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Optimization of Adsorption of Eugenol-Silica Gel Composites to Metal Ions Cu (II) Using Box Behnken Method

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Abstract

Silica gel can be used as an absorbent so that it can also be used as a process for making eugenol-silica gel composites. This study aims to optimize the adsorption of the eugenol-silica gel composite to Cu (II) metal ions and to determine the maximum absorption capacity and adsorption constant of the Langmuir and Freundlich isotherm model. This research begins with determining the optimization of contact time 10-60 minutes, temperature 20-45 ºC and the weight of the adsorbent is carried out in the range of 0.10-0.20 grams in a 50 mL solution and the experimental design of the box Behnken method will determine three variables through (Response Surface Methodology). Furthermore, the determination of Cu levels by preparing 100 ppm Cu mother liquor, making a standard series, and testing with Atomic Absorption Spectroscopy (AAS), and then determination of Langmuir and Freundlich adsorption isotherms. The results showed that the optimum conditions for the adsorption process using Response Surface Methodology (RSM) were at a contact time of 30 minutes, a temperature of 45 ºC with an adsorbent weight of 0.10 grams in a 50 mL solution, in Langmuir and Freundlich isothermal adsorption. The adsorption process takes place in two directions (reversible) the values of Kf, b and n are 8331; -5.2224 and -0.4541. The adsorption process takes place in two directions (reversible) the values of Kf, b and n are 8331; -5.2224 and -0.4541. The adsorption process takes place in two directions (reversible) the values of Kf, b and n are 8331; -5.2224 and -0.4541.

Keywords: Isothermal Adsorption; Eugenol-Silica Gel; Response Surface Methodology

1. INTRODUCTION

Heavy metals are dangerous pollutants because they cannot be degraded naturally and tend to accumulate in water and organisms [1]. Copper metal Cu is one of the materials that is widely used in industry, manufacturing, mechanics, electricity, and architecture [2], as well as industries such as textiles, mining, electroplating, fertilizer pipes and batteries [3]. Therefore, it is necessary to reduce waste, especially those containing Cu metal ions. One method that can be used to reduce Cu waste is the adsorption method which has several advantages such as the processing is relatively simple, effective and does not have a negative impact on the environment [4].

In a study conducted about the adsorption of Cu metal ions using $ZnFe_2O_4$ nanoparticle adsorbent resulted in an increase in temperature with a percentage of 100%. When using Fe_3O_4 there is a decrease in temperature with a percentage of 88.9%, Cu metal ions are not completely adsorbed. This shows that Cu^{2+} metal ions are adsorbed effectively with $ZnFe_2O_4$ [5]. Research related to glutaraldehyde crosslinked chitosan as an adsorbent in vetiver oil has been carried out. Experiments have been carried out on the effect of adsorption of Glutaraldehyde as an adsorbent on vetiver oil. An experiment has been conducted about the effect of adsorption on Cu (II) ions on the colour of vetiver oil is one of the one quality parameter that is the focus of this research. The results showed the percentage of Fe (III) ion adsorption decreased from 91,9521% to 79.724% due to a decrease in the percentage of adsorption both for the adsorption of Cu (II) metal ions. This is because the metal ions in vetiver oil have lower mobility compared to artificial conditions, and vetiver oil produces a dark colour due to the presence of Cu (II) metal ions bound to the components of vetiver oil (vetiverol) [6].

This research is the development of the research about synthesized a eugenol-silica gel composite made from corn cob ash [7]. The resulting silica gel eugenol composite will be tested as an adsorbent for the adsorption of Cu (II) metal ions on the composite, there are silica gel and eugenol as the basic ingredients, where the silica gel obtained is made from corn cob ash and Eugenol can improve the quality of silica gel as an adsorbent against metal ions such as Cu(II) metal ions. Eugenol or clove oil is an essential oil obtained from the distillation of the leaves and twigs of the clove plant. Clover leaf oil from the distillation of the people is often brownish black and

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dirty, so to increase the selling value it is necessary to purify it. From several refining results show that the oil can be purified by adsorption. Adsorption is the process of diffusion of a component on a surface or between particles. The adsorbent that can be used is the eugenol silica gel composite adsorbent. However, in this follow-up study, the eugenol-silica gel composite will be tested for optimum conditions on three variables to be determined, namely contact time, temperature and weight of the adsorbent using the Box Behnken method and the adsorption properties tested by the absorption of Cu (II) metal ions using Atomic Absorption Spectroscopy (AAS).

2. METHODS

The tools used in this research include: stirring rod, funnel, beaker, measuring cup, watch glass, Whatman 41 filter paper, measuring flask, digital balance, Mohr pipette, thermometer, stopwatch and the instrument used is an atomic absorption spectrophotometer. PG-990 models.

The materials used in this research include: distilled water, eugenol-silica gel composite, Cu^{2+} mother liquor.

2.1 Optimization of contact time, temperature, adsorbent weight and experimental design of the Box Behnken Design [6]

It can be determined that the experimental variables were made including temperature time and adsorption weight at a concentration of Cu 10 ppm and an adsorbent weight of 0.10-0.20 grams. Experimental variables were determined using the Box Behnken method using the Minitab application. Prepare a 100 mL volumetric flask with 15 volumetric flasks, add 10 mL of Cu (II) solution, each of which is inserted into the prepared volumetric flask. Added aquadest and marked as limiting, the standard Cu (II) solution is put into a beaker then heated on a hotplate waiting for a predetermined contact time and checking the optimal temperature of the solution using a thermometer, after checking the optimal temperature, the weight of the adsorbent is added (the weight of the adsorbent has been determined). Continued testing using the AAS instrument with a wavelength of 324.8 nm.

2.2 Determination of Cu Content, Cu^{2+} Standard Series Solution (SNI 6989.6:2009)

The standard $CuSO_4.5H_2O$ was weighed as much as 0.3929 grams and put into a 1 L volumetric flask and then aquadest was added up to the tera mark, in order to obtain 100 ppm Cu mother liquor. Preparation of a standard curve by preparing 6 volumetric flasks for the preparation of a standard series solution with a concentration of 2 volumetric flasks for each 0; 2; 4; 6; 8; 10 ppm followed by testing using the AAS instrument with a wavelength of 324.8 nm. Calculation of Cu content is carried out using the formula (SNI 6989.6.2009):

$$\mathrm{Cu}\left(\frac{\mathrm{mg}}{\mathrm{L}}\right) = \mathrm{C \ x \ fp}$$

The adsorption capacity can be determined using the equation:

$$qe = \frac{(Co - Ce)V}{m}$$

Meanwhile, the entrapment efficiency can be calculated based on the equation:

$$\%\mathrm{RE} = \frac{(\mathrm{Co-Ce})}{\mathrm{Co}}\mathrm{x}100\%$$

2.3 Equation of Adsorption Isotherm

After obtaining optimization data from the experiment using the Behnken response surface box, the next step is to prepare 5 volumetric flasks, each measuring flask is added with 10 mL of Cu solution and then 0.15 grams of adsorbent is added (weighed carefully) with a concentration of 10, 20, 30, 40, 50 ppm. The resulting solution was filtered with Whatman filter paper no. 41, followed by testing using an AAS instrument with a wavelength of 324.8 nm. The concentration is measured as the Ce value [8].

2.4 Determination of the Adsorption Isotherm Equation Determination of Langmuir Equation and Maximum Absorption Capacity

After the chart preparation process has been carried out by measuring the Cu (II) solution adsorption by silica gel corn cob ash, then calculations and plots are made on the graph so that the peak data will be obtained [8]. The results of the analysis of Cu content from the adsorption treatment were channelled to the absorption capacity with the equation:

$$\frac{\mathrm{Ce}}{\mathrm{Q}} = \frac{1}{\mathrm{Kb}} + \frac{1}{\mathrm{b}}\mathrm{Ce}$$

$$\mathrm{Y} \;=\; \mathrm{a} \;+\; \mathrm{b\,X}$$

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2.5 Determination of the Freundlich Adsorption Isotherm Equation

The logarithmic data of adsorption capacity (mg/g) as the Y axis is plotted against the final concentration log (equilibrium) as the X axis based on the equation:

$$\log Q = \log k + 1/n \operatorname{Log} Ce$$

$$Y \quad = \quad a \quad + \quad b\ X$$

2.6 Data processing

To get the optimum conditions, it is done by plotting the tested variables against the adsorbed %Cu to produce a 2-dimensional or 3-dimensional curve. Optimization results of temperature, time, adsorbent. And to get the Langmuir constant and maximum absorption capacity is done by graphical method based on Langmuir equation, and to get Freundlich constant is done by graphical method based on Freundlich equation.

3. RESULTS AND DISCUSSION

3.1 Determination of The Optimization Parameter with The Experimental Design of The Box Behnken Method

This research was conducted by using eugenol-silica gel composite as an adsorbent, to determine whether the adsorbent could adsorb metal ions Cu (II). The composite sample was made by a synthesis and characterization process which was carried out in previous studies through the FTIR testing stage aimed at showing the presence of an aromatic group (C=C) and testing the absorption capacity is known to be higher by 68-75% as a comparison in testing the water absorption capacity of food grade silica gel [7].

In the use of the atomic absorption spectrophotometer (AAS), the standard mother liquor was prepared using $CuSO_4.5H_2O$ at a concentration of 100 ppm, then followed by measuring the calibration curve, so that the resulting concentration and absorbance data were known.

In determining the linearity test regression from the calibration standard curve, a correlation value of r was obtained of 0.999 which indicates a close linear relationship between the concentration measured and the adsorbent produced.

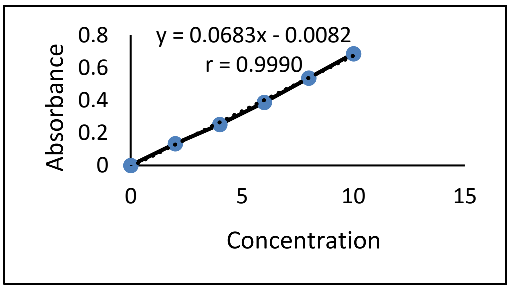

Figure 1. Cu Standard Calibration Curve

The linear regression obtained is in accordance with that determined by SNI, which is 0.995, which means that the concentration obtained is proportional to the absorbance value obtained so that the resulting curve meets the requirements to be used as a test work standard area so as to minimize the opportunity for errors during analysis dan After going through the standard, it can be argued that R^2 which stands for 99.5% are derived from analyte and 0.5% as additives. After going through the standard curve linear regression calculation, y = ax + b, then we get y = 0.0683x -0.0082 so that we can calculate the concentration in the sample.

3.2 Results of Determining Optimum Conditions for Cu (II) Metal Ion Adsorption by Eugenol-Silica Gel Composites

Determination of the optimization of Cu (II) ion adsorption by eugenol-silica gel composite has been carried out by determining three independent variables, namely contact time, temperature and weight of the adsorbent using the experimental design of the box Behnken method. Where the optimum condition is a solution that can be absorbed maximally by an adsorbent. In order to obtain maximum and effective results, the application of eugenol-silica gel composite as an adsorbent in the adsorption of Cu (II) metal ions was determined at a concentration of 10 ppm in an optimum condition that could be adsorbed by the adsorbent.

Based on the response from the results obtained from determining the optimization of contact time, temperature and weight of the adsorbent with the experimental design of the box Behnken method, it can be seen in Table 1.

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Table 1. The results of measuring the response of the Behnken Box surface from the adsorption of Cu (II) metal ions by Silica Gel-Eugenol with this 10-60 min you should give more time range so we can see the curve of the maximum composites.

No	Time (minute) (X_1)	Temperature (X_2)	Weight adsorbent (g) (X_3)	[Cu (II)] (ppm)			Adsorption (%)
				Beginning	End	Adsorbent	
1	10	25	0.15	10	5.680	4.32	43.2%
2	60	25	0.15	10	7.256	2,744	27.44 %
3	10	45	0.15	10	2.872	7.128	71.28%
4	60	45	0.15	10	5.680	4.32	43.2%
5	10	35	0.10	10	5.165	4.835	48.35%
6	60	35	0.10	10	3.696	6.304	63.04 %
7	10	35	0.20	10	7,985	2.105	21.05%
8	60	35	0.20	10	8,681	1.319	13.19%
9	35	25	0.10	10	3.689	6.311	63.11%
10	35	45	0.10	10	3,690	6.31	63.1 %
11	35	25	0.20	10	6.485	3.515	35.15%
12	35	45	0.20	10	6,985	3.015	30.15%
13	35	35	0.15	10	5.680	4.32	43.2%
14	35	35	0.15	10	5.165	4.835	48.35%
15	35	35	0.15	10	5.160	4.84	48.4%

One way to show the response surface is to make a contour plot and find out the results of the response consisting of several colours with three independent variables that affect the response, namely contact time, temperature and weight of the adsorbent.

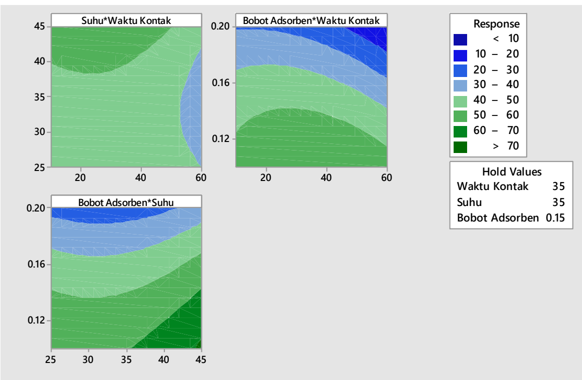

Figure 2. Contour Plots Response Surface Method Box Behnken

Where each variation shows the magnitude range of the resulting response. At the maximum condition for the plot above, it is in dark green with a response value above 70%. This range will provide an outline of the location of the optimum point. Based on the results of the Behnken response surface box experiment, a decision was made to determine the optimum conditions set by the response optimizer on the Minitab application with the results obtained, namely a contact time of 30 minutes, temperature 45 ^oC and adsorbent weight of 0.10 grams.

3.3 Results of Analysis of Surface Response Data by Box Behnken method

In the results of the data analysis of the contact time response that has been obtained from the measurement data input in the Minitab 17 application, it shows that the p-value obtained from the response to the model is 0.148 which this value states that the model cannot be tolerated or is not significant because the p-value > 0.005 and the linear p-value is 0.033, where the value for linearity obtained cannot be tolerated, meaning that the data cannot be accepted or has no effect on the response. so that it is known that the model formula used from the three factors namely contact time, temperature, and weight of the adsorbent does not provide a significant difference. The data obtained at the contact time and temperature are not appropriate because they exceed the standard data which means that it does not have a significant effect on the response. when the weight of the adsorbent is

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added, the p-value obtained is p-value, which is 0.005, the data can be tolerated, so it is known that the model formula used from the three factors, namely contact time, temperature, and weight of the adsorbent, gives a significant difference. Obtained R^2 value of 82.69% which states the influence of the three variables. The adjusted R^2 value of 62.70% states that there is a relationship between the three variables on the contact time response [9]. The difference between the R^2 value and the adjusted value in this response is 19.99% so that the difference is not in accordance with the expected conditions, which is less than 20% or 0.2% [10]. So, it is known that the model formula used from the three factors namely contact time, temperature, and weight of the adsorbent gives a significant difference. Obtained R^2 value of 82.69% which states the influence of the three variables. The adjusted R^2 value of 62.70% states that there is a relationship between the three variables on the contact time response [9].

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Lack of fit is a deviation or inaccuracy to the model, if the lack of fit value is unreadable or insignificant, it becomes a requirement for a good model because it can indicate a match between the response data and the model [11]. In the Lack of fit data, the three variables obtained have a p-value of 0.039 which states that the value is not significant, thus indicating that this model is classified as good.

Data analysis was carried out using quadratic model analysis to obtain an optimization model with the equations obtained for the contact time response expressed in the following equation:

$$Y = 55 + 1.58\ X_1 - 2.67\ X_2 + 424\ X_3 - 0.00965\ X_1^2 - 0.0566 X_2^2 + 1774\ X_3^2 - 0.0123\ X_1X_2 - 4.33\ X_1X_3 - 2.5\ X_2X_3$$

If the response variables are concluded, namely contact time (X_1), temperature (X_2), adsorbent weight (X_3) and response variable (Y).

3.4 Determination of Adsorption Capacity Langmuir and Freundlich Equations

Determination of adsorption isotherm is done by processing the data obtained from research with variations in the concentration of Cu (II) metal ion solution. The weight of the adsorbent is conditioned according to the known optimum conditions of 0.10 gram.

From the result data in Table 2. A curve of the relationship between the adsorption capacity and the initial concentration of Cu (II) metal ions was drawn up at equilibrium in solution. The Freundlich and Langmuir adsorption isotherm curves can be seen in Figure 2.

Table 2. Isothermal Adsorption Experimental Data of Cu(II) metal ions by Eugenol-Silica Gel . Composite

C_0 (mg/L)	Ce (mg/L)	Volume (L)	Adsorbent Weight (g)	C_{Ae} (mg/L)	q_e (mg/g)	Adsorption Efficiency (%)
10	3.6916	0.05	0.10	6.3084	3.1542	63.08 %
15	3.8659			11.1341	5.5671	74.23%
20	4.1226			15.8774	7.9387	79.39%
25	4.4981			20.5019	10.2510	82.01%
30	4.7835			25.2165	12.6083	84.06 %

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The Freundlich isotherm curve is the curve of the relationship between log qe and log Ce, from the curve obtained, it can be seen that the results of linear regression analysis (R^2) are 0.9168. while the Langmuir isotherm curve is from the relationship between Ce and Ce/qe, from the determination of the Langmuir adsorption isotherm equation, y = -0.6092x + 3.1946 with an R^2 value of 0.7338.

In the isothermal analysis of the adsorption of Cu (II) metal ions by the eugenol-silica gel composite, two models were used, 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. Where qe is the weight of solute per gram of adsorbent and Ce is the concentration of solute in solution. The data from the Langmuir and Freundlich isotherm analysis are presented in Table 3.

The data obtained in the study with variations in the concentration of the solution can be used in determining the pattern of adsorption isotherms. The line equation for determining the Langmuir adsorption isotherm is y = -0.6092 x+ 3.1946 and the line equation for determining the Freundlich adsorption isotherm is y = 4.9207 x -2.020. From Figure 2 it can be seen that the Langmuir and Freundlich isotherm equation with an R^2 value of 0.7338 and 0.9168 for the R^2 value of the Freundlich isotherm.

Judging from the R^2 value, the Cu (II) metal ion adsorption model by Eugenol-Silica Gel Composite which is in accordance with the Freundlich isotherm is compared with the Langmuir isotherm. These results are known to be in accordance with the results of research conducted by Oon, LL, et al [12] that in the study of cadmium adsorption using seaweed (*Kappaphycus alvarezii* and *Eucheuma denticulatum*) followed the Freundlich isotherm pattern. It is known that the adsorption of Cu (II) metal ions by the Eugenol-Silica Gel Composite according to the Freundlich isotherm pattern indicates that the metal ions from the solution are adsorbed from many layers (multilayer) of adsorbate molecules on the adsorbent where adsorption on the adsorbent surface occurs at active sites that heterogeneous.

Judging from the line equation of the Freundlich isotherm curve, namely y = 4.9207 x - 2.2020, it can be determined the relative ability of the silica gel-eugenol composite to adsorb metal ions Cu (II) and can determine the strength of the interaction between Cu (II) ions with silica gel-eugenol composite surface. The Freundlich isotherm linear equation is log Qe = log Kf + 1/n log Ce, which is the relative ability of an adsorbent to adsorb, for the strength of the interaction between adsorbent and adsorbate can be seen from 1/n, the smaller the value of 1/n, the more strong interaction between adsorbent and adsorbate [13]. It is known that the relative adsorption ability of the eugenol-silica gel composite in adsorption of Cu (II) metal ions with a Kf value of 8331 indicates the interaction strength between Cu (II) ions with a 1/n value obtained of -0.4541. In the eugenol-silica gel composite adsorbent, the Kf and 1/n values were 8331 and -0.4541, respectively. This indicates that the ability of the eugenol-silica gel composite to adsorb metal ions Cu (II) is smaller when the weak interaction strength between the adsorbent and the adsorbate can be recovered more easily.

Table 3. Langmuir and Freundlich Adsorption Isothermal Analysis Data Cu metal ions by Eugenol-Silica Gel Composite

Ce	Qe	Isothermal Analysis			
		Langmuir		Freundlich	
		Ce	Ce/qe	Ce logs	qe logs
3.6916	3.1542	3.6916	1.1704	0.5672	0.4989
3.8659	5.5671	3.8659	0.6944	0.5873	0.7456
4.1226	7.9387	4.1226	0.5193	0.6152	0.8997
4.4981	10.2510	4.4981	0.4388	0.6530	1.0108
4.7835	12.6083	4.7835	0.3794	0.6797	1.1007

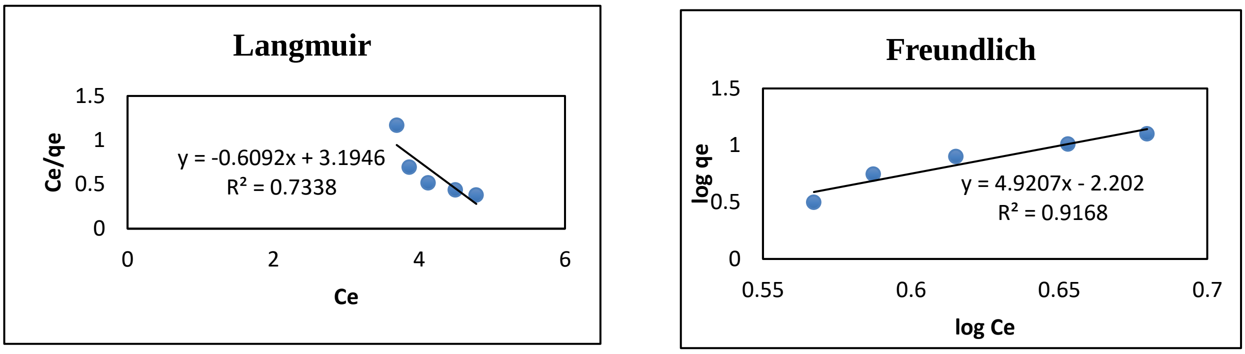

Figure 3. Langmuir and Freundlich isotherms for the adsorption of Cu (II) metal ions by Eugenol-Silica Gel Composites.

Table 4. Langmuir and Freundlich Adsorption Isothermal Parameters of Cu (II) Metal Ions by Eugenol-Silica Gel Composites

Langmuir			Freundlich		
q_m (mg/g)	B	R^2	K_F	n	R^2
0.3031	-5.2444	0.7338	8331	-0.4541	0.9168

4. CONCLUSION

The adsorption of Cu (II) ions by the eugenol-silica gel composite using the box Behnken method can determine the optimum conditions in the response optimizer design in Minitab software, which absorbs three independent variables, namely contact time, temperature and weight of the adsorbent, namely 30 minutes, 45 °C and 0, 10 grams and the adsorption isotherm of Cu (II) metal ions by the eugenol-silica gel composite according to the Freundlich adsorption isotherm with the equation y = 4.9206 x -2.3781 where the K_F value is 8331, the 1/n value is -0.4541 and get a linear line value (R^2) of 0.9167.

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