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Optimization of Cr(VI) Adsorption on Eugenol-Silica Gel Composites Using Behnken Box Design

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Abstract

This study aims to find the optimum conditions for the adsorption of Cr (VI) ion to the eugenol-silica gel composite as an adsorbent and to determine the adsorption parameters using the Langmuir and Freundlich isotherm model. The three optimization parameters analyzed were pH, contact time, and weight of the adsorbent of the eugenol-silica gel composite. The optimization experiment was designed using the Box Behnken Design Response Surface Methodology (RSM) on the MINITAB software. Then, the maximum adsorption capacity was evaluated using the Langmuir and Freundlich isotherm equation model. The results showed that the optimum conditions for the adsorption process were the weight of the eugenol-silica gel composite adsorbent of 0.15 grams, contact time of 35 minutes and pH of 5. The adsorption process under these conditions was favorable because the R_L obtained was 0.0297. The maximum absorption capacity of Cr (VI) was indicated by the q_m value of 1.0328 mg/g, and the K_F, K_L, and n values observed 0.7961, 1.0885 and 14.0056, respectively. Adsorption of Cr(VI) ion by eugenol-silica gel composite followed the Langmuir isotherm equation.

Keywords: *Adsorption Isotherm, Adsorption Optimization, Box Behnken, Eugenol-Silica Gel*

1 Introduction

Cr(VI) ion is one of the heavy metals found in textile, laboratory, glass ceramics, and leather tanning industrial wastes. The toxicity of the chemical compound Cr (VI) to aquatic organisms depends on its form, oxidation number, and pH. Cr(VI) almost all of its compounds are anionic, highly soluble in water and relatively stable even though these compounds are strong oxidizing agents in acidic solutions.

The level of Cr (VI) has threshold value of 0.002 mg/L which has been determined by the minister of the environment Number 51 of 2004. Meanwhile, if the amount of pollutant material such as Cr (VI) increases in high doses and has accumulated in the long term, it will be carcinogenic and cause problems for the environment and can cause disease if it enters the body of living things.

One way to remove heavy metals is through adsorption using an adsorbent. One of the materials that can function as an adsorbent is silica gel. Silica gel has various advantages including being hydrophilic, inert, having good thermal stability, and easy desorption process. Silica gel also has several weaknesses, such as the active site is only a silanol group and siloxane (-SiOH) which has low acidity and has oxygen as a weak electron pair donor atom, so it is less capable when applied as an adsorbent for certain metal ions. One way to increase the adsorption is by conducting various modification and characterization methods [1].

One of the studies that evaluated silica gel as an adsorbent in the adsorption process against waste problems was the optimization of contact time and pH in the synthesis of silica gel from corn cob ash to adsorb methylene blue [2]. The study stated that the highest adsorption by silica gel from corn cob ash took place at pH 3 which was 8.250 ppm with a percentage of 40.867% from the initial ppm of 20 ppm, while the contact time required for equilibrium was 10 minutes with an adsorbent weight of 0.1 grams. However, from this research, there is no further step whether the maximum adsorption capacity produced meets the Langmuir or Freundlich isotherm equation.

This study aims to study the adsorption of eugenol-silica gel composites. The eugenol-silica

gel composite was then applied as an adsorbent to heavy metal ions Cr(VI). The addition of eugenol to silica gel is done because the three functional groups in eugenol can attract metal ions that move freely and are expected to increase the adsorption effectiveness if combined into a composite, because it is predicted to further improve the quality of the silica gel itself.

2 Research Method

2.1 Tools and materials

The research, which was conducted at the Research Laboratory of the Faculty of Mathematics and Natural Sciences, Universitas Pakuan, Bogor, used equipment including: analytical balance, universal indicator, bulb pipette, beaker, watch glass, erlenmeyer, volumetric pipette, burette, beaker, spatula, tools - other supporting glass and plastic instruments, and a set of Genesys 10uv Scanning UV-Vis spectrophotometer models.

Meanwhile, the materials used in this study include the eugenol-silica gel composite in the form of xerogel and chemicals which include: The eugenol-silica gel composite obtained from previous studies [3], Sodium Hydroxide (NaOH) pa (Merck), Sulfuric Acid (H_2SO_4) pa (Merck), Phosphoric Acid (H_3PO_4) pa (Merck), Potassium Dichromate ($K_2Cr_2O_7$) pa (Merck), Nitric Acid (HNO_3) pa (Merck), 1,5-diphenylcarbazide ($C_{13}H_{14}N_4O$) (CAS No. 140-22-7), Acetone (C_3H_6O), distilled water, filter paper, Whatman paper No. 41, and universal pH indicator paper.

2.2 Preparation of Cr(VI) stock solution

Cr(VI) stock solution was prepared from potassium dichromate ($K_2Cr_2O_7$) with the desired concentration of 500 mg/L (ppm). The formula for making the stock solution is in accordance with SNI, namely by weighing 141.4 mg of $K_2Cr_2O_7$ and put into a 100 mL volumetric flask, then adding distilled water to the mark and homogenized.

2.3 Preparation of Cr(VI)-diphenylcarbazide Complex Working Solution

The working solution was made with one blank and in various concentrations, including: 10 ppm, 15 ppm, 20 ppm, 25 ppm, and 30 ppm. From these various levels, as much of the stock solution was pipetted as calculated in the dilution. The solution that has been pipetted is put into a 50 mL beaker, then add 0.125 mL (2.5 drops) of H_3PO_4 into each working solution and adjust the pH until the pH is at 2.0 ± 0.5 with the addition of 0.2 N H_2SO_4. Transfer the working solution into a 50 mL volumetric flask, adjust to the mark with distilled water. Add 1.0 mL of diphenylcarbazide solution, shake and let stand 5 to 10 minutes and the working solution is ready to be measured for absorption.

2.4 Determination of Cr(VI) Ion Content with UV-Vis Spectrophotometer

The initial concentration of Cr(VI) ion was determined by measurement using a UV-Vis spectrophotometer. Analysis of the initial levels of Cr(VI) ions in the Cr(VI)-diphenylcarbazide complex solution was carried out using standards at various concentrations, that is concentrations of 10 ppm, 15 ppm, 20 ppm, 25 ppm, and 30 ppm. The absorption of each working solution was then measured using a UV-Vis spectrophotometer at a maximum wavelength of 530 - 540 nm so that further data peaks could be obtained from the measurement results in mg/L (ppm) (recorded as Co). A calibration curve is made from the measurement data and determine the equation of a straight line so that the coefficient value of r 0.995 is obtained.

2.5 Optimization of Cr(VI) Ion Adsorption on Eugenol-Silica Gel Composites

The optimum conditions for the adsorption of Cr(VI) ions by the eugenol-silica gel composite were determined using three parameters, that is contact time, pH, and adsorbent weight. The pH optimization was carried out in the range of 3 - 7, then the contact time was 10 - 60 minutes, and the adsorbent weight was 0.10 - 0.20 grams. Eugenol-silica gel composite flakes were added to 50 mL of 10 ppm Cr(VI)-diphenylcarbazide complex solution, then the mixture was stirred under experimental conditions according to the Box Behnken experimental design under room temperature conditions and filtered using Whatman No. 41 to take the filtrate which is then measured using a UV-Vis spectrophotometer with a wavelength of 530 - 540 nm so that data can be obtained from the measurement results. Then graphed the optimum absorption to show the highest absorption and recorded the contact time, adsorbent weight, and pH which were used as the basis for further research.

2.6 Isothermal Adsorption of Eugenol-Silica Gel Composites against Cr (VI) Ions

The optimization data obtained from the experiment using the Box Behnken response surface, was used to test the maximum adsorption capacity using the Langmuir and Freundlich isotherm model. The metal ion content of Cr(VI) was measured in a complex solution of Cr(VI)-diphenylcarbazide made in various concentrations, that is concentrations of 10 ppm, 15 ppm, 20 ppm, 25 ppm, and 30 ppm which had been added to the eugenol-silica gel composite according to conditions that have been obtained from the previous optimum conditions. Then each solution was then filtered with Whatman No. 41 and the obtained filtrate was measured using a UV-Vis spectrophotometer to obtain peaks of data from the measurement results in mg/L (ppm). The concentration after treatment will be measured as Ce [4].

3 Result and Discussion

3.1 Optimization Results of Cr (VI) Ion Adsorption on Eugenol-Silica Gel Composites

Optimization of Cr(VI) ion adsorption by eugenol-silica gel composites has been carried out using three independent variables, that is adsorbent weight, pH, and contact time, while the dependent variable or response is the concentration of Cr(VI) ions from the test solution at equilibrium in the adsorbent so that the percent efficiency of the adsorption can be displayed. **Table 1** shows the percent adsorption efficiency data which was then used to determine the optimum conditions of the Cr(VI) ion adsorption process on the eugenol-silica gel composite.

Optimum conditions are conditions where the solution can be absorbed maximally by an adsorbent. In the adsorption process, the adsorption capacity of a pollutant is not only affected by pH, contact time, and the weight of the adsorbent but is also influenced by the concentration of the solution used. Therefore, in the application of the eugenol-silica gel composite as an adsorbent in the adsorption of metal ions Cr (VI) is determined in a solution with a concentration of 10 ppm to obtain the optimum conditions that can be adsorbed by the adsorbent to obtain maximum and effective results.

Table 1. Data on the Percentage of Cr (VI) Ion Adsorption Efficiency on Eugenol-Silica Gel Composites using Behnken's Response Surface Box

No	Time (minute)	pH	Weight adsorbent (g)	Adsorption Efficiency (%)
1	35	5	0.15	25.03
2	60	7	0.15	1.75
3	35	7	0.10	2.85
4	35	5	0.15	24.44
5	10	5	0.10	12.95
6	10	7	0.15	1.77
7	60	3	0.15	2.52
8	60	5	0.20	6.99
9	10	3	0.15	3.63
10	60	5	0.10	9.87
11	35	7	0.20	1.70
12	35	5	0.15	28.94
13	35	3	0.10	9.78
14	35	3	0.20	3.45
15	10	5	0.20	7.30

The analysis carried out on the response surface data includes analysis of the estimated regression coefficients and analysis of variance. The analysis was carried out using the MINITAB software. The results of the analysis of variance showed that the adsorption of Cr (VI) ions by the eugenol-silica gel composite followed a linear and square pattern, meaning that the interaction was based on the magnitude of the P value (P-Value 0.000) which was smaller than the significance value ($\alpha = 0.050$).

Table 2. Analysis of Variance of Adsorption of Cr (VI) Ions on Eugenol-Silica Gel Composites using RSM Box Behnken

Source	F-value	P-value
Model	37,920	0.000
Linear	20,250	0.003
Contact Time	0.520	0.503
pH	0.090	0.249
Adsorbent Weight	58,540	0.001
Square	108,120	0.000
Contact Time * Contact Time	94,620	0.000
pH * pH	215,110	0.000
Adsorbent Weight * Adsorbent Weight	57,970	0.001
2-Way Interaction	0.058	0.369
Contact Time * pH	0.090	0.543
Contact Time * Adsorbent Weight	0.550	0.341
pH * Adsorbent Weight	0.101	0.161

In the linear model, the P-Value value of the adsorbent weight is 0.001 which is smaller than the significance value which indicates that the variation in the control of the adsorbent weight has a large effect on the test. While the pH contact time has a P-Value of 0.503 and 0.249, respectively or more than ($\alpha = 0.050$).

In the square interaction model, all parameter variations (contact time, pH, and adsorbent weight) have a P-Value value of 0.000, 0.000 and 0.001 respectively, which means that H_1 or the model with one factor can be accepted because it has a significant effect and provides evidence stating that the independent variable of the hypothesis has a significant effect on the adsorption study. While H_0 is rejected or can be said to have no significant effect on the adsorption study [5].

The variables of contact time, pH, adsorbent weight, and response are symbolized as X_1, X_2, X_3, and Y respectively. Then the estimation of the regression coefficient based on the analysis using the response surface model can be concluded that follows the equation:

$$Y = - 12.41 + 0.924\ X_1 + 3.276\ X_2 + 76.4\ X_3 - 0.001509\ X_1^2 - 0.3556\ X_2^2 - 295.4\ X_3^2 + 0.00055\ X_1X_2 + 0.0554\ X_1X_3 + 1.267\ X_2X_3$$

The equation above shows that: if the other variables are constant then the Y value will change automatically by the constant value, that is -12.41; if other variables are constant, the Y value will change by 0.924 for every one X1 unit; if other variables are constant, the Y value will change by 3,276 for every X_2 unit; if the other variables are constant then the Y value will change by 76.4 every one unit of X_3. The quadratic variables (X_1^2, X_2^2 and X_3^2) have a minus sign indicating the meaning that the quadratic variable has an antagonistic effect on the response, while the linear and interaction variables (X_1X_2, X_1X_3, X_2X_3) show a synergistic effect on the response as seen from the positive sign on the coefficient number [6].

Based on the F-Value test, it can be concluded that the F test in regression functions as a simultaneous test, that is to determine whether simultaneously all independent variables have a significant influence on the dependent variable and can be seen from the F test value. The analysis model of the response surface predicts that it can be said X variable together has a significant contribution to the Y variable [5].

The magnitude of the effect of the interaction between the two independent variables on the response that has been described is then presented in the form of contours in **Figure 1**. Contour plots are used to help determine critical points on the Surface Plot.

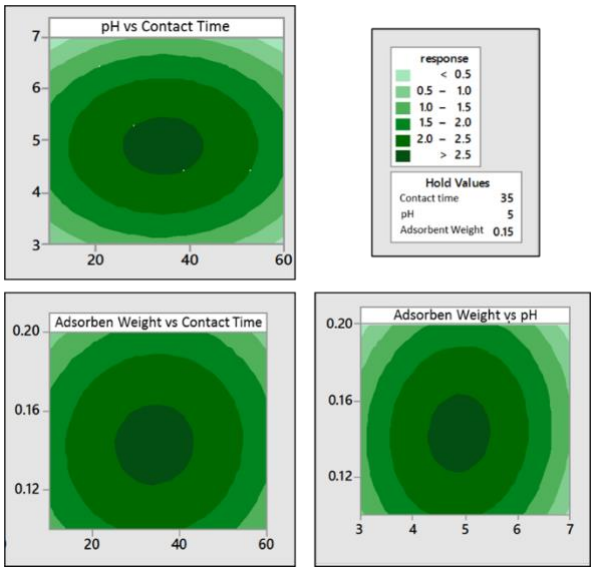

Figure 1. Plot Contour of the Adsorption Surface Response of Cr(VI) Ions on Eugenol-Silica Gel . Composites

Figure 1 shows that in general the optimal response area from the influence of contact time, pH, and adsorbent weight is in the middle of each minimum and maximum value. This indicates that the condition of Cr (VI) which is absorbed by the eugenol-silica gel composite has reached equilibrium.

The independent variable of contact time presented in **Figure 1** shows that in general the optimal response region is produced under medium conditions with a contact time of about 35 minutes, pH generally indicates the optimal region is produced at pH 5, and the weight of the adsorbent is 0.15 gram.

The time required to reach the equilibrium state is called the optimum contact time. At that time the adsorption reaction equilibrium has occurred because the adsorbate molecules have entered the pores of the adsorbent. Increasing the contact time can indeed increase the adsorption rate, but at the contact time that exceeds the equilibrium limit it can cause a decrease in the active adsorption center which causes fewer metal ions to be adsorbed and results in a decreased response or no desorption has occurred [6].

Likewise, the higher the pH, if there is a decrease in absorption at alkaline pH, it is possible

to form metal hydroxide complexes which will reduce the effectiveness of adsorption. The optimal region is generated when the test is at pH 5 or acid. At acidic pH conditions, there is a competition between analyte ions and H^+ ions to interact with functional groups on the surface of the adsorbent.

Based on the theory of HSAB (Hard Soft Acid Base) the tendency of the reaction will be easier for the interaction between an acid and a hard-hard and soft-soft base compared to a hard-soft mixture in a reactant [7]. The eugenol-silica gel composite which is soft base has better adsorption ability because the eugenol-silica gel composite has a relatively large surface size and is easily polarized and can bind to Cr (VI) metal ions in a solution with a weak acid pH. If the weight of the adsorbent is too much or excessive, the adsorption capacity will decrease which causes the adsorbent of the eugenol-silica gel composite to no longer occur in an equilibrium state on the adsorbate so that absorption does not occur optimally and must be absorbed by the new eugenol-silica gel composite.

Based on the results of the Behnken response surface box experiment, it was decided to use the optimum conditions set by the response optimizer on the Minitab application with the results of a contact time of 35 minutes, pH of 5, and adsorbent weight of 0.15 grams. This decision was taken because the estimated adsorption of Cr(VI) ions by the eugenol-silica gel composite under these conditions would obtain the greatest adsorption power.

3.2 Isothermal Adsorption of Cr(VI) Metal Ions by Eugenol-Silica Gel . Composites

Isothermal adsorption was carried out to determine the effect of the initial concentration of Cr (VI) ions on the amount of Cr (VI) ions that have been adsorbed by the adsorbent (eugenol-silica gel composite) at an optimum condition and the form of interaction that occurs between the adsorbent and the adsorbate. The relationship curve between the initial concentration of Cr(VI) ion and adsorption capacity is presented in Figure 2.

Based on Figure 2, at first the curve rise significantly and then tended to be constant. This shows that the adsorption capacity of Cr(VI) ions increases with the increase in the initial concentration of Cr(VI) ions. The curve tends to be constant at a higher initial concentration because the surface of the eugenol-silica gel composite has been saturated.

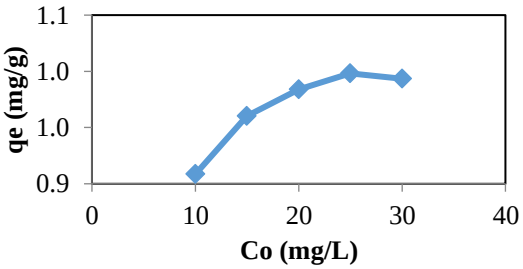

Figure 2. Relationship Curve between Adsorption Capacity and Initial Concentration of Cr (VI) Ions

The maximum adsorption of Cr (VI) metal ions that can be absorbed by the eugenol-silica gel composite when tested is 27.26 % at a concentration of 10 ppm with a solution volume of 50 mL. The adsorption process is closely related to the active site where the adsorbate attaches to the adsorbent. The number of active sites is proportional to the surface area of the adsorbent. The increase in the concentration of adsorbate can increase the number of substances that can be adsorbed [8].

Table 2. Langmuir and Freundlich Ion Cr (VI) Adsorption Isothermal Analysis Data on Eugenol-Silica Gel Composites

Ce	qe	Isothermal Analysis			
		Langmuir		Freundlich	
		Ce	Ce/qe	Ce logs	qe logs
7.2745	0.9085	7.2745	8.007	0.8618	-0.0417
12.1190	0.9603	12.1190	12,619	1.0835	-0.0176
17.0490	0.9837	17.0490	17,332	1.2317	-0.0072
22.0055	0.9982	22.0055	22,046	1.3425	-0.0008
27.0195	0.9935	27.0195	27.196	1.4317	-0.0028

Information:
Ce : Concentration of Cr(VI) ions at equilibrium in solution (mg/L or ppm)
qe : Adsorption capacity (mg/gr)

The isothermal analysis of Cr(VI) metal ion adsorption by the eugenol-silica gel composite was carried out using two models, namely the Langmuir and Freundlich models. For the analysis of the Langmuir model, it is done by making a linear equation between Ce and Ce/qe, while for the analysis of the Freundlich model, it is done by making a linear equation of Log Ce to Log qe. The data from the Langmuir and Freundlich isotherm analysis are presented in Table 2.

Based on the data in Table 2, the Langmuir and Freundlich isothermal curves are then drawn

up which are presented in Figures 3a and 3b, respectively.

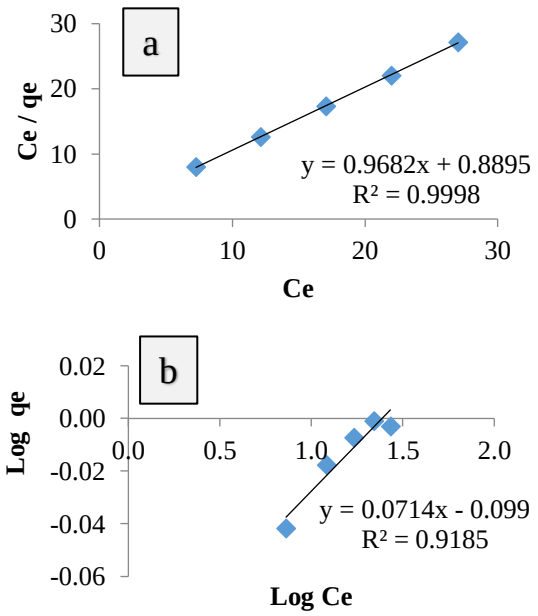

Figure 3. Langmuir (a) and Freundlich (b) Isothermal Analysis Curves Adsorption of Cr (VI) Ions on Eugenol-Silica Gel Composites

The isothermal parameter values (qm, K_L, R_L, K_F, and n) can be determined based on the straight-line equation obtained from the Langmuir and Freundlich isothermal adsorption curves which are presented in Table 3.

Table 3. Parameters of Langmuir and Freundlich Adsorption Isothermal Cr (VI) Ions on Eugenol-Silica Gel Composites

Langmuir			
q_m (mg/gr)	K_L	R_L	R^2
1.0328	1.0885	0.0297	0.9998
Freundlich			
n	K_F	R^2	
14.0056	0.7961	0.9185	

Based on the data in Table 3, the R^2 value in the Langmuir model is greater than the Freundlich model, so that the adsorption properties of this composite follow the Langmuir equation. The maximum adsorption ability of the eugenol-silica gel composite in the Langmuir equation is indicated by the qm value of 1.0328 mg/g, this means that every 1 gram of the eugenol-silica gel composite can absorb 1.0328 mg Cr (VI) in a 50 mL solution. The relationship between the amount of adsorbate that can be adsorbed and the concentration of the adsorbate in the solution at a constant state and temperature and is expressed by the Langmuir adsorption isotherm model means that absorption can only occur in one layer or monolayer [9].

Another parameter that can be related to an adsorption process is R_L. This R_L is one of the separation factors to state that the adsorption process is not profitable if the value of $R_L > 1$; if the adsorption process is linear then the R_L value is = 1; if the adsorption process can be considered favorable or reversible, then the value is $0 < R_L < 1$; but if $R_L = 0$ then it can be said that the adsorption process is irreversible [10].

Table 3 shows that the R_L value for the adsorption process of Cr (VI) ions on the eugenol-silica gel composite is 0.0297 or is in the range of values $0 < R_L < 1$. This means that the adsorption process of the eugenol-silica gel composite on Cr (VI) can be said to be advantageous because the desorption process can be carried out [7]. The desorption process can occur due to the interaction that occurs between the metal ions of the eugenol-silica gel composite to absorb Cr (VI) in the test solution, or it can be said that it tends to be physically adsorption.

Physical adsorption occurs due to differences in energy or electrically charged attractive forces or commonly called Van der Walls forces and causes adsorbate molecules that have relatively weak interactions to begin to be physically bound to adsorbent molecules [11]. This interaction in physical adsorption occurs in substances at low or constant temperatures with relatively low adsorption so that the forces that restrain the adsorption of fluid molecules can usually be achieved quickly and are reversible, because they only require very little energy, so they are easy to break.

4 Conclusion

Based on the results of the research that has been carried out, it can be concluded that the optimum conditions for the adsorption process of Cr(VI) ions on the eugenol-silica gel composite using the Behnken response surface box method are contact time of 35 minutes, pH of 5, and weight of adsorbent 0, 15 grams at a concentration of 10 ppm Cr(VI) ion in a 50 mL solution volume. The maximum adsorption capacity (qm) is 1.0328 mg/g. The R_L value of 0.0297 indicates a favorable adsorption process and the K_F, K_L, and n values obtained respectively are 0.7961; 1.0885 and 14.0056. The adsorption of Cr(VI) ions on the

eugenol-silica gel composite followed the Langmuir isotherm.

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