters do not have a significant relationship with the optimization of the liquid

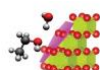
PAC dose (p-value > 0.05)

H_1 : Both parameters have a significant relationship with the optimization of liquid PAC dose (p-value < 0.05)

Based on **Table 7**, a significant interaction is observed between the turbidity parameters and the PAC liquid (AB) dose, with a p-value of 0.0186, indicating that H_0 is rejected and H_1 is accepted. This indicates the effect of PAC dose on the percentage decrease in turbidity; the higher the concentration of the liquid PAC dose, the greater the percentage decrease in turbidity. The interaction between turbidity and pH (AC) has a p-value of 0.6849, so H_0 is accepted and H_1 is rejected. This suggests that the turbidity parameter and pH do not have a significant relationship. The interaction between the liquid PAC dose parameter and pH (BC) has a p-value of 0.3105, so that H_0 is accepted and H_1 is rejected. This indicates that the PAC dose parameter and pH do not have a significant relationship.

Table 7. ANOVA statistical results of the response surface model for turbidity reduction.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	348.14	9	38.68	11.5	0.0020	significant
A-Turbidity	5.17	1	5.17	1.54	0.2552	
B-Dose PAC liquid	5.03	1	5.03	1.5	0.2609	
C-pH	0.0547	1	0.0547	0.0163	0.9021	
AB	31.3	1	31.3	9.31	0.0186	
air conditioning	0.6024	1	0.6024	0.1791	0.6849	
BC	4.02	1	4.02	1.2	0.3105	
A2	2.44	1	2.44	0.7258	0.4224	
B2	38.29	1	38.29	11.38	0.0119	
C2	3.57	1	3.57	1.06	0.3372	
Residual	23.55	7	3.36			
Total Cor	371.69	16				
Std. Dev.	1.83		R²		0.9366	
Mean	4.12		Adjusted R²		0.8552	
CV %	44.52		Predicted R²		0.4416	
			Adequate Precision		11,571	



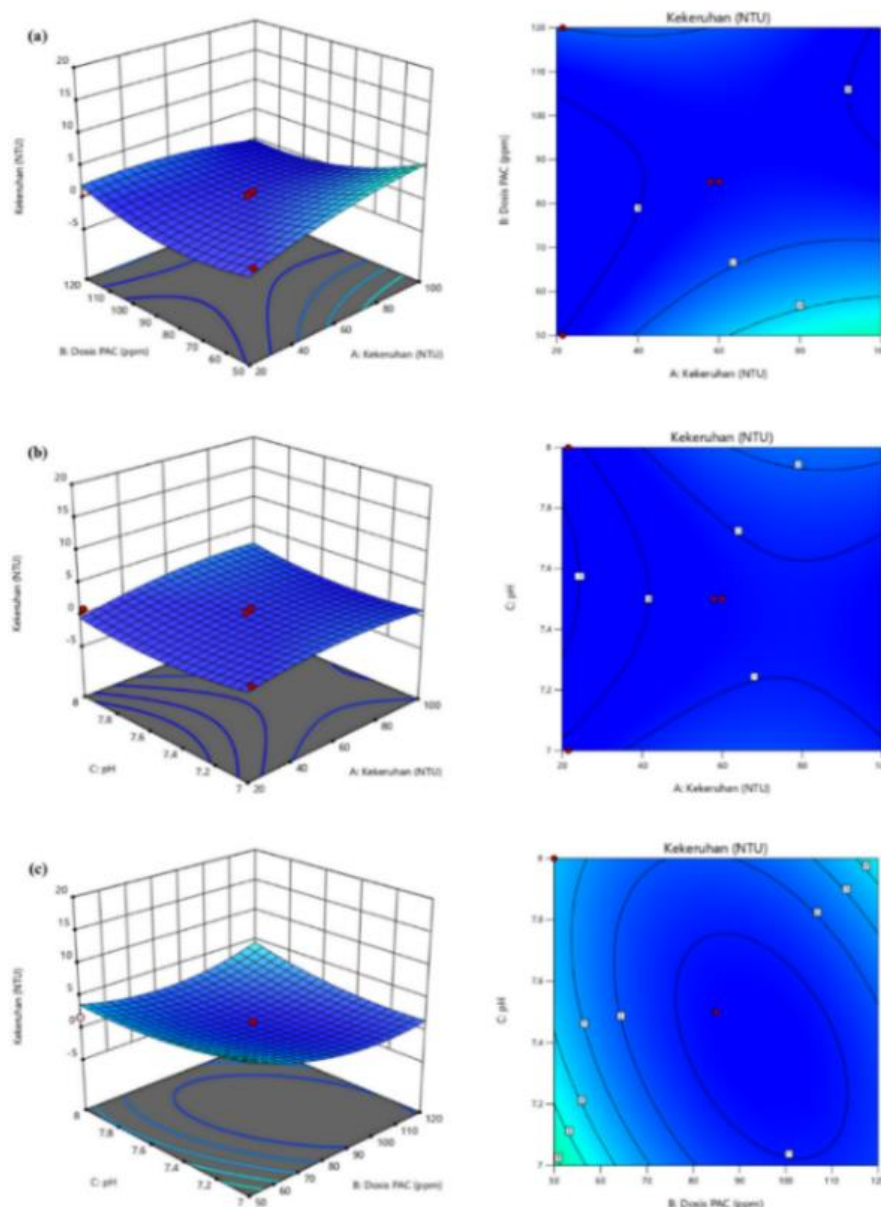


Figure 4. 3D Surface Plot and Contours

4 Conclusion

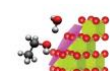
In this study, the BBD method was used to optimize the PAC liquid dose, samples were made in three different turbidities to represent the minimum level, namely 20 NTU, medium at 60 NTU and maximum at 100 NTU, the PAC liquid dose was optimum concentration was 50 ppm with a turbidity reduction percentage of 95.72%, 97.13%, and 97.37%, respectively. The BBD response revealed a significant interaction between two parameters: the turbidity parameter and the liquid PAC dose.

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