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UTILIZATION OF SENGON WOOD SAWDUST WASTE AS ESTERIFIED CELLULOSE-BASED BIOADSORBENT FOR CD(II) WASTEWATER PURIFICATION

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ABSTRACT

Cadmium (Cd) is a heavy metal with high toxicity that can enter the body through contaminated food chains, so wastewater containing cadmium needs to be treated before being discharged into the environment. This study uses adsorption methods to reduce Cd(II) concentration in artificial wastewater by utilizing modified sengon wood sawdust cellulose as a bioadsorbent. Adsorption tests were conducted in batch mode at room temperature with constant stirring, and the results were analyzed using AAS. The objective of this study was to determine the optimum adsorption conditions using Response Surface Methodology (RSM) and to determine the adsorption capacity of the modified bioadsorbent based on the Freundlich or Langmuir adsorption isotherm models. The variations in bioadsorbent dose in this study were 2, 4, and 6 g/L; adsorption time variations were 30, 60, and 90 minutes; pH variations were 3, 4, and 5. Based on the design expert 13 RSM software, the optimum adsorption conditions for citric acid-modified bioadsorbent are a bioadsorbent dose of 3.957 g/L, time of 31.655 minutes, and pH of 4.968. For the EDTA-modified bioadsorbent, the optimum conditions were a bioadsorbent dose of 3.836 g/L, time of 30 minutes, and pH of 4.708. The results of the adsorption isotherm modeling indicate that the citric acid-modified and EDTA-modified bioadsorbents are more suitable for the Freundlich isotherm model due to their higher determination coefficient (R^2). Thus, the adsorption capacities for the citric acid-modified and EDTA-modified bioadsorbents were determined to be 1.7828 mg/g and 1.5776 mg/g, respectively.

Keyword: *Adsorption, Cadmium, Esterification, Sengon wood, Wastewater*

1. INTRODUCTION

Cadmium (Cd) is a type of non-essential heavy metal with high toxicity [1]. This heavy metal can be found in textile industrial waste and mining waste. Cadmium waste is classified as hazardous and toxic waste (B3), and its treatment and disposal must be carefully managed. The concentration of Cd in aquatic environments needs to be precisely monitored because it can accumulate and poison biotic components [2]. According to the Regulation of the Minister of Environment of the Republic of Indonesia Number 05 of 2014 concerning wastewater quality standards for industries and other activities, the maximum permissible limit for cadmium concentration in wastewater is 0.1 ppm. Cadmium can accumulate in the human body through a contaminated food chain, leading to acute illnesses due to its carcinogenic properties. According to the World Health Organization, the maximum acceptable limit for cadmium is 7 mg per kg of body weight. Therefore, to prevent the widespread impact of this heavy metal, a method is needed to reduce its concentration. One alternative for treating cadmium-containing waste is the adsorption method.

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Adsorption is the process where a specific substance is adsorbed by a particular solid on its surface due to the attractive forces of atoms or molecules on the solid's surface, without penetrating the solid [3]. The adsorbing substance, or adsorbent, commonly uses organic materials because they have been proven effective in reducing the concentration of heavy metals in wastewater. One organic material that can be used as an adsorbent is cellulose. The main advantage of using cellulose as an adsorbent is its abundant availability.

Wood sawdust is a type of waste that can be used as a bioadsorbent because it contains cellulose. Another study utilizing sawdust to adsorb cadmium was conducted using sawdust from teak wood [4]. Besides reducing waste, sengon wood sawdust has a high cellulose content of 41,17%, followed by 22,26% hemicellulose and 17,51% lignin [5]. The high cellulose content has the potential to be used for adsorbing divalent cadmium ions. This is because cellulose has an abundance of active carboxyl and hydroxyl groups.

In this study, the researchers innovated by utilizing cellulose from sengon wood sawdust, which was modified through esterification with citric acid and EDTA to enhance the bioadsorbent's capacity for adsorbing cadmium metal. Research by Sulistyawati et al. mentioned that activating cellulose with EDTA can increase heavy metal adsorption capacity by forming carboxyl and amine functional groups [6]. Similarly, researchers Ulfa et al. stated that the esterification reaction of cellulose with citric acid can increase the hydroxyl and carboxyl groups, which act as adsorbents for metal ions [7].

Sengon wood (*Albizia chinensis*) is widely used as a raw material for housing, such as boards, beams, poles, and for making crates, pulp, and matches. The use of cellulose in this bioadsorbent also serves as an alternative for treating the ever-increasing amount of sawdust waste. Therefore, further research is needed to determine the optimum adsorption operating conditions, including the adsorbent dose, adsorption time, and pH. Appropriate operating conditions can increase adsorption effectiveness, which is indicated by an increased adsorption capacity of the bioadsorbent sample.

2. RESEARCH METHODOLOGY

2.1 Materials and Equipment

The materials and equipment used are shown in Table 1.

Table 1. Materials and Equipment

Materials	Equipment
Sengon wood sawdust	Blender
NaOH p.a	Sieve shaker
Citric acid monohydrate p.a	3-neck round-bottom flask
Na_2EDTA p.a	Thermometer
Aquadest	Stirring motor
Universal pH indicator	Oil bath
Filter paper	Buchner funnel
Aluminium foil	Vacuum pump
Cadmium sulfate hydrate p.a	Measuring pipette
	Measuring flask
	Hot plate
	Oven
	Magnetic stirrer
	Analytical balance
	Beaker glass
	Atomic absorption spectrophotometer

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2.2 Methods

The research procedures are shown in Figure 1.

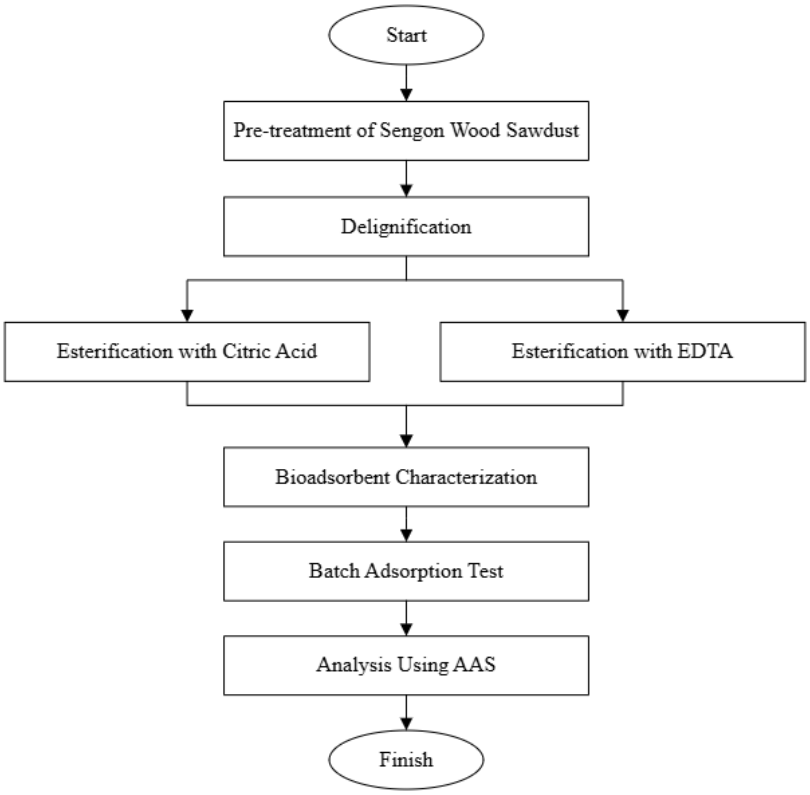

Figure 1. Research Procedures

2.2.1 Pre-treatment of Sengon Wood Sawdust

The pre-treatment of sengon wood sawdust was carried out by washing and reducing the size of sengon wood sawdust using a blender, after which it was dried in the oven at 110 ℃ for 24 hours, after drying it was sieved using a sieve shaker and particle diameter size of 0.149-0.210 mm was selected.

2.2.2 Delignification

A total of 50 grams of sengon wood sawdust (0.149-0.210 mm) was mixed with 1000 mL of 0.5 M NaOH solution in a three-neck flask. The mixture was stirred at 180 rpm at ambient temperature for 2 hours, then the residue was filtered, neutralized using distilled water to pH ± 7, and dried in an oven at 50 ℃ for 24 hours. The results of the delignification process were calculated to determine the final weight.

2.2.3 Esterification of Sengon Wood Sawdust Cellulose with Citric Acid

A total of 20 grams of delignified Sengon wood sawdust was mixed with 69 grams of citric acid and 300 mL of aquadest into a three-neck flask. The esterification process was carried out at 120°C for 6 hours. After the reaction process was complete, the slurry was neutralized with warm distilled water until pH ± 7, then filtered, and dried at 50 ℃ for 24 hours.

2.2.4 Esterification of Sengon Sawdust Cellulose with EDTA

A total of 20 grams of delignified sengon wood sawdust was mixed with 69 grams of EDTA and 300 mL of aquadest into a three-neck flask. The esterification process was carried out at 100℃ for 2 hours. After the reaction process was complete, the slurry was neutralized with warm distilled water until pH ± 7, then filtered and dried at 50 ℃ for 24 hours.

2.2.5 Bioadsorbent Characterization

Bioadsorbents before and after esterification were characterized using FT-IR (Fourier Transform-Infra Red) to obtain functional groups and also using SEM-EDX (Scanning Electron Microscope-energy Dispersive X-ray) to obtain surface morphology of bioadsorbents.

2.2.6 Batch Adsorption Test

Adsorption tests were conducted on a laboratory scale with a batch operating system and room temperature. The test was conducted by mixing a certain amount of Cellulose-Citrate bioadsorbent into

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a reactor containing 25 ml (30 ppm) Cadmium solution. The reaction was carried out at room conditions and constant stirring with experimental variations of pH, the amount of cellulose-citrate bioadsorbent dose, and contact time. Then the solution was filtered, and the filtrate was measured for the unadsorbed Cd (II) metal content using AAS (Atomic Absorption Spectrophotometry). Cd (II) concentration data were analyzed using Response Surface Methodology (RSM) to obtain the optimum conditions of the process used for adsorption tests to determine adsorption capacity with variations in Cd (II) concentrations of 6; 12; 18; 24; 30 mg/L. The batch adsorption test procedure was repeated to cellulose-EDTA bioadsorbent.

2.2.7 Analysis of Adsorption Test Results Using AAS

Quantitative analysis of samples to determine the concentration of Cadmium was carried out on the intial solution (at t = 0 minutes) and the adsorption test results solution. The concentration was determined using AAS (Atomic Absorption Spectrophotometry) at a wavelength of 228.8 nm.

2.3 Data Analysis

The optimum operating conditions can be known from the percentage reduction in Cd (II) content, analyzed by RSM (Response Surface Methodology). The adsorption capacity was obtained using the Langmuir adsorption isotherm method. The Langmuir isotherm equation can be seen in equation 1.

$$\frac{C}{m} = \frac{1}{bK} + \frac{C}{b} \quad (1)$$

Where:
C = Adsorbate concentration after adsorption (mg/L)
m = Mass of adsorbate per unit mass of adsorbent (mg/g)
K = Langmuir constant
b = Maximum adsorption capacity of bioadsorbent (mg/g)
Plotting C/m against C will result a straight line with slope = 1/b and intercept = 1/bK, so that the K and b constants can be determined.
Another method used is the Freundlich adsorption isotherm method. The Freundlich isotherm equation can be seen in equation 2.

$$\log m = \log K + \frac{1}{n} \log C \quad (2)$$

Where:
m = Mass of adsorbed adsorbate per unit mass of adsorbent (mg/g)
C = Equilibrium concentration of adsorbate after adsorption (mg/L)
K = Freundlich constant associated with adsorption capacity (mg/g)
n = Freundlich exponent
Plotting log m against log C will result a straight line with slope = 1/n and intercept = log K, so that the constants n and K can be determined.

3. ANALYSIS AND RESULT

3.1 Characterization Using SEM-EDX

The presence of lignin will reduce the adsorption process because lignin blocks the ion transfer process (in this case cadmium ions) to the active side of the adsorbent. Therefore, a delignification process using 0.5 M NaOH was carried out to destroy lignin which is a binding material and open the inside containing cellulose. OH^- ions from NaOH will break the bonds of the basic structure of lignin so that lignin will dissolve easily (Farida et al., 2019).

Figure 2 shows the SEM-EDX test results before the delignification process, seen the morphology of sengon wood sawdust which is tight and has no voids, this part is indicated as lignin which fills the surface of plant cells. While after the delignification process in Figure 3, the morphology of sengon wood sawdust appears to have cavities containing cellulose.

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Figure 2. Surface Morphology of Sengon Wood Sawdust Before Delignification

Figure 3. Surface Morphology of Sengon Wood Sawdust After Delignification

Sengon wood sawdust was then modified through an esterification process with citric acid and EDTA to increase the active groups that will adsorb Cd(II). Based on the SEM-EDX test results of sengon wood sawdust modified with citric acid in Figure 4, the fiber structure was not damaged after the esterification process. While the sengon wood sawdust modified with EDTA in Figure 5, also shows there are still cavities or pores.

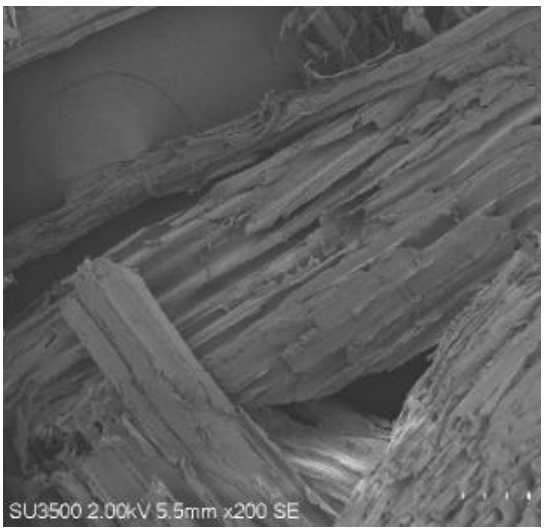

Figure 4. Surface Morphology of Citric Acid-Modified Sengon Wood Sawdust

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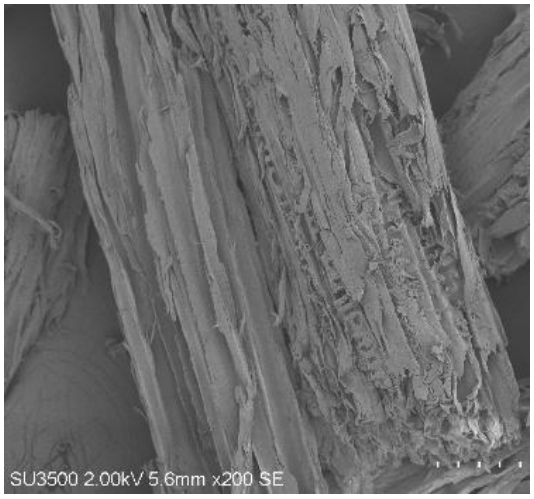

Figure 5. Surface Morphology of EDTA-Modified Sengon Wood Sawdust

3.2 Characterization Using FT-IR

FT-IR tests were carried out on delignified sengon wood sawdust or before modification, citric acid modified, and EDTA modified. FT-IR testing before and after modification aims to see changes or additions of functional groups on bioadsorbents.

Based on the FT-IR test results in Figure 7 which is citric acid-modified sengon wood sawdust, there is a new spectra at wave number 1726.29 cm^{-1} which is the spectra of C=OOR or ester group. In addition, the undetected C=OOH spectra can be caused by an overlap with the C=OOR spectra because the wave numbers are both close together. The carboxylic group on citric acid forms an ester bond with the hydroxyl group on cellulose. The esterification reaction of cellulose with citric acid results in an increase in -OH and COO- groups that are active to adsorb metal ions [7].

In Figure 8, which is EDTA-modified sengon wood sawdust, there is a medium-strong absorption intensity at a wave number of 1595.13 cm^{-1} which is the spectra of N-H or amine groups, but there are no spectra that show ester groups. This could be due to insufficient esterification time or the operating temperature is not high enough because the reaction does not involve a catalyst, so ester groups have not formed after esterification with EDTA. Although there are no ester groups, there are still other functional groups to adsorb cadmium, including hydroxyl groups and amine groups. Hydroxyl groups (-OH) play a role in the adsorption process of Cd(II). According to Farida et al., the more functional groups such as -OH and -COOH, the more metals can be adsorbed by the adsorbent through ion exchange or complex formation so that it will increase the adsorption capacity of the adsorbent [8].

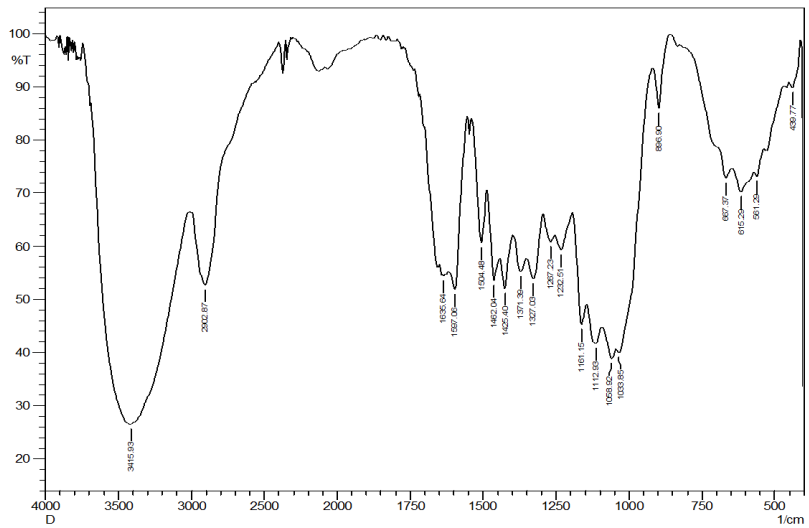

Figure 6. FT-IR Spectrum of Sengon Wood Sawdust Before Modification

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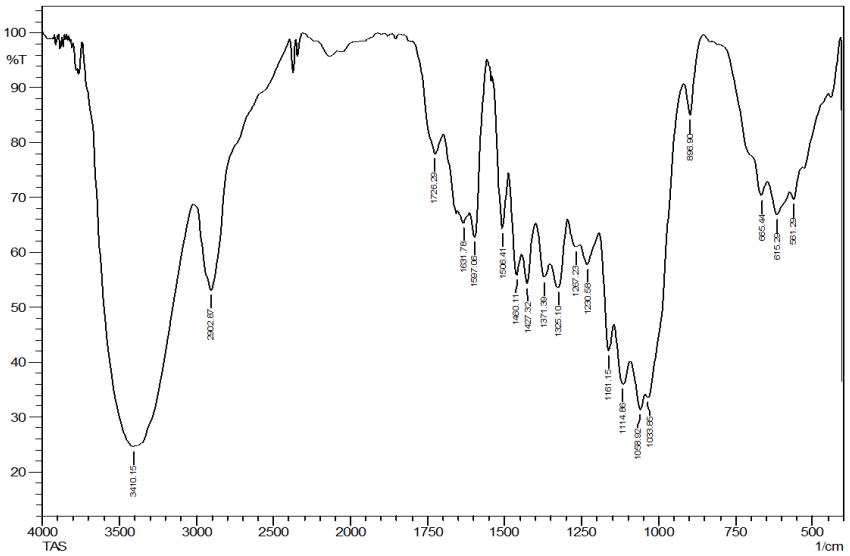

Figure 7. FT-IR Spectrum of Citric Acid-Modified Sengon Wood Sawdust

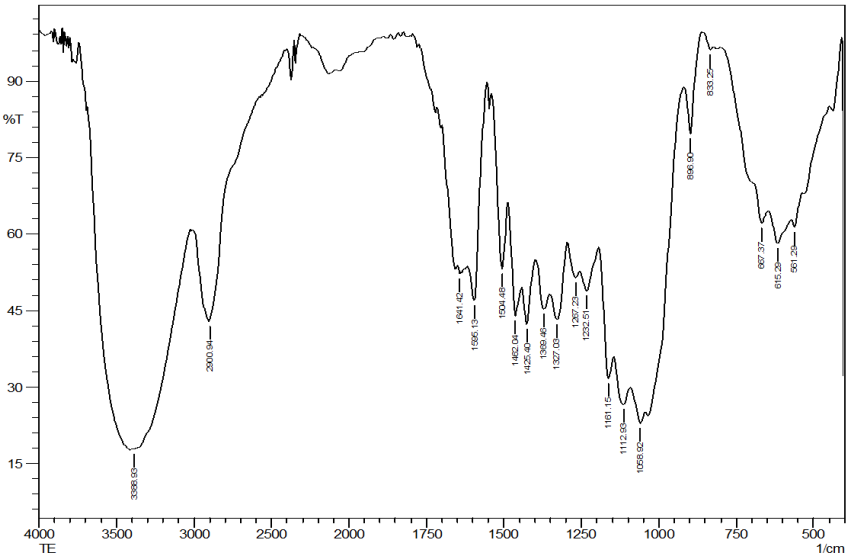

Figure 8. FT-IR Spectrum of EDTA-Modified Sengon Wood Sawdust

Table 2. Interpretation of FT-IR Spectrum Data on Sengon Wood Sawdust Before and After Modification

Functional Groups	Reference	Wave Number (cm^{-1})		
		Before Modification	Citric Acid-Modified	EDTA-Modified
-OH	3650-3200	3415.93	3410.15	3388.93
C-H	3000-2850	2902.87	2902.87	2900.94
C=OOH	1725-1700	-	overlap with the C=OOR spectra	-
C=OOR	1750-1725	-	1726.29	-
C-O	1300-1000	1033.85	1033.85	1058.92
		1058.92	1058.92	1112.93
		112.93	1114.86	1161.15
		1161.15	1161.15	1232.51
		1232.51	1230.58	1267.23
		1267.23	1267.23	
N-H	1640-1550	-	-	1595.13

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3.3 Determination of Optimum Adsorption Conditions

Based on Figures 9 and 10, the adsorption time for pH 5 has reached optimum conditions at 30 minutes and is quite stable until 90 minutes. Cd(II) adsorption test by citric acid-modified and EDTA-modified cellulose bioadsorbent conducted at pH 5 resulted in a greater adsorption capacity value than pH 3 and 4. Based on the 3-dimensional curve, the adsorption capacity is more stable at pH 5. Whereas at pH 3 and 4, adsorbed Cd(II) easily dissolves again. Based on the research of Rofiansyah & Setiarso and Riskadita, the adsorption pH of Cd(II) is below 8 or acidic pH to pH 7. When the pH is above 7, it causes Cd(II) to form $Cd(OH)_2$ [9], [10]. While at pH 3 which is an acidic condition and has excess H^+, there can be competition between H^+ ions and Cd^{2+} ions to bind with cellulose during the adsorption process [11].

The optimum conditions for Cd(II) adsorption by citric acid-modified cellulose bioadsorbent obtained from design expert 13 RSM software are bioadsorbent dose of 3.957 g/L; time 31.655 minutes; and pH 4.968 while for EDTA-modified cellulose bioadsorbent is bioadsorbent dose of 3.836 g/L; time 30 minutes; and pH 4.708.

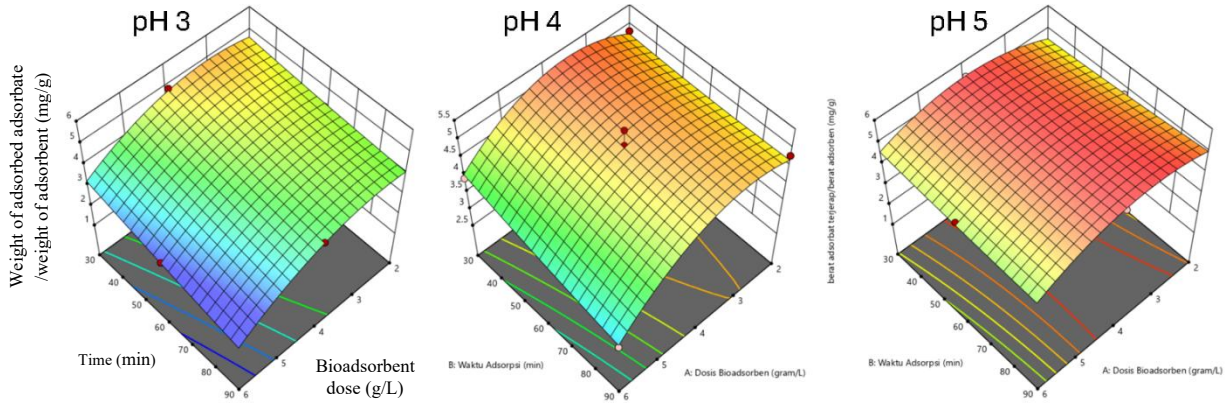

Figure 9. Surface 3-Dımensıonal Curve for Optimum Condition Analysis of Cd(II) Adsorption Test by Citric Acid-Modified Cellulose Bioadsorbent using RSM

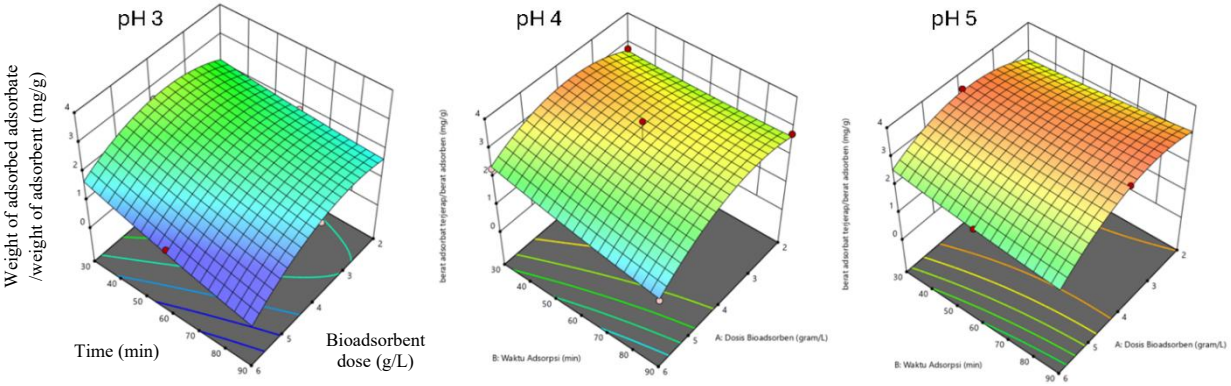

Figure 10. Surface 3-Dımensıonal Curve for Optimum Condition Analysis of Cd(II) Adsorption Test by EDTA-Modified Cellulose Bioadsorbent using RSM

3.4 Determination of Adsorption Isotherm Model

Adsorption isotherms are used to describe how the interaction between adsorbate molecules or ions with active sites on the adsorbent surface [12]. Adsorption isotherms state the equilibrium relationship between concentration in the fluid phase and concentration in adsorbent particles at a certain temperature [13].

The Langmuir isotherm model represents quantitatively the formation of a monolayer adsorbate on the outer surface of the adsorbent after which no further adsorption occurs. The assumption used in this

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model is that active sites have the same affinity and are independent of other active sites [12]. The Langmuir adsorption isotherm modeling results curve can be seen in Figure 11 and 12.

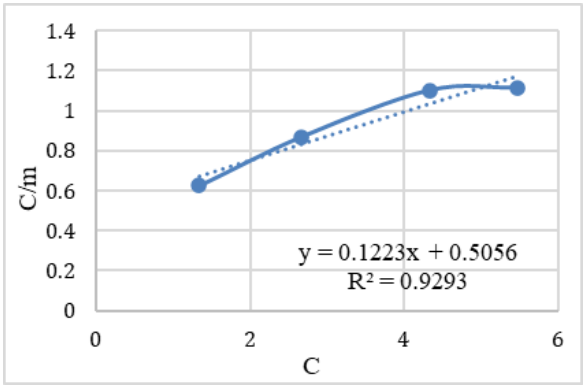

Figure 11. Langmuir Isotherm Curve of Cd(II) Adsorption by Citric Acid-Modified Cellulose Bioadsorbent

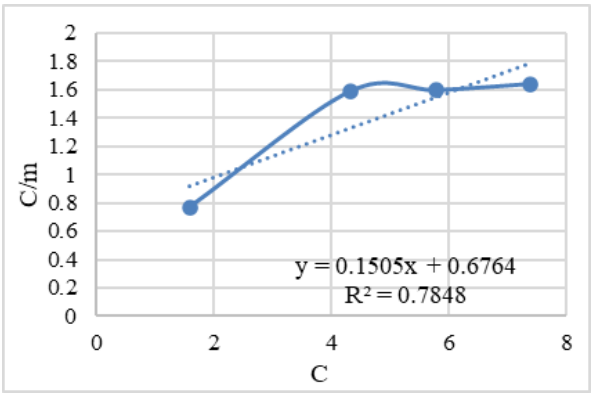

Figure 12. Langmuir Isotherm Curve of Cd(II) Adsorption by EDTA-Modified Cellulose Bioadsorbent

The Freundlich isotherm model is used to explain heterogeneous adsorption and describes the nature of adsorption that occurs is a physical process, multilayer and the bond is not strong [12]. The Freundlich adsorption isotherm modeling results curve can be seen in Figure 13 and 14.

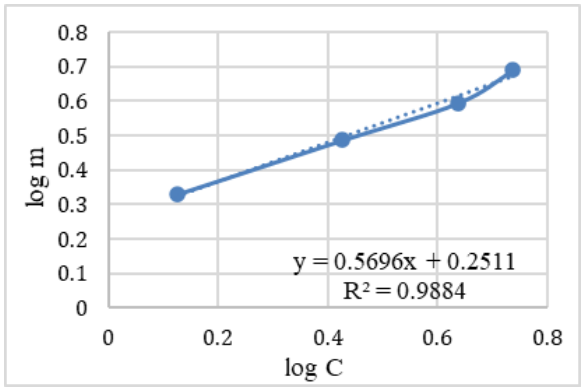

Figure 13. Freundlich Isotherm Curve of Cd(II) Adsorption by Citric Acid-Modified Cellulose Bioadsorbent

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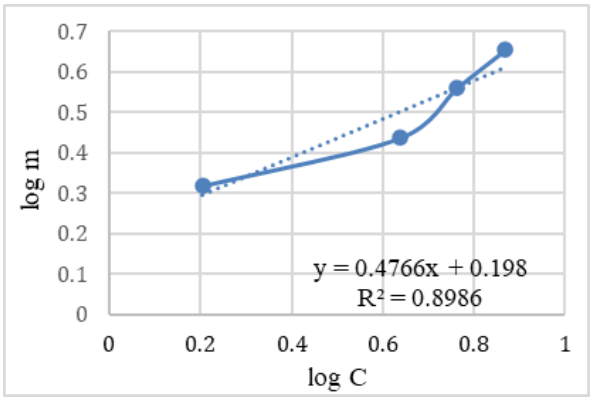

Figure 14. Freundlich Isotherm Curve of Cd(II) Adsorption by EDTA-Modified Cellulose Bioadsorbent

For citric acid-modified and EDTA-modified cellulose bioadsorbents, it is more suitable to use Freundlich adsorption isotherm modeling, because the coefficient of determination (R^2) value of the Freundlich isotherm curve is greater than the Langmuir isotherm curve. This indicates that multilayer adsorption occurs on heterogeneous adsorbent surfaces with different energy sites or without having a saturation term [14], [15].

The adsorption capacity obtained and the constants of the two adsorption isotherm models can be seen in Table 3. The data proves that citric acid-modified cellulose bioadsorbent provides greater adsorption capacity than EDTA-modified cellulose bioadsorbent.

Table 3. Adsorption capacity and constant values of the Freundlich and Langmuir Adsorption Isotherm Models

Modified Bioadsorbent	Freundlich		Langmuir	
	n	K (mg/g)	K_L	b (mg/g)
Citric acid	1.7556	1.7828	0.2419	8.1766
EDTA	2.0982	1.5776	0.2225	6.6445

Where:
n = Freundlich exponent
K = Adsorption capacity of Freundlich isotherm (mg/g)
K_L = Langmuir constant
b = Adsorption capacity of Langmuir isotherm (mg/g)

4. CONCLUSION

The adsorption process of Cd(II) in an aqueous solution at room temperature with constant stirring in batches is influenced by bioadsorbent dosage, contact time, and solution pH. The results of analysis using Response Surface Methodology (RSM) showed the optimum adsorption conditions for citric acid-modified bioadsorbent were a bioadsorbent dose of 3.957 g/L; time of 31.655 minutes; and pH 4.968. While the optimum adsorption conditions for EDTA-modified bioadsorbent are a bioadsorbent dose of 3.836 g/L; time of 30 minutes; and pH of 4.708. Adsorption isotherm modeling results show that citric acid-modified and EDTA-modified bioadsorbents are more suitable using the Freundlich isotherm model, so the adsorption capacity values for citric acid-modified and EDTA-modified cellulose bioadsorbents are 1.7828 mg/g and 1.5776 mg/g, respectively.

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